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Improved Electricity Generation by a Microbial Fuel Cell after Pretreatment of Ammonium and Nitrate in Livestock Wastewater with Microbubbles and a Catalyst

Jae Kyung Jang*, Taeyoung Kim, Sukwon Kang, Je Hoon Sung, Youn Koo Kang, and Young Hwa Kim

Energy and Environmental Engineering Division, National Institute of Agricultural Science, Rural Development Administration, Jeonju 54875, Republic of Korea

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**Corresponding author*
Phone: +82-63-238-4074;
Fax: +82-63-238-4078;
E-mail: jkjang1052@korea.kr

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Livestock wastewater containing high concentrations of ammonium and nitrate ions was pretreated with microbubbles and an Fe/MgO catalyst prior to its application in microbial fuel cells because high ion concentrations can interfere with current generation. Therefore, tests were designed to ascertain the effect of pretreatment on current generation. In initial tests, the optimal amount of catalyst was found to be 300 g/l. When 1,000 ml/min O_2 was used as the oxidant, the removal of ammonium- and nitrate-nitrogen was highest. After the operating parameters were optimized, the removal of ammonium and nitrate ions was quantified. The maximum ammonium removal was 32.8%, and nitrate was removed by up to 75.8% at a 500 g/l catalyst concentration over the course of the 2 h reaction time. The current was about 0.5 mA when livestock wastewater was used without pretreatment, whereas the current increased to 2.14 ± 0.08 mA when livestock wastewater was pretreated with the method described above. This finding demonstrates that a 4-fold increase in the current can be achieved when using pretreated livestock wastewater. The maximum power density and current density performance were 10.3 W/m^3 and 67.5 A/m^3, respectively, during the evaluation of the microbial fuel cells driven by pretreated livestock wastewater.

Keywords: Microbial fuel cell, livestock wastewater, electricity generation, microbubble, catalyst, ammonium

Introduction

In Korea, interest in the treatment of livestock waste has increased since the ocean dumping of livestock wastewater was banned in 2012. Livestock wastewater contains high concentrations of organic matter and nitrogen ions; therefore, advanced treatment processes are now required in order to ensure that such wastewaters meet water quality standards [24]. Presently, livestock wastewater is typically treated by aerobic or anaerobic digestion processes, and then the waste is recycled either as compost or in liquefied fertilizer [11]. However, organic wastes such as livestock wastewater have a high potential energy, and thus, these wastes can potentially be converted to more valuable bioenergy [2, 3, 27]. To produce this bioenergy, an anaerobic digestion process can be used, but the process is not easy to operate because the C/N ratio of livestock wastewater is low [10]. To alleviate this problem, food waste leachates can be mixed into the livestock wastewater to control the C/N ratio; however, in addition to the costs of generating and transporting the food waste leachates, this process can be problematic because noxious odors are generated. This type of process therefore requires a desulfurization system because of the highly corrosive hydrogen sulfide that is produced; without desulfurization, the hydrogen sulfide will contaminate the methane gas [12].

Microbial fuel cells (MFCs), which are actively being investigated for bioenergy applications, have the ability to produce electrical currents from a wide range of organic substrates. Some MFCs can even use inorganic matter and

toxic compounds as electron donors via microbial metabolic reactions [4, 6, 8, 16, 17, 19, 21, 23]. In the future, MFCs could be used to generate valuable electricity from impure or biodegradable organic matter, as well as assist in wastewater treatment, thereby decreasing the need for secondary treatment of useless products. This means that MFCs show great promise for providing new opportunities for sustainable energy production, and interest in them is growing. However, in spite of these positive aspects, the widespread application of MFCs is still constrained by some limiting factors [4, 8]. For example, when a MFC is operated with livestock wastewater, the high ammonium concentrations can adversely affect the microorganisms, and nitrate-nitrogen can reduce current generation because it serves as an electron acceptor. Therefore, it is very important to control the nitrogen ions for successful operation of a MFC. For this study, a device that generates microbubbles was employed to remove N from the livestock wastewater source.

