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Influence of applied voltage and COD on the bioelectrochemical degradation of organic matter

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ABSTRACT

Organic matter degradation was examined in an air cathode bioelectrochemical system (BES) operated in a fed-batch mode operation. The organic matter degradation rate in the air cathode BES was significantly affected by the initial concentration of organic matter with a chemical oxygen demand (COD) ranging from 25 to 600 mg/L, and by the applied voltage of 100 and 300 mV. When the initial COD concentration was less than 100 mg/L, the substrate affinity to bacteria at an applied voltage of 300 mV was slightly higher than that at 100 mV, but the substrate degradation potential that was evaluated by the ratio of maximum substrate degradation rate to saturation constant did not vary at 100 and 300 mV applied voltages. For initial COD concentrations higher than 300 mg/L, the substrate degradation potential at an applied voltage of 100 mV was higher than that at 300 mV. The anode and cathode potentials in the BES were affected by both the applied voltage and initial COD concentration, but the effect of applied voltage on the potential was increasingly important with decrease in COD concentration. The coulomb efficiency at an applied voltage of 100 mV was higher than that at 300 mV, but decreased with increase in initial COD concentration in the BES. The air cathode BES with applied voltage is an advanced wastewater treatment system for organic pollutants.

Keywords: Bioelectrochemical system; Applied voltage; Air cathode; Organic matter

1. Introduction

Aerobic biological treatment based on the activated sludge process has been a common method for organic wastewater treatment. The activated sludge process is a technology that converts organic matter into biomass using assimilation of aerobic bacteria and separates the biomass from clean water [1]. The aerobic biological treatment process, however, requires a large amount of energy to maintain aerobic condition

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in the aeration tank, and entails additional outlays for the large excess of biological sludge [2,3]. The overall performance of the biological treatment process is determined by the operating expenses for the sludge treatment and aeration, the microbial degradation rate of organic pollutant, and the effluent quality. Anaerobic digestion is a good way to reduce the excess sludge production and the operating cost of the wastewater treatment plant. Traditional anaerobic digestion, however, has some disadvantages including low growth rate of anaerobic bacteria, heating requirement, and poor effluent quality [1,4]. The substrate degradation rate in the biological treatment process is generally affected by the amount of bacteria, mass transfer in the system, substrate degradation kinetics, and organic loading rate [5]. Among them, the substrate degradation kinetics is very dependent on the bacterial characteristics. The bacteria in the concerning aerobic biological treatment of wastewater is chemoheterotrophs, and the bacterial assimilation is coupled with the catabolism to obtain energy for the bacterial growth through the degradation of some organic matter into water and carbon dioxide [6–8]. This implies that the substrate degradation rate of bacteria is closely connected with bacterial growth [6,7]. The impetus for the biological degradation of organic matter is the energy available from the difference in the energy carried by the organic matter with that of the products [8–12]. In the case of acetate as an example substrate, the theoretical reduction potential of the anodic half reaction producing a proton, an electron, and carbon dioxide is −0.296 V against the normal hydrogen electrode (NHE) for a typical condition (HCO_3^- = 5 mM, CH_3COO^- = 5 mM, pH 7, $T = 275$ K), and the reduction potential from an electron, a proton, and oxygen to form water is 0.805 V against the NHE ($PO_2 = 0.2$, pH 7) [3]. The overall reduction potential for the aerobic bacteria is theoretically 1.101 V against the NHE. The aerobic bacteria obtain the energy from the degradation of organic matter and use the energy for new cell synthesis and maintenance [6–8]. The growth yield of bacteria, then, differs according to the species of bacteria and substrate types, and the growth yield for some bacteria is uncoupled to the substrate degradation [3,8,13,14]. A good uncoupling illustration between the substrate degradation and the bacterial growth is the electrochemically active micro-organisms (EAB) in bioelectrochemical systems (BESs), such as microbial fuel cells (MFCs) or microbial electrolysis cell. In MFCs, the bacteria utilize a small part of the energy carried by organic matter for their growth, while a large part of the energy is converted to electricity [3,8]. The excess sludge produced from BESs for wastewater treatment is smaller than that from the aerobic biological process [3,5,8]. In BESs, a half reaction that degrades the organic matter is catalyzed by the EAB on the anode surface, and the other half reaction that disposes off the electrons is completed on the separated cathode [3,5,8]. In the BES, the potential applied using an external power supply may have some influence on the organic matter degradation [9,11,12,14]. Recently, the basic principles of the voltage applied to the BES have been transferred to hydrogen production from the anaerobic cathode [2,10,12]. However, little research has examined the organic matter degradation in an air cathode BES as a function of the voltage applied to the BESs. In this study, the effect of applied voltage on the organic matter degradation in an air cathode BES was investigated under different organic concentrations. Two different voltages (100 and 300 mV) were applied to the air cathode BES in which the initial chemical oxygen demand (COD) ranged from 25 to 600 mg/L. The effect of the applied voltage on the COD degradation kinetics, and the factors affecting the COD degradation and coulomb efficiency were investigated.

