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# Simultaneous removal of odourous and organic compounds in septic tanks using an electrolytic oxidation system

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ABSTRACT

Odour, which is emitted from septic tanks treating excrement and manure, has become public concerns. Reduced sulphur compounds, such as hydrogen sulphide, are the main sources of the malodour, and readily degradable organics play an important role in the production of the reduced sulphur compounds under anaerobic conditions. In this study, an electrolytic batch reactor was applied to reduce both organics and odourous sulphur compounds in excrement slurry. In the reactor, direct currents at fixed voltages of 10, 20 and 30 V were applied to a Ti/ $IrO_2$ anode and a stainless steel cathode, both submerged into the slurry. The predominant chemical reaction at the surface of the anode was the production of HOCl, resulting in the simultaneous oxidation of the odourous and organic compounds and the increase in oxidation/reduction potential. At the applied voltages greater than 20 V, the concentration of $H_2S$ in the headspace of the reactor exponentially dropped within initial 30 min period. In addition, the chemical oxygen demand (COD) in the slurry was gradually declined at all the voltage conditions, and a rapid removal of the COD was observed only when the fixed voltage of 30 V and corresponding current density of 150 A/$m^2$ was applied. Energy consumptions expressed as kWh/kg-$H_2S$ and kWh/kg-COD varied greatly, and they need to be experimentally evaluated depending on wastewater characteristics. Consequently, the electrolytic oxidation can be applied to effectively control odours emitted from the excrement slurry, and an appropriate current density and energy consumption as a threshold value should be selected to optimize the system for the simultaneous removals of organics and reduced sulphur compounds.

*Keywords:* Electrolytic oxidation; Odour; Hydrogen sulphide; Septic tank; Excrement

## 1. Introduction

Public complaints and concerns about odours emitted from sewer systems are continuously increasing, and the odour problem has become an important environmental issue in the vicinity of sewage treatment works. In particular, the odour emissions from combined sewer facilities, such as manholes, pump stations, and septic tanks, in urban areas can cause

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unpleasantness to general publics as an aesthetic pollution [1]. Sediment organic matters deposited at the bottom of the sewer facilities are known to be the precursors of malodourous compounds when the oxidation/reduction potential (ORP) drops and anaerobic conditions are formed [1]. Especially, in septic tanks, where high-density organic solid matters from toilets and kitchens are introduced, odourous compounds are produced mainly by microbial-mediated decomposition of organic compounds in the anaerobic condition [2,3].

The quantity and quality of odour emissions from septic tanks treating excrement and manure are affected by many factors such as temperature and solid retention time and also greatly influenced by the amounts of odour precursors and organic matters introduced to the facilities as well as the oxidation state of the sediment. The odourous compounds generated from the septic tanks are largely classified into volatile sulphur compounds, volatile nitrogenous compounds, and volatile fatty acids. The volatile sulphur compounds in reduced states are known to be the major odourous compounds of the sewer and septic tank emissions [3]. Sulphate ($SO_4^{2-}$) ions contained in the slurry and sediment can be reduced under low ORP conditions to hydrogen sulphide ($H_2S$) and sulphur-containing organic compounds such as methyl mercaptan [1,4]. These reduced sulphur compounds cause highly intensive odours even at very low concentrations. The odour problems from these small-scale area sources are not easy to handle because not only the odour sources are scattered in many metropolitan areas but also the odour concentration and flux vary greatly depending on seasonal and climatic conditions.

Various odour treatment methods, including physical and chemical processes, have been applied to control the emissions of hydrogen sulphide and other odourous compounds [4–7]. Physical processes such as dilution, absorption and adsorption are mostly applied to gas streams where both flow and odour concentrations are high; however, these physical processes are generally infeasible since odour emission rates from the combined sewer septic tanks (i.e. small-scale area sources) are low and fluctuated substantially. Chemical processes, such as spraying deodorants and oxidants (e.g. $H_2O_2$, NaOCl, $O_3$), are practicable applications for the small-scale odour sources, but they have many drawbacks such as by-product formation, expensive chemical usages, maintenance difficulties and short duration. Consequently, there is strong necessity to develop a new method that is easy to install and operate as well as that achieves continuous and reliable reductions for the odour emissions.