Microbubbles are small bubbles with diameters in the range of 10–50 μm, and they have several useful characteristics such as a large gas-liquid interface area, long residence time in the liquid phase, and a fast dissolution rate; thus, they can be used to efficiently transfer oxygen from air into water [1, 25]. The use of microbubbles has been explored for various applications. In Japan, microbubbles have been employed in the treatment of wastewater and for industrial purposes [14, 18, 26]. Studies of microbubbles have largely focused on their potential to degrade toxic compounds, disinfect water, and clean or defoul solid surfaces (including membranes); microbubbles are useful in such applications because they produce free radicals that are extremely reactive and can break down wastes [1, 14, 18, 25, 26].

In addition, various oxidation treatments with catalysts have been introduced as advanced treatment methods. Lee *et al*. [15] converted ammonia in the aqueous phase to N_2, NO_2^-, and NO_3^- by manipulation of the pH, catalyst concentration, temperature, and reaction time while using Ru/TiO_2 as a catalyst. Lee [13] showed that humic acid and phenol in wastewater could be oxidized by an Fe/MgO catalyst, which is an abundant, low-cost, iron-based catalyst. The main principle of the catalyst process is that oxidation reactions initiated by OH radicals are promoted by the presence of the catalyst. Recently, the combination of microbubbles and a catalyst was investigated as an advanced method for the removal of nitrogen ions from livestock wastewater [9].

The purpose of this study was to investigate whether the ammonium- and nitrate-nitrogen contained in livestock wastewater could be reduced by treatment with microbubbles and an Fe/MgO catalyst, and to determine the effects of the pretreated livestock wastewater on current generation in MFCs.

Materials and Methods

Livestock Wastewater and the Pretreatment Reactor Using Microbubbles and a Catalyst

A cylindrical reactor was constructed for the removal of nitrogen ions such as ammonium and nitrate, following the application of microbubbles and/or a catalyst (Fe/MgO). Microbubbles were generated from a microbubble device, and the bubbles were allowed to flow into the catalyst embedded in the reactor during the combined treatments. The inner structure of the microbubble device was the spiral liquid type. The diameter of this reactor was 150 mm, and the void volume was 8.8 L. Here, three concentrations of catalyst were tested (100, 300, and 500 g of catalyst per liter of wastewater), and the catalyst was filled to an ~5 cm thickness above the bottom of the reactor. The livestock wastewater was first filtered by using a 250 μm sieve to remove large particles, and 4 L of the filtered material was used in each experiment. The livestock wastewater was introduced from a side hole in the reactor and then was circulated by a pump during the course of the reaction. During this time, air was injected into a tube installed before the pump. When the livestock wastewater was circulated, air was supplied at the rate of 300 ml/min through the use of a flow meter.

Microbial Fuel Cells and Their Operation

The MFCs were designed to obtain electricity from organic wastewater. The anode and cathode compartments (void volume of 50 ml each) were separated by a cation-exchange membrane (NE424; Nafion, Dupont Co., USA). Graphite felt (GF series; Electro-Synthesis Co., USA) was used as the electrodes, and platinum wires were used to connect the felt sheets to detect the resistance; the graphite felt (50 mm × 50 mm × 6 mm) was stocked in the amount of four sheets in the anode compartment and four sheets in the cathode compartment. The anode compartment was kept anoxic by nitrogen gas supplied at a constant flow rate through the use of a flow meter into the wastewater storage tank. The wastewater was fed into the biotic anode at the rate of 1.9 ± 0.1 ml/min. Air-saturated water (2.4 ± 0.1 ml/min) was fed into the compartment with the abiotic cathode in order to supply the oxygen required for the electrochemical reactions. All MFCs were operated while circulating the wastewater in the storage tank. The MFCs were operated at room temperature (25 ± 3°C). The resistance of 50 Ω was selected by use of a resistance box. Organic matter in the livestock wastewater was used as the electron donor, and its chemical oxygen demand ($COD_{chromate}$) was about 2,000 mg/l.