2. Materials and methods

2.1. Bioelectrochemical system

A cube-type batch bioelectrochemical reactor (9 cm × 9 cm × 12.4 cm) with an effective volume of 1.0 L was prepared (Fig. 1). An air-breathable cathode was installed in the square window (4 cm × 4 cm) on a vertical wall of the cube-type reactor, and a separator and an anode were fitted on the inside of the cathode. The anode was a graphite fiber fabric (Samjung C&G Co., Daegue, Korea) which was fixed on the current collector

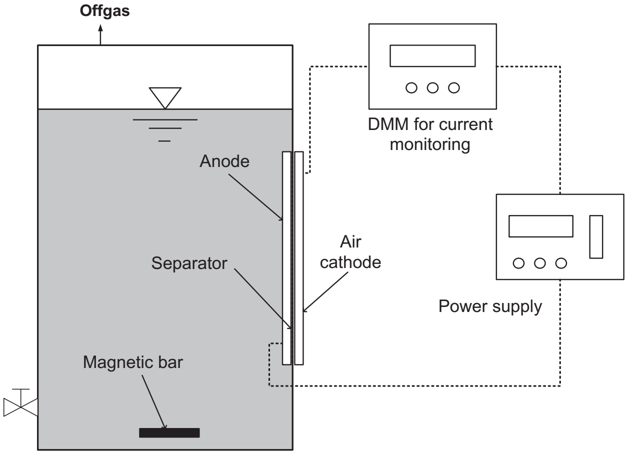

Fig. 1. A schematic of the air cathode BES used for organic matter degradation.

(stainless steel mesh, #30, STS 316L, 4 cm × 4 cm, a local hardware store) by sewing with a nylon thread. The separator was a non-woven polypropylene cloth sourced from a local hardware store. The cathode was a multiwall carbon nanotube (MWCNT) electrode (Carbon Nano-material Technology Co., Ltd, Pohang, Korea) loaded with multi metals (Fe, Cu, Mn) as the oxygen-reduction catalyst. Briefly, for the cathode preparation, a pristine MWCNT was immersed in nitric acid solution for 24 h to remove any impurities, washed with running tap water to neutralize, and dried in a drying oven. The prepared MWCNT (70% dry wt.) was mixed with polytetrafluorethylene (PTFE) solution (30% dry wt., dispersion (60% wt.) in water, Sigma–Aldrich Korea Co., Kyeonggi, Korea) as a hydrophobic binder, and the mixture (0.04 g wt.) of MWCNT and PTFE was screen printed on the current collector (stainless steel mesh, #30, STS 316L, 4 cm × 4 cm) to form a hydrophobic MWCNT layer. A primitive electrode was prepared by brushing a PTFE solution on the surface of the hydrophobic MWCNT layer three times, and then hot pressing at 370˚C for 20 min to form a waterproof and air permeable layer using a hot presser (SH-498D, Sahok Co., China). On another side of the primitive electrode, a multi-metal catalyst fixed on the MWCNT was brushed using Nafion solution as a binder. For the multi-metal catalyst, a multi-metal solution was prepared by dissolving sodium dodecyl sulfate 12.5 g, $MnSO_4 \cdot H_2O$ 30.125 g, $KMnO_4$ 19.75 g, FePc 0.5684 g, CuPc 0.5761 g, and MWCNT 1.875 g in 1.0 L of distilled water according to a previous study [15]. The solution was autoclaved at 135˚C for 4.5 h, and then after three washes with ethanol solution and centrifugation at 2000 rpm for 10 min, a catalyst mixture consisting of MWCNT and the multi-metals was obtained. The current collectors for the anode and the cathode were connected with a copper wire to make an electric circuit in which a power supply (OPM series, ODA Technologies, Incheon, Korea) was installed to apply the voltage between the electrodes. The anode chamber of the air cathode BES was filled with synthetic wastewater and a digital multimeter (DMM; Keithley Inc., 2700 Multimeter, Ohio, USA) connected with a personal computer was installed into the circuit to monitor the current.