To reduce the odour emissions from septic tanks treating excrements, an electrochemical oxidation process using electrolysis has been proposed [8]. In the electrochemical oxidation reactor, a direct current (DC) is applied by using a power supply to electrodes (i.e. anodes and cathodes), both submerged into excrement slurry. The oxidation reaction of the electrolytic process can be divided into two major parts: (1) the direct oxidation by the electrical potential on the surface of electrodes, and (2) the indirect oxidation by reactive ions and radicals that are formed as intermediates [9]. A hydroxide ion ($OH^-$) is formed at the surface of the cathode, and hypochlorite (HOCl) and its anion ($OCl^-$) are produced at the same time from the anodic reaction according to the Faraday's law when chlorine ions exist in the liquid phase [9,10]. The concentrations of HOCl and $OCl^-$ vary depending on input voltage, allowable current, electrical resistance and pH of the reaction [10]. As a result, the strong oxidizers destroy odour precursors and odourous compounds, and these oxidations minimize odour emissions from septic tanks where the ORP drops and anaerobic conditions are developed.

The electrolytic oxidation process has known to be effective to remove organic constituents in industrial wastewater [10]. Because this electrochemical method can be directly applied to the slurry phase, it is also believed to be an effective control technique that is feasible and reliable to operate for the small-scale odour sources such as combined sewer septic tanks. In this study, the electrolytic oxidation was applied to simultaneously remove organic matters in the excrement slurry and malodourous compounds such as hydrogen sulphide from the headspace. In the electrolytic oxidation process, changes of the organic content determined by chemical oxygen demand and the concentration of gaseous hydrogen sulphide were monitored. In addition, the energy consumption and the removal efficiency during the electrolytic oxidation were calculated and presented as a function of applied voltage and allowable current density.

## 2. Materials ans methods

### 2.1. Excrement slurry

In order to determine chemical characteristics of excrement, the slurry was collected from a septic tank installed as a part of a combined sewer facility in a residential area, Korea. Organic constituents, pH, ORP and ionic concentrations of the slurry were immediately measured. The total chemical oxygen demand (TCOD) was found to be 4,550 mg/L; and the filtered soluble COD (SCOD) was 590 mg/L. The pH of the slurry was 7.4, and the ORP was −233 mV, indicating the excrement slurry was in an absolute anaerobic condition.

### 2.2. Electrochemical oxidation

Fig. 1 shows the configuration of the laboratory-scale electrolytic oxidation reactor, which was made of flexiglas with a total volume of 6.0 L (W 200 mm × L 150 mm × H 200 mm). The liquid volume was set to 4.0 L, and it was thoroughly mixed with a magnetic stirrer placed at the bottom of the reactor. The slurry described previously was filled in the reactor, and the electrolytic oxidation was performed as a closed, batch cell. The headspace of the reactor was 2.0 L, and a flexible bag (Tedlar, SKC inc., USA) containing one litre of nitrogen gas was connected to the headspace to prevent the pressure change of the reactor from periodic gas and liquid sampling events.

The electrolysis system consisted of three electrode plates in a raw, one anode at the centre and two cathodes at both sides with an electrode distance of 10 mm. The electrodes had dimensions of 60 × 100 mm, and they were mounted vertically in the middle of the reactor and connected to a DC power supply (Model DADP-3003, Dawoo Nanotech, Korea). The cathodes were stainless steel plates fabricated in a local machine shop. The anode, supplied by a metal coating company (Sungwon, Korea), was made of a titanium plate surface-coated with iridium dioxide (Ti/$IrO_2$) to prevent dimensional losses of the electrode.