Analysis

Ammonium-nitrogen was measured by using a HACH kit

(Nessler method, 0.02–2.5 mg/l NH_3-N), and nitrate-nitrogen was measured by using the HACH kit Chromotropic Acid method, which can quantify concentrations in the range of 0.2–30.0 mg/l NO_3-N. All experiments were carried out in triplicates, and the figures show the mean values. The potential between the anode and cathode was measured by using a multimeter (Keithly Co., USA) hooked up to the platinum wires, and data were recorded every 5 min with a personal computer and a data acquisition system (Testpoint, Capital Equipment Co., USA). The measured potential was converted to current according to the following relationship: current = potential/resistance. Coulombs (expressed as current × time) was calculated by integrating the current over time.

Results

Effect of the Catalyst Amount on Ammonium- and Nitrate-Nitrogen Removal

These studies were conducted to determine the effect of the amount of catalyst on the reduction of nitrogen ions (*e.g.*, ammonium and nitrate ions). For these studies, the Fe/MgO catalyst was used at concentrations of 100, 300, and 500 g/l in the presence of microbubbles. The system was operated while feeding air in at a rate of 400 ml/min. The initial ammonium concentration was between 303.3 ± 7.3 and 340.0 ± 11.5 mg/l, and the initial nitrate concentration was 7.6 ± 0.5 to 9.0 ± 0.1 mg/l. When 100 g/l of catalyst was used, the ammonium- and nitrate-nitrogen concentrations were reduced by 14.3% and 66.8%, respectively, after a 2 h reaction time. When 300 or 500 g/l of catalyst was used, the ammonium was reduced from 335.0 ± 12.5 to 230.8 ± 5.2 mg/l and from 335.0 ± 12.5 to 225.1 ± 2.9 mg/l, respectively. Furthermore, the nitrate concentration was reduced from 7.6 ± 0.5 and 9.0 ± 0.1 mg/l to 2.0 ± 0.0 and 2.2 ± 0.0 mg/l, respectively, when 300 and 500 g/l of catalyst was used. At the same catalyst application rate, the removal efficiencies of ammonium- and nitrate-nitrogen were 31.1% and 32.8%, and 73.8% and 75.5%, respectively, in the 300 and 500 g/l treatments. When 500 g/l catalyst was used, the removal of ammonium- and nitrate-nitrogen was slightly higher than that at 300 g/l, but the difference was not significant. Therefore, it is preferable to use 300 g/l of catalyst in consideration of the economic costs and operational simplicity. Overall, the removal of ammonium- and nitrate-nitrogen increased with increasing amounts of catalyst, as shown in Fig. 1.

Effect of Oxidants on Ammonium- and Nitrate-Nitrogen Removal

These studies were performed to ascertain the effect of oxidants on the removal of ammonium- and nitrate-nitrogen.

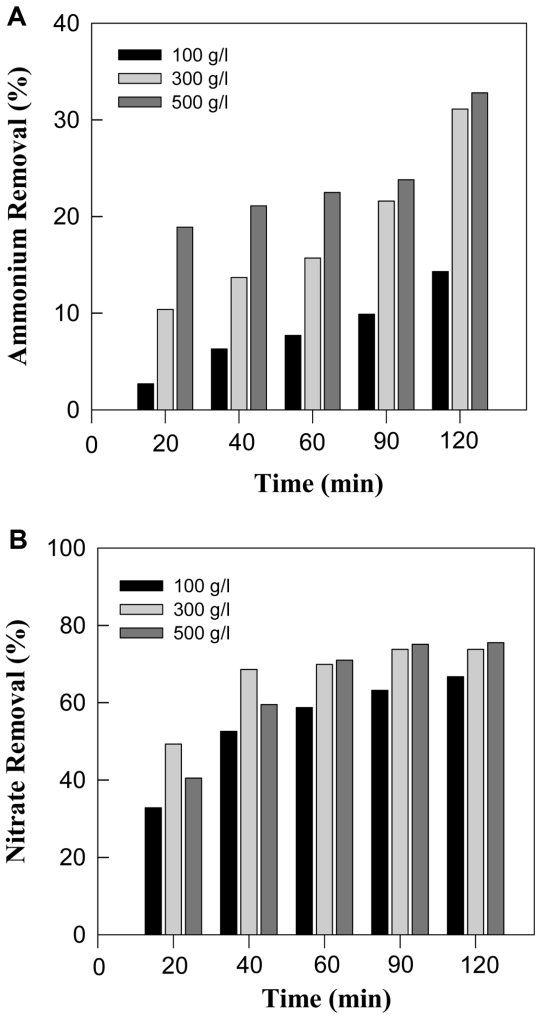

Fig. 1. Removal of ammonium- and nitrate-nitrogen with increasing amounts of catalyst.
(**A**) Ammonium removal (%); (**B**) nitrate removal (%).