2.2. Start-up, operation, and monitoring

Anaerobic sludge was taken from a sewage sludge digester (B metro-city, Korea) and screened for seed sludge. The initial characteristics of the seed sludge were 4.2 g/L of volatile suspended solids and pH of 7.12. A sample of synthetic wastewater containing phosphate buffer (50 mM), mineral solution (12.5 mL/L), and vitamins (5 mL/L) was prepared according to the previous study [9], and the COD of the wastewater was controlled within the range of 25–600 mg/L using sodium acetate. For the start-up, the seed sludge (270 mL), amounting to 30%v of the effective volume of the BES, was added to two air cathode BESs and the synthetic wastewater (630 mL) of 600 mg COD/L was also filled into each air cathode BES. In order to form an EAB biofilm on the surface of the anode, an external resistance of 200Ω was connected in the electric circuit, and the air cathode BES was placed in a thermostat room (30˚C). The solution in the anode chamber was mixed with a magnetic bar (200 rpm) during operation and the voltage of the air cathode BES was monitored with the DMM (Keithley 2700, Keithley Instruments Inc., Ohio, USA), connected with a personal computer. As the voltage was decreased below 10 mV, the anode solution was replaced with fresh synthetic wastewater (600 mg COD/L). After three cycle experiments with fed-batch mode, the EAB biofilm was considered to have been well formed on the anode surface from the consistent voltage. Then, the two experimental voltages (100 mV, 300 mV) were applied to the two different air cathode BESs using an external power supply and the COD degradations over the experimental COD range were investigated. During the operation of the air cathode BESs, the currents were monitored at 15 min intervals using the DMM, and the potentials of the anode and the cathode were intermittently measured using Compact Stat (Ivium Technologies B.V., Eindhoven, The Netherlands). Ag/AgCl electrode (CH Instruments Inc, Austin, the USA) was used as the reference electrode. The anode solution was intermittently taken and analyzed for the COD, according to the Standard method [16]. The pH of the anode solution was measured with a pH meter (Orion 370, Thermo Fisher Scientific, MA, USA).

2.3. Calculation

The COD change of the anode solution in the air cathode BES was described according to the following saturation kinetics (Eq. (1)).

$$\frac{\mathrm{d}S}{\mathrm{d}t} = -\frac{K_x S}{K_s + S} \tag{1}$$

where S is the COD (mg/L), t the hour (h), K_s the saturation constant (mg/L), and K_x the maximum COD degradation rate (mg/L/h). The saturation kinetics was integrated to obtain Eq. (2).

$$t = (K_s/K_x)\,\log(S_0/S) + (S_0 - S)/K_x \tag{2}$$

The COD data from the air cathode BES were fitted into the integrated form (Eq. (2)) of the saturation kinetics (Eq. (1)) by a nonlinear regression to obtain the kinetic constants (K_s, K_x). The nonlinear regression was performed using the curve fitting toolbox of Matlab (R2010, The Mathworks, Inc., MA, USA). During COD degradation in the air cathode BES, the measured electric charge was estimated by integrating the current over time, and the coulomb efficiency based on the removed COD was calculated using Eq. (3).

$$E_c(\%) = M \int_0^{t_c} \mathrm{Idt}/nF\,V_{\mathrm{an}}\ \Delta\mathrm{COD} \tag{3}$$

where M is the molecular weight of oxygen (32), F is the Faraday's constant, n is the number of electrons exchanged per mole of oxygen (4), V_{an} is the volume of liquid in the anode chamber, and ΔCOD is the change in COD over the cycle time (t_c).