The voltage differences between the electrodes were varied at 10, 20 and 30 V, and the current was allowed to vary at the given input voltage. As a reaction parameter, the energy consumption in terms of input electrical energy per mass of target compounds removed was used as a function of current density. All the experiments at given conditions were conducted in triplicate, and the averaged values and standard deviations are given.

### 2.3. Analytical methods

The excrement slurry samples were drawn from a liquid sampling port located at the middle of the reactor as illustrated in Fig. 1. To determine the change of organic constituents in the slurry, both TCOD and SCOD were periodically measured during the electrolytic reaction. An appropriate amount of the slurry sample was withdrawn and added to a COD test tube (Hach, USA), and the COD was measured according to the closed reflux, colorimetric method. The concentration of sulphate ion was measured using

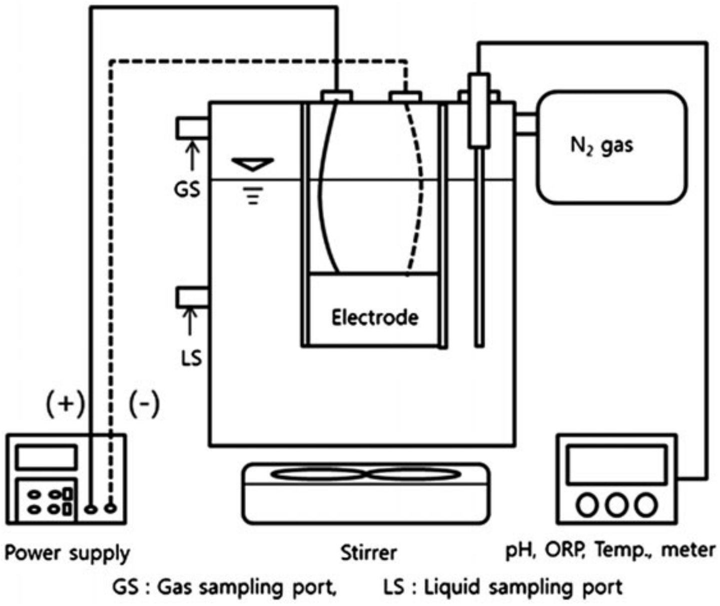

Fig. 1. Schematics of the electrolytic oxidation system used in this study.

an ion chromatography equipped with a conductivity detector and an anionic packed column (792 IC, Metrohm, Switzerland). Changes in temperature, pH and ORP were directly measured using a combined probe (Istek, Korea) installed into the slurry phase of the reactor. The gas sample was directly collected by using a gastight syringe from the reactor headspace. The concentration of reduced sulphur compound, $H_2S$, in the gas sample was quantified by using a gas chromatography (GC2010 Plus, Shimadzu, Japan) equipped with a flame photometric detector and a packed column. Helium was used as a carrier gas at a flow rate of 20.0 mL/min. The oven temperature was initially held at 70˚C for 0.5 min and heated up at a rate of 3˚C/min to a final temperature of 110˚C. The injection port and the detector temperatures were held constant at 170˚C.

## 3. Results and discussion

### 3.1. Electrolytic reactor operation

The electrolytic oxidation reactor was initially operated for two hours with an applied voltage remained constant at 20 V, while the DC was allowed to change. Fig. 2 shows the changes of temperature, allowable current, pH, and ORP during the electrolytic oxidation period. The slurry temperature increased rapidly from 25 to 36˚C over the 2 h period, which indicated an active electrolytic reaction and an exothermic joule heating in this batch cell operation. Foaming, due both to evolution of gaseous compounds and to production of natural surfactants from the decomposition of organic matters in the excrement slurry, was found at the top of the slurry. Bejan et al. [8] also reported a foaming from an electrolysis reactor treating hog manure where natural surfactants were presented in the reactants. With electrolysis time, the allowable current increased considerably from 2.0 to 2.5 A as shown in Fig. 2(b). This current increase attributed to the increase in temperature and the conversion of sulphur compounds in the slurry to dissolved sulphate ions. In a similar study using an electrolysis system operated in a batch mode and at a constant DC of 2.0 A [10], the voltage between the electrodes increased significantly from 30 to 47 V due to an accumulation of electrolytes during a one-hour experiment. In this study, where the three-electrode-plate arrangement was applied at the fixed voltage of 20 V, the initial current density was 83.3 A/m$^2$ that was a typical value ranging from 5 to 120 A/m$^2$ reported in the literature for wastewater treatments [9–11]. These findings confirmed that the active electrolytic oxidation took place in the excrement slurry without an addition of external electrolytes such as chloride ions to increase conductivity in the slurry phase.