The following three types of oxidants were used: air, oxygen (O_2), and hydrogen peroxide (H_2O_2). Oxygen at 400 ml/min served as the control, and these results were compared with those obtained with the other conditions. Specifically, oxygen was supplied at 400 or 1,000 ml/min for Condition 1, oxygen was supplied at 300 ml/min along with 2 ml of H_2O_2 per 4 L of waste for Condition 2, and air was supplied at 800 ml/min for Condition 3. When oxygen at 400 ml/min was fed into the reactor, the ammonium removal gradually increased to 31.1% from 335.0 ± 12.5 to 230.8 ± 5.2 mg/l. When oxygen was supplied at 1,000 ml/min, ammonium removal was two times higher than that for the other conditions in the early stages. The reaction rate seemed very fast at this time; however, the difference was not significant by the late stage. This value displayed a

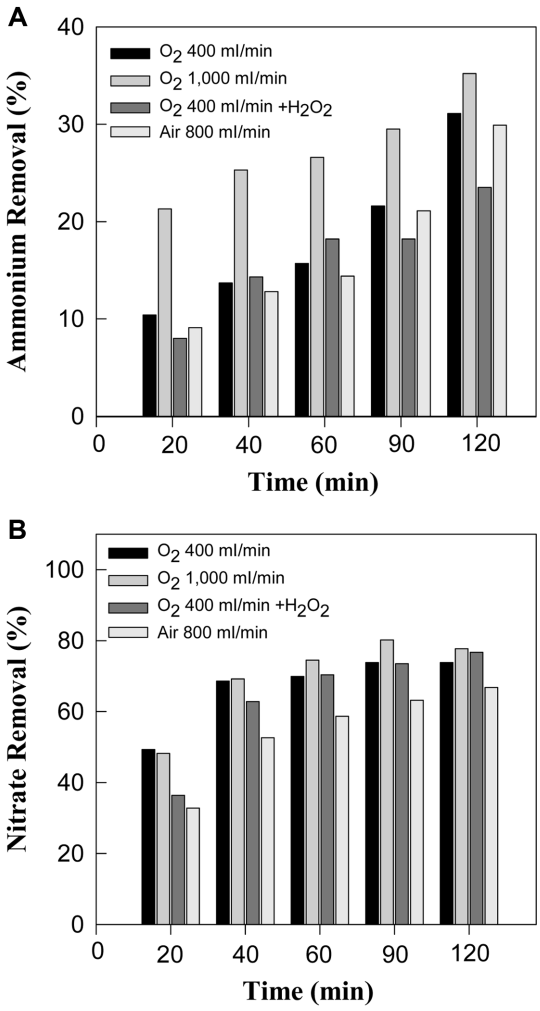

Fig. 2. Removal of ammonium- and nitrate-nitrogen following the addition of various oxidants at different feeding rates.
(**A**) Ammonium removal (%); (**B**) nitrate removal (%).