3. Results and discussions

3.1. Degradation of organic matter in the air cathode BES

The substrate degradation rate in a biological system is affected by several factors including substrate type, bacterial activity, and environmental conditions. In particular, the reduction potential difference between electron donor and electron acceptor determines the available energy for bacterial growth and maintenance according to the equation $\Delta G = -n \times F \times \Delta E$, where $\Delta E = E'_{\mathrm{donor}} - E'_{\mathrm{acceptor}}$ [9,11,14,17]. This indicates that the applied voltage in BES can affect the bacterial activity that contributes to the degradation of substrate [9,11,14,15]. In this study, the concentration of COD in the batch BES gradually decreased with increase in incubation time (Fig. 2). The degradation rate of COD was higher at the initial stage of incubation and it decreased with decrease in COD concentration over time. However, at an applied voltage of 100 mV, the degradation rate of COD was different from that at an applied voltage of 300 mV. These indicate that the organic matter degradation in the air cathode BES is affected by the applied voltage as well as the organic matter concentration.

The behavior of bacterial growth kinetic in a batch culture depends on the ratio of the substrate concentration to the biomass concentration that reflects the food availability normalized to unit biomass [18]. The degradation of organic matter in the BES was assumed to be described by the saturation kinetic using Eq. (1), and the kinetic constants were obtained by a nonlinear regression (Table 1). The correlation coefficients (r^2) were over 0.99 for all COD data. Overall, K_s and K_x were increased with increase in initial COD concentration. However, when the initial COD was ranged between 25 and 100 mg/L, the values of K_s and the BES at an applied voltage of 300 mV were slightly lower than those at an applied voltage of 100 mV, while the values in the initial COD range of 300–600 mg/L exhibited an opposite trend. In a biological system, a lower K_s implies better substrate affinity with micro-organisms, but it is not constant for a wide range of bacterial growth conditions and it is closely related with the initial substrate concentration in batch reactor [1,19]. The K_x is the product of maximum specific substrate degradation rate and bacteria cell concentration, which is a kind of potential for substrate degradation in BES.

Table 1
Maximum substrate degradation rate and saturation constant in the air cathode BES with initial COD concentration at an applied voltage of 100 and 300 mV

Voltage (mV)	COD (mg/L)	K_s (g/m^3)	K_x(g/m^3 h)	R-square
100	600	102.1	18.29	0.9987
100	300	85.15	13.91	0.9968
100	100	78.54	9.154	0.9964
100	50	57.08	7.177	0.9995
100	25	33.65	4.513	0.9999
300	600	223.1	23.72	0.9911
300	300	190	16.89	0.9974
300	100	62.85	7.723	0.9972
300	50	39.06	4.648	0.9978
300	25	24.23	4.412	0.9999

Unfortunately, information on the concentration of bacterial cell is not available in this BES study. However, if it is assumed that the substrate availability affects the bacterial growth kinetic, the value of K_x/K_s can be used as an index to evaluate the degradation potential of organic matter in BES. As shown in Fig. 3(B), for an initial COD concentration less than 100 mg/L, the difference between K_x/K_s values obtained at applied voltages of 100 and 300 mV is unclear. However, for the initial COD of 300–600 mg/L, the K_x/K_s values at an applied voltage of 300 mV were 0.09–0.11, which is smaller than 0.16–0.18 at an applied voltage of 100 mV. Above results indicate that organic matter could be effectively degraded using the air cathode BES applied voltage of 100 mV.