At this given voltage and current condition, the predominant chemical reaction at the surface of the anode was assumed to be the production of hypochlorous acid (HOCl) [9,10]. In contrast, the cathode reaction

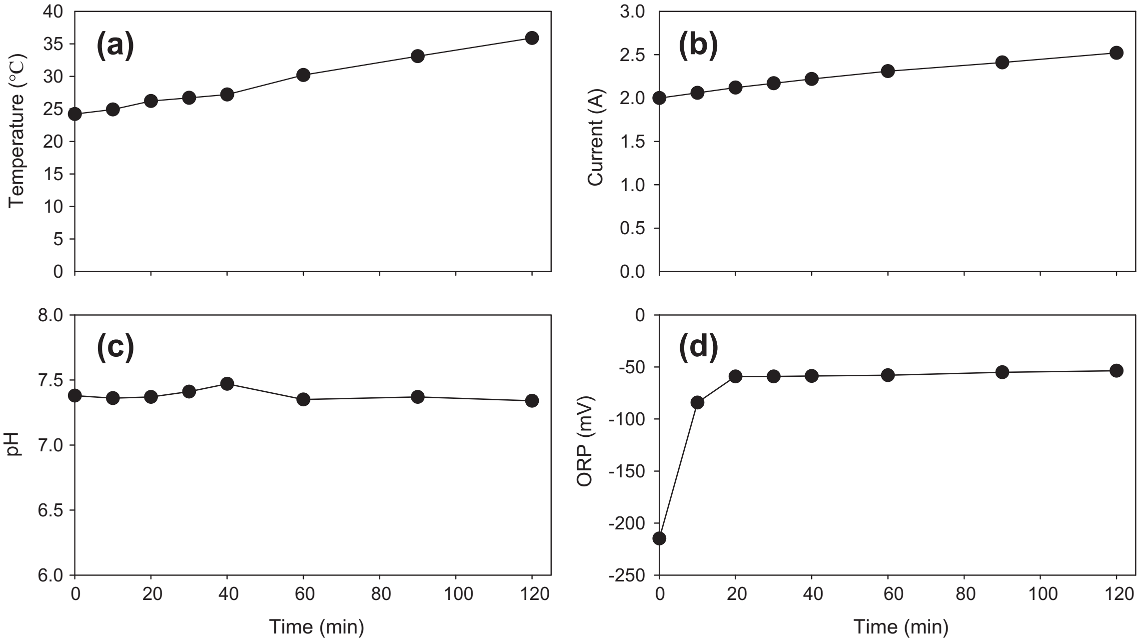

Fig. 2. The changes of (a) temperature, (b) allowable current, (c) pH, and (d) ORP in the electrolytic oxidation reactor during the 120 min operational period.

might produce hydroxide ion ($OH^-$) corresponding to an increase in pH. Because the slurry phase in the electrolytic reactor was thoroughly mixed, the reactants from the anode and the cathode were combined, and the pH value remained nearly constant throughout the reactor operation as shown in Fig. 2(c). Therefore, the changes of pH cannot be an indicator of the electrolytic oxidation in this reactor configuration. Meanwhile, the ORP in the slurry phase increased rapidly from −220 to −55 mV due to the oxidation reaction at the beginning of the operation as illustrated in Fig. 2(d). A similar trend in the change of ORP was observed in another study for the electrochemical oxidation of sulphide in oil wastewater, in which the ORP of the solution changed from negative to positive when a high current density was applied and sulphide ions were completely oxidized [12]. After 20 min in the batch reaction of this study, the ORP remained nearly constant throughout the operation. Overall, this result indicated that the electrolytic oxidation converted the absolute anaerobic state in the excrement slurry to the moderate condition that could minimize the formation of the reduced odour compounds.