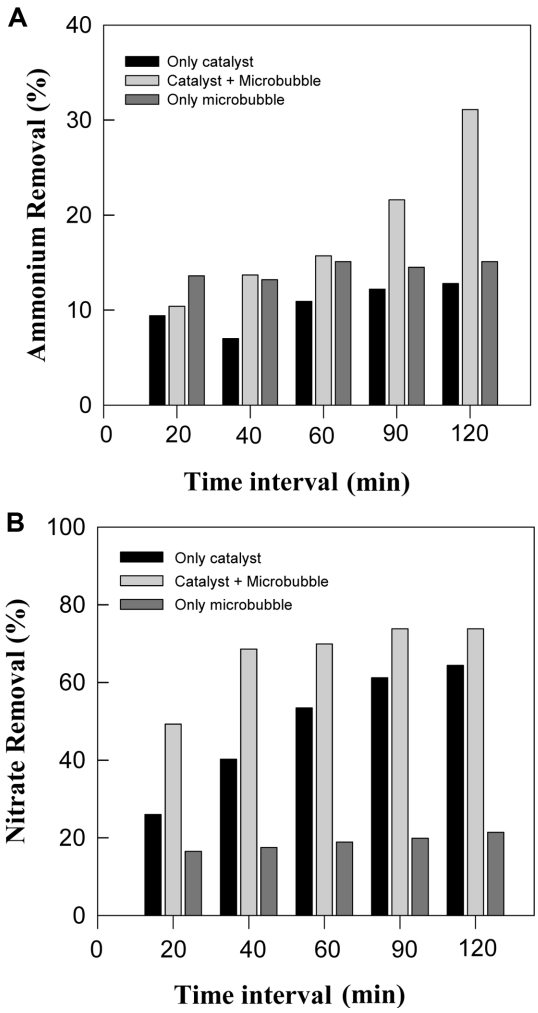

Fig. 3. Effect of the catalyst and/or microbubbles on the removal of ammonium and nitrate ions.
(**A**) Ammonium removal (%); (**B**) nitrate removal (%).

difference of about 12% compared with the control value at 400 ml O_2/min. However, the ammonium removal was the highest compared with any of the other conditions. As more gaseous oxygen was supplied, the removal of ammonium increased, as shown in Fig. 2. Air, instead of oxygen, was also tested to compare the results for ammonium removal. The ammonium removal that occurred with air supplied at 800 ml/min was similar to that for the treatment with oxygen gas supplied at 400 ml/min.

The nitrate-nitrogen removal showed no significant difference except for with the air supply. When oxygen was used, the ammonium removal showed a difference of less than 5% under all conditions, whereas the nitrate removal was up to 91.8–95.9% after a 2 h reaction time (except for when air was used). This result showed that the nitrate-nitrogen removal was almost saturated after 60 min under all conditions in which oxygen was used as the oxidant. When air was supplied at 800 ml/min, the nitrate-nitrogen removal reached 94.6% after 90 min. Because air was the source of the oxidant, the nitrate-nitrogen removal was found to be slightly lower than that obtained with the other oxidants.

Impact Evaluation of the Catalyst and/or Microbubbles

This experiment was conducted to learn about the effect of microbubbles and/or catalyst on the removal of ammonium- and nitrate-nitrogen. The following three sets of conditions were tested: 1, only Fe/MgO catalyst at 300 g/l in livestock wastewater; 2, Fe/MgO catalyst at 300 g/l in wastewater with the presence of microbubbles; and 3), only

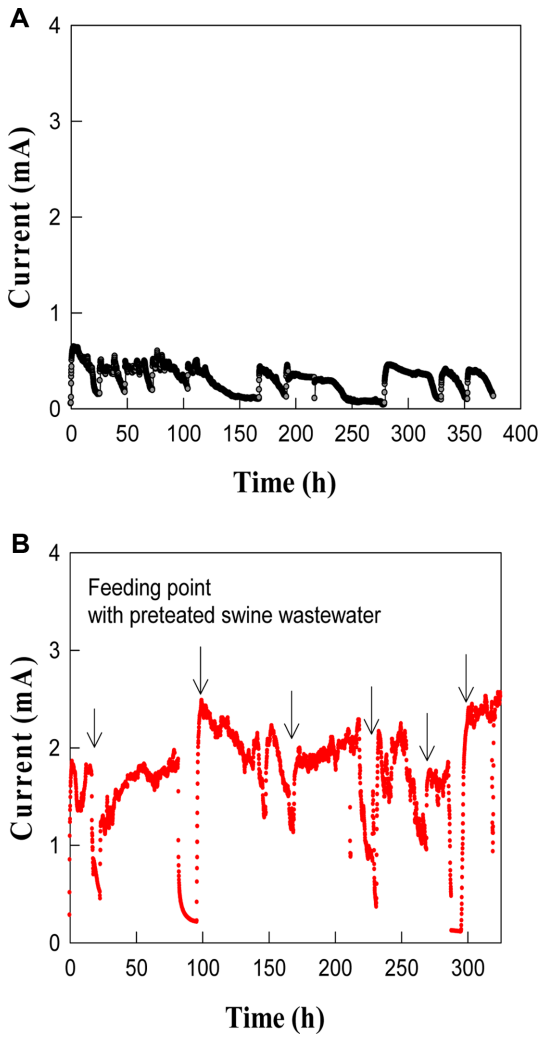

Fig. 4. Current generated by the use of livestock wastewater with and without pretreatment.