3.2. Factors affecting on the COD degradation under the applied voltage

The degradation reaction of organic matter at the air cathode in a BES comprises two half reactions, an anodic half and a cathodic half. The anodic half reaction produces electrons, protons, carbon dioxides, and new cells by EAB and the cathodic half reaction forms water molecules from the electrons, protons produced by the anodic half reaction and oxygen from air. The anodic half reaction can be continuous if the electrons produced from organic matter degradation are transferred to the anode and flow to the cathode where they get disposed effectively forming water. The transfer of electrons to the anode is affected by the anode potential and the electron disposal at the cathode is

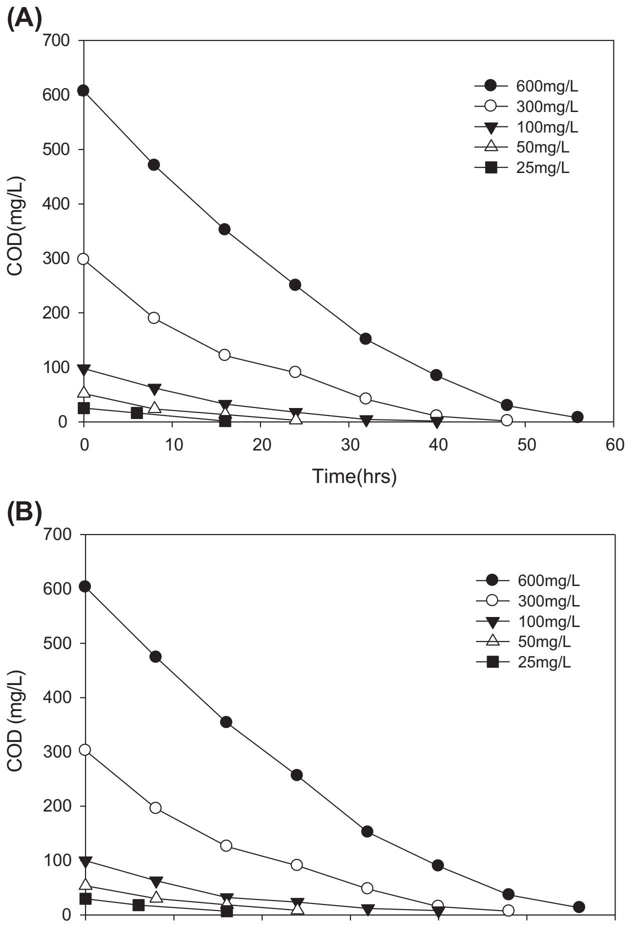

Fig. 2. Degradation of organic matter in the air cathode BES with initial COD concentration at an applied voltage of 100 (A) and 300 mV (B).