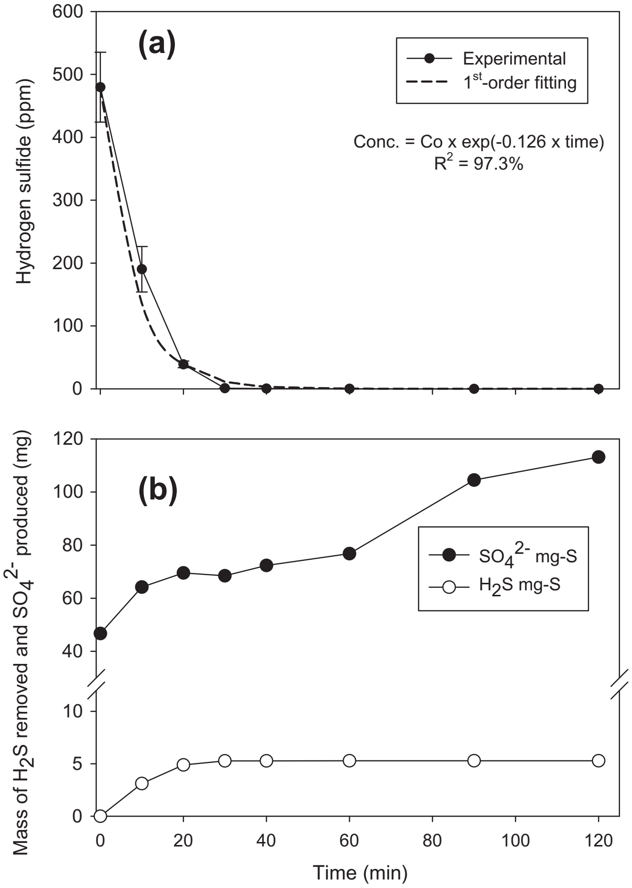

Fig. 3. (a) Change of the $H_2S$ concentration in the head space of the electrolytic reactor, and (b) the accumulative mass of sulphide removed from the reactor and the mass of sulphate produced in the slurry phase. The error bars indicate the standard deviations.

### 3.2. Oxidation of sulphur compounds

The oxidation of $H_2S$ in the electrolysis reactor was quantified by measuring the gaseous concentration in the headspace. Fig. 3(a) shows the concentration profile of $H_2S$, which rapidly declined in the headspace initially and dropped to less than 1 $ppm_v$ after 30 min. Another reduced sulphur compound, methyl mercaptan, was also detected by the gas chromatographic analysis, and its concentration decreased from 0.7 $ppm_v$ to below the detection limit within 30 min following the same decline pattern as $H_2S$. For comparison, a control test without the current supply was also conducted at the same system configuration, and a decline of less than 3% of the $H_2S$ concentration was observed in the headspace during the 120 min period (data not shown). Therefore, the electrolytic oxidation was an effective method to remove the odourous compounds from the excrement slurry within the relatively short period of time.

The depletion of target compounds by the electrolytic oxidation in a batch reactor can be described by [13];

$$\frac{dC}{dt} = \sum k_i [C]^m [O_i]^n \tag{1}$$

where $C$ is the concentration of the target compound in the electrochemical reactor at a given time, $k_i$ is the reaction constant, $O_i$ is the concentration of oxidant species produced by the indirect electrolysis reaction, and $m$ and $n$ are the partial orders.