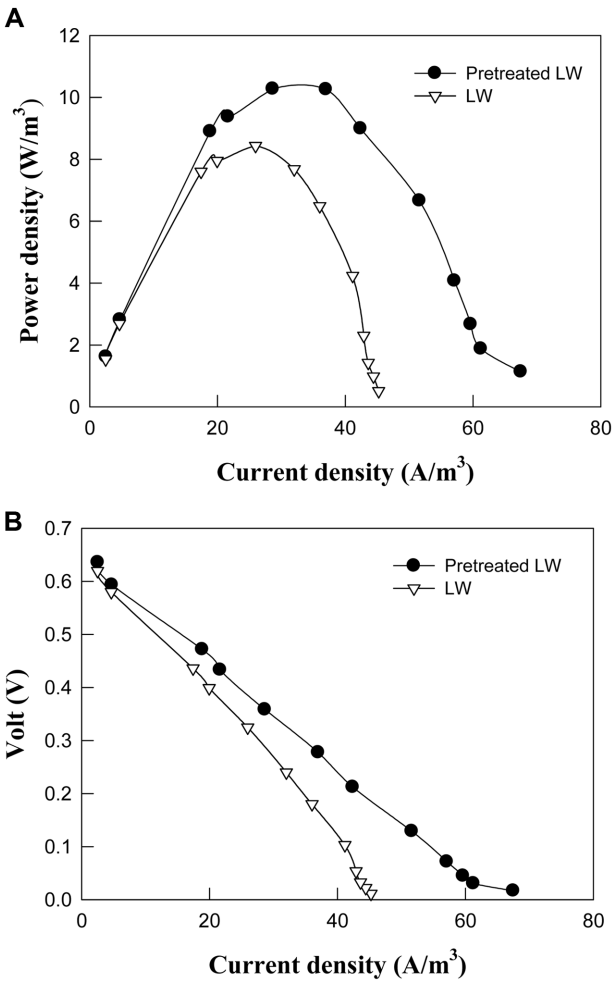

Fig. 5. Performance of the microbial fuel cell when using livestock wastewater with and without pretreatment.

with microbubbles. When using only the catalyst or microbubbles, the ammonium removal was 12.8% or 15.1%, respectively, after 120 min. The ammonium removal did not change with time under the conditions described above. However, when the microbubbles and the Fe/MgO catalyst were used at the same time, the ammonium removal increased up to 31.1%.

As shown in Fig. 3, when only microbubbles were used, the nitrate-nitrogen removal changed little over time to 21.4% from 16.5%. However, when catalyst only or the combined catalyst with microbubbles treatment was used, the nitrate-nitrogen removal increased to 64.4% and 73.8%, respectively. For both ammonium- and nitrate-nitrogen, the removal rate was highest when the catalyst and microbubbles were used together. This treatment seemed to create a synergistic effect.

Current Generation Using Pretreated Livestock Wastewater

These studies were conducted with pretreated livestock wastewater to determine how the decrease of ammonium- and nitrate-nitrogen affects electricity generation in MFCs. For this experiment, the current was measured in MFCs that were fed with either untreated livestock wastewater, which was merely filtered, or pretreated livestock wastewater. For the latter treatment, the ammonium- and nitrate-nitrogen were removed by applying microbubbles to the 300 g/l of catalyst embedded in the reactor. The livestock wastewater was fed into the MFCs after the pretreatment operation was run for 2 h. The ammonium- and nitrate-nitrogen of the livestock wastewater were decreased by about 50% and 76%, respectively, following

the pretreatment procedure. Fig. 4 shows a diagram of the current generation patterns that resulted from driving the MFCs with livestock wastewater that had been treated by the above methods.