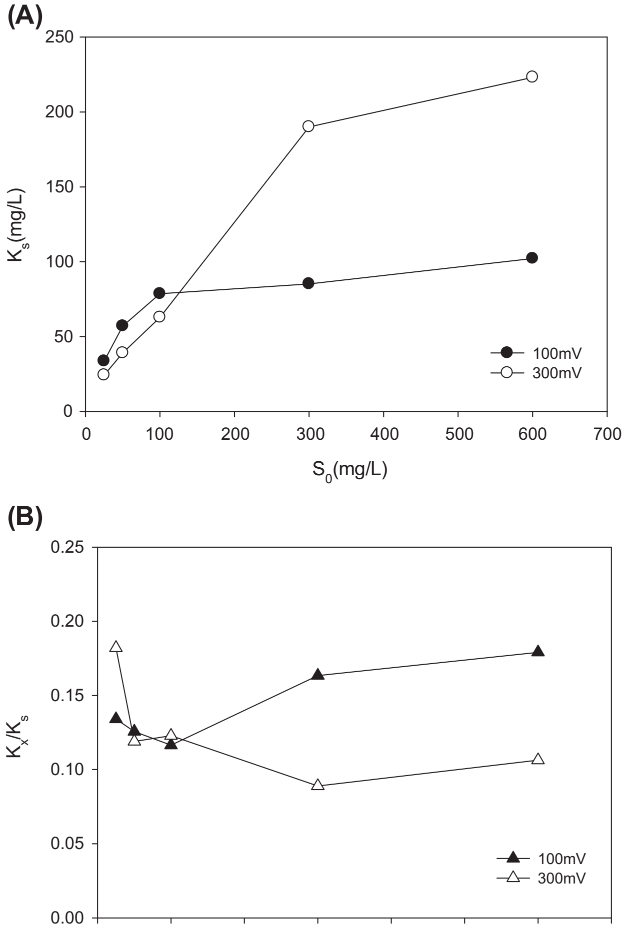

Fig. 3. Ratio of maximum substrate degradation rate (K_x) to saturation constant (K_s) according to the initial COD concentration (S_0).

affected by the cathode potential indicating that the degradation reaction of organic matter is governed by the potentials of the anode and the cathode. The protons have some influence on the microbial activity for the organic matter degradation and on the electron disposal at the cathode.

The potential changes of the anode and the cathode are shown in Fig. 4. When a voltage of 100 mV was applied to the BES, the initial potentials of the anode were in the range of −149 to −244 mV, which had a lower value at a higher initial COD (Fig. 4(A)). The theoretical thermodynamic potential of anode when acetate is substrate is about −500 mV vs. Ag/AgCl reference electrode [3]. The positive anode potentials, compared to the theoretical value, were attributed to the influence of the applied voltage difference. An EAB at higher COD concentration can produce more electrons which can be supplied to the anode. This indicates that the initial COD significantly influences the anode potential of BES. The cathode potentials ranged from −184 mV to −152 mV. The cathode potentials were more negative at higher initial COD concentration due to the disposal of more electrons at the cathode. The anode and cathode potentials gradually increased with increase in incubation time. The increase of anode potential was at a slightly faster rate than that of the cathode. At the end of incubation time, the anode potential was higher than that of cathode due to the shortage of remaining COD. It seems that the potential of the BES is governed more by the applied voltage than by the electrons produced by EAB if the available organic matter in BES is limited.

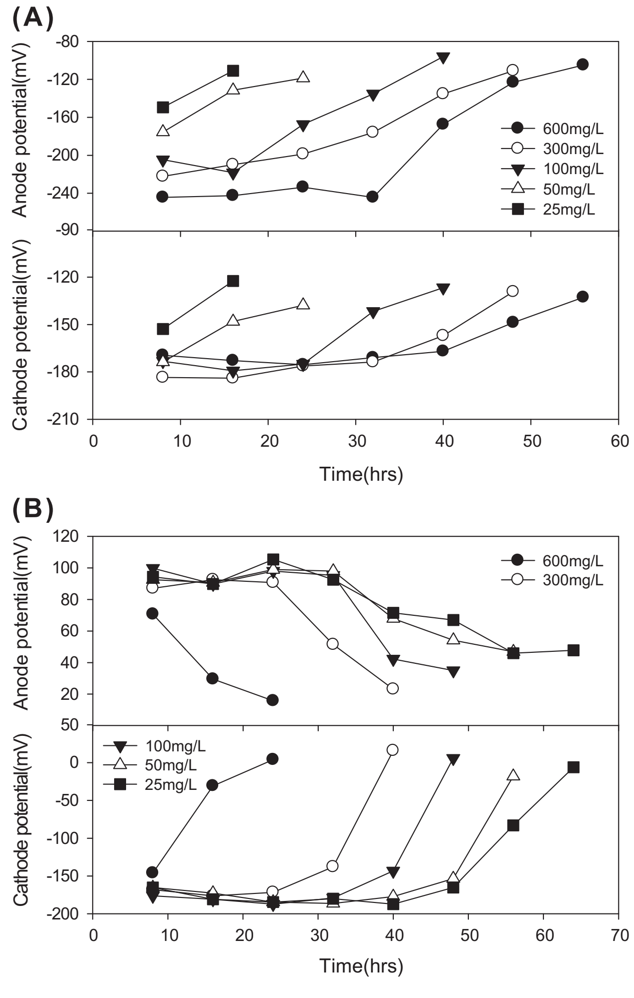

Fig. 4. Potential changes of anode and cathode in the air cathode BES with initial COD concentration at an applied voltage of 100 (A) and 300 mV (B).