Assuming stationary conditions and pseudo-steady-states under which there was no accumulation of predominant oxidant species in the liquid phase and their production and consumption rates were equal, the equation for the $H_2S$ oxidation simplifies as follows;

$$\frac{d[H_2S]}{dt} = k'_{H_2S}[H_2S] \tag{2}$$

where $[H_2S]$ is the $H_2S$ concentration in the headspace ($ppm_v$), and $k'_{H2S}$ is the observed rate constant for the $H_2S$ oxidation. As illustrated in Fig. 3(a), the decline profile of the $H_2S$ concentration with time can be fitted to the pseudo-first-order kinetic. A non-linear fitting agrees well with the experimental data, and the first-order decay constant is calculated to be

$0.126\,\text{min}^{-1}$. Several studies demonstrated that the first-order decay rates for electrolytic wastewater treatments substantially vary as a function of the current density, the concentration of electrolytes, temperature, and the initial concentration of hydrogen sulphide [14,15]. Additional studies under various experimental conditions would be required to determine limiting factors affecting the first-order decay rate.

Along with the $H_2S$ removal in the reactor, the concentration of sulphate ion ($SO_4^{2-}$) significantly increased in the slurry phase. This is clear evidence that the sulphide was actually oxidized to sulphate, the final sulphur oxidation product in the electrolysis. The total mass of $H_2S$ removed from the headspace was 5.3 mg as *S*, and the sulphide oxidation took place mostly during the initial 40 min period as shown in Fig. 3(b). In contrast, the sulphate in the slurry phase increased from its initial mass of 46.7 mg as *S* to its final mass of 113.1 mg as *S* after two hours, that is, the overall 66.4 mg as *S* increase, and it gradually increased with time. This implies that the electrolysis at the given current condition oxidized not only $H_2S$ but also other organic sulphur compounds in the excrement slurry.

During the electrolytic oxidation of sulphide, other sulphoxy intermediates or elemental sulphur might be produced and eventually oxidized to the final product. Another study reported in the literature [11] showed that thiosulphate as an intermediate was produced during the initial period of direct sulphide electro-oxidation and the intermediate was eventually oxidized to sulphate. Since the excrement slurry used in this electrolytic oxidation study might have a variety of sulphur-containing compounds, a complete mass balance on sulphur was necessary to obtain an insight into sulphur oxidation mechanisms. Nevertheless, the findings of this study revealed that the electrolytic oxidation is a promising method to reduce odour-causing potential from the excrement slurry containing hydrogen sulphide at high concentrations.

### 3.3. Removals of organic constituents

The electrolytic reaction resulted in the oxidation of organic constituents in the slurry. Fig. 4 shows the changes of TCOD and SCOD in the slurry phase over the two-hour period. The TCOD steadily decreased from its initial concentration of 4,620 mg/L to its final value of 2,780 mg/L when the batch reaction was terminated, while the concentration change of the SCOD was relatively low from 412 to 328 mg/L during the same period of time. Consequently, the electrochemical reaction was mainly targeted to dismantle the organic solid matters in the excrement slurry and further oxidized readily degradable organics to the final product, $CO_2$ (data not shown). As mentioned and showed in Fig. 3(b) above, the increase in sulphate ion closely reflected the decrease in TCOD, indicating that organic compounds containing sulphur were effectively oxidized at the applied electrical energy in this study. Contrary to the $H_2S$ decline profile, the TCOD decline was linear with time, and the profile can be fitted into the zero-order kinetic equation, that is, the m value is zero in equation (1), as follows;