A current of about 0.5 mA was generated when livestock wastewater was used without pretreatment. However, when pretreated livestock wastewater was supplied, the current increased to 2.14 ± 0.08 mA. This current was 4 times higher than that obtained when using untreated livestock wastewater. These data provide important evidence that ammonium and nitrate can act as inhibitors of current generation in MFCs. Therefore, to drive MFCs using livestock wastewater as fuel, it is important to remove these inhibitors.

Discussion

Effect of Livestock Wastewater Treated by Microbubbles and a Catalyst on Current Generation of a Microbial Fuel Cell

Livestock wastewater with high concentrations of ammonium- and nitrate-nitrogen was difficult to use in MFCs. In particular, the high concentrations of nitrogen ions, evaluated as NH_3^+ and NO_3^- in this study, had negative effects on the electrical current generation. Therefore, the livestock wastewater was treated by using a powerful oxidizer (*i.e.*, OH^- radicals) generated in the process of reactions induced by microbubbles and an Fe/MgO catalyst. Microbubbles are presumed to be advantageous for wastewater treatment because they can enhance the mass transfer of oxygen, which promotes oxidation reactions. Moreover, the dissolved oxygen concentrations can be maintained at high levels because microbubbles in the size range of 10–50 μm remain suspended for long periods of time in water. The catalyst also produced OH^- radicals during the reaction process, which helped to oxidize ammonium and nitrate. The nitrate concentration was not as high as the ammonium concentration in the livestock wastewater. However, the current can be critically affected at >60 mg/l nitrate-nitrogen concentrations, as shown by Jang *et al.* [7]; moreover, they found that nitrate at 123.3 ± 0.1 mg/l will inhibit the current generation severely (*i.e.*, the current was reduced from 9.08 ± 0.11 mA in the control treatment to 0.16 ± 0.02 mA in the nitrate treatment). Because nitrate can act as an electron acceptor, nitrate levels should be controlled before using wastewater in MFCs [7]. In the case of ammonium, some researchers have published that it can be used by microorganisms in a MFC [5]. He *et al.* [5] actually showed that the current can increase with increasing amounts of ammonium. However, Jang *et al.* [7] showed that >100 mg/l ammonium can negatively influence the microorganisms in a MFC [7]; additionally, Nam *et al.* [20] reported that a MFC was significantly inhibited by ammonium at >500 mg/l. Rajagopal *et al.* [22] also observed inhibition of anaerobic digestion by excess ammonia. The process of anaerobic digestion becomes unstable at higher pH values, which allows for the rapid conversion of ionized ammonium-nitrogen into free ammonia-nitrogen (FAN), as shown in Eq. (1) [22]:

$$NH_4^+ + OH^- \leftrightarrow NH_3 + H_2O \quad (1)$$

At high pH values, the un-ionized form (*i.e.*, free ammonia) dominates, and this form is more inhibiting than is the ammonium ion (NH_4^+) [22]. Therefore, it is necessary to apply appropriate control strategies for ammonia as well, because it plays a vital role in the MFC.

Performance Evaluation of the Microbial Fuel Cell Using Pretreated Livestock Wastewater

To determine the potential power density, this test was conducted with a 50 mm × 50 mm MFC and a working volume of 50 ml. Fig. 5 indicates the power density and current density generated from the MFC. In this study, the MFC was fed with 300 mg/l livestock wastewater at a rate of 0.83 ml/min. The resistance was randomly changed from 10 to 10,000 Ω, and the voltage was measured at an open circuit. The maximum power density of the livestock wastewater with and without pretreatment was 0.75 and 0.82 V, respectively. This open circuit voltage is similar to the values that can be generated in a MFC when using artificial wastewater or sewage. The maximum power density and current density generated from the MFC using pretreated livestock wastewater were 10.3 W/m^3 and 67.5 A/m^3, respectively. These values were 17.9–33.0% higher than those obtained when using untreated livestock wastewater, and both showed a maximum power density of 300 Ω. These results indicate that the current generation was improved by controlling the nitrogen-containing ions. In order to drive the effective operation of a MFC using livestock wastewater, it was confirmed that nitrogen-containing ions in the livestock wastewater must be controlled. The likely reason is that high levels of ammonium-nitrogen can inhibit microorganisms, and the nitrate-nitrogen can act as electron acceptors in the anode cell.

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