At an applied voltage of 300 mV, the anode and cathode potentials were significantly different from those at 100 mV (Fig. 4(B)). The initial anode potentials ranged from 70 to 100 mV, and were affected by the initial COD concentration. On the other hand, the cathode potentials in the initial stage ranged from −146 to −176 mV. After incubation of the BES, the cathode potential remained constant for a certain period of time, and then quickly increased. The increase of cathode potential was faster at a lower initial COD concentration than at a higher COD concentration. On the contrary to the cathode potential, the anode potential remained constant for a certain period of time and then slightly decreased. It is possibly that the constant anode and cathode potentials indicate the equilibrium state between electron transfer from organic matter and the disposal at the cathode. At the end of the incubation time, the increase of cathode potential indicates that the reduction reaction for electron disposal was faster than the electron supply from anode. When the organic matter was depleted at the end of the BES incubation, the anode and cathode potentials were constant at equilibrium again. The equilibrium potential, however, was different from the applied voltage because of some losses such as current leakage.

The protons were a product of the anodic reaction. The trends of pH according to the incubation time give important information for understanding the degradation of organic matter in the BES. At an applied voltage of 100 mV, the pH slightly decreased with increase in incubation time, and then increased to surpass the initial pH value (Fig. 5). At higher initial CODs, the pH change was larger and it took longer to reach the final pH. The pH decrease is attributed to higher rate of proton production from the anodic organic matter degradation compared to the rate of disposal on the cathode. When 300 mV was applied to the BES, the pH trends were similar to those at 100 mV, but the change in pH was larger than that at

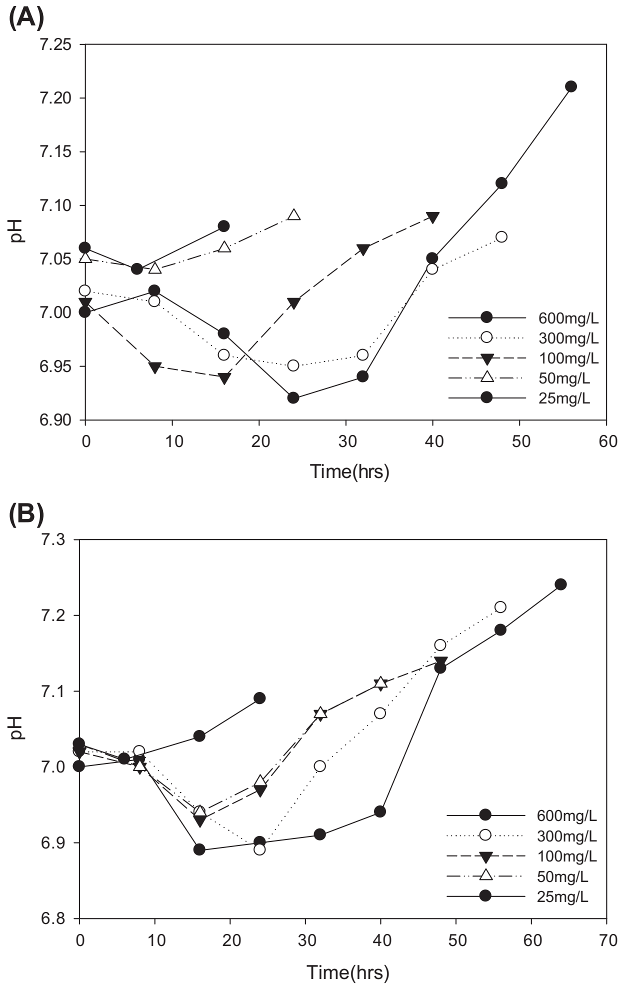

Fig. 5. pH changes in the air cathode BES with initial COD concentration at an applied voltage of 100 (A) and 300 mV (B).

100 mV. This is a further proof that the anode and cathode potentials were significantly involved in both the electrons transfer at the anode and the disposal of electrons and protons at the cathode.