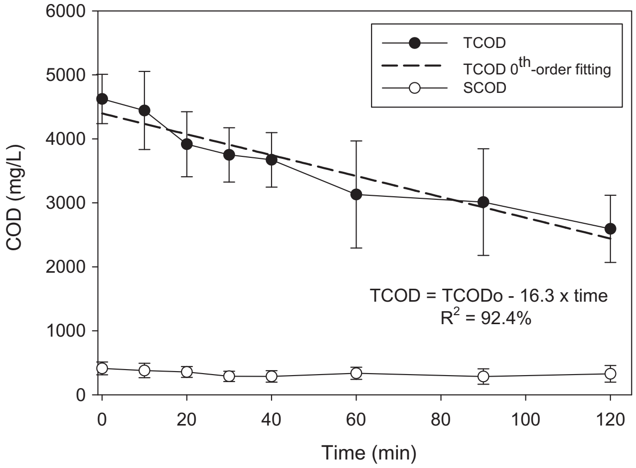

Fig. 4. Changes of the TCOD and the SCOD during the batch electrolytic reaction. The error bars indicate the standard deviations.

$$COD_t = COD_o - k_0 \times t \tag{3}$$

where $COD_t$ is the TCOD in the slurry phase (mg/L), $COD_o$ is the initial TCOD (mg/L), and $k_0$ is the observed pseudo-zero-order constant for TCOD decay (mg/L/min). As illustrated in Fig. 4(a), linear fitting agrees well with the TCOD results, and the zero-order decay constant is calculated to be 16.3 mg/L/min.

### 3.4. Effects of applied current density on removals

In order to investigate the effect of electrical potential on the electrolytic oxidation, different voltages were applied at 10 and 30 V, respectively, using the same excrement slurry and experimental setup, and these results were compared with that obtained at the applied voltage of 20 V. Since these experiments were conducted at the fixed voltages, the DC at each voltage was allowed to vary. The current density of each voltage condition, expressed as $A/m^2$, is calculated and listed in Table 1. At the fixed voltage of 30 V, the

Table 1
Current densities and energy consumptions as a function of applied voltages

| Voltage (V) | Current density (A/m$^2$) | Energy consumption during the initial 30 min period | | |
|---|---|---|---|---|
| | | kWh/m$^3$ | kWh/kg-$H_2S$ | kWh/kg-TCOD |
| 10 | 34.6 | 1.4 | 794.4 | 1.8 |
| 20 | 83.3 | 7.1 | 2564.1 | 8.3 |
| 30 | 125.0 | 17.1 | 7341.4 | 9.2 |

$H_2S$ concentration in the headspace dropped more rapidly than that at 20 V, and the TCOD in the slurry also degraded at an increased decay rate of 35.0 mg/L/min. Meanwhile, at the fixed voltage of 10 V, the removal rates of $H_2S$ and TCOD were the lowest among the three conditions, as anticipated.

The energy consumption is an important reaction parameter to evaluate the electrolytic system. In this study, the energy consumptions, normalized by the slurry volume (kWh/m$^3$) or by the removals of 1 kg of $H_2S$ (kWh/kg-$H_2S$) and of 1 kg of TCOD (kWh/kg-TCOD), were evaluated. For comparisons between each experimental condition, the accumulative amounts of the electrical energy applied and the masses of $H_2S$ and TCOD removed during the initial 30 min period when the most active oxidation took place were selected. As the voltage and current density increased as listed in Table 1, the energy consumptions for the unit masses of $H_2S$ and TCOD removals increased, indicating that undesirable reactions other than the $H_2S$ and organic oxidations proceeded on the surface of electrodes at higher voltages. Rajkumar and Palanivelu [10] reported that an electrolytic oxidation for the treatment of drug manufacturing wastewater yielded an energy consumption of 17.0 kWh/kg-COD, while the same reactor treating oil refinery wastewater required 253.3 kWh/kg-COD that was two-order higher than the values determined in this study. As a result, the energy consumption needs to be experimentally evaluated for the electrolysis configuration and the wastewater characteristics.

In addition, the removal efficiencies for $H_2S$ and TCOD during the initial oxidation period were evaluated as a function of the current density, as shown in Fig. 5. At the lowest voltage and corresponding