The data for the measured electric charge in BES and the theoretical electric charge give some important information about the organic matter degradation [3,8]. At an applied voltage of 300 mV, the measured electric charge at higher initial COD (300–600 mg/L) was substantially smaller than the theoretical value. However, the differences between the measured electric charge and the theoretical value decreased with decreasing initial COD. In BES, micro-organisms convert a portion of organic matter into electrons and a new cell, while rest of the organic matter in the BES disappears as unknown losses such as cellular maintenance energy for micro-organism, and heat [3,8]. It is possible that a considerable portion of the difference between the measured and theoretical electric charges was a result of bacterial growth, which decreased with decreasing initial COD.

At an applied voltage of 100 mV, more electric charges were measured compared to those at the applied voltage of 300 mV. This indicates that the losses at 100 mV is smaller than that at 300 mV (Fig. 6). The electric charge values at lower initial COD (25–100 mg/L) were more than their theoretical values, indicating some leakages of current in BES. In earlier studies, the electrode potentials in a BES affected the growth and activity of microbes [9,11,12,14]. These results suggest that the bacterial growth and activity for organic matter degradation depend on the initial COD (concentration) and the applied voltage.

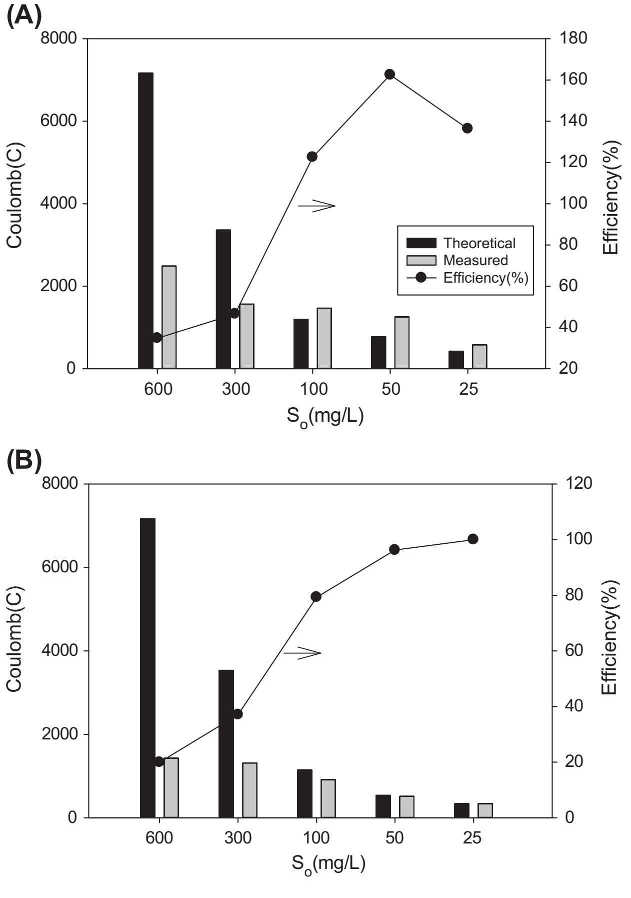

Fig. 6. Comparison of theoretical and measured coulomb in air cathode BES with initial COD concentration at an applied voltage of 100 (A) and 300 mV (B).

4. Conclusions

The bioelectrochemical degradation of organic matter was examined in an air cathode BES operated in a fed-batch mode and the following conclusions were drawn. The organic matter degradation rate is significantly affected by the organic matter concentration and the voltage applied to BES. The effective voltage applied to BES for organic matter degradation depends on the organic matter concentration. In particular, for initial COD concentrations higher than 300 mg/L, the substrate degradation potential at 100 mV is higher than that at 300 mV. The anode and cathode potentials in a BES are affected by both an applied voltage and organic matter concentration. The coulomb efficiency is smaller at higher organic matter concentration in the BES, but at an applied voltage of 100 mV, it is higher than that at 300 mV. The air cathode BES with applied voltage is an advanced wastewater treatment system for organic pollutants.

Acknowledgments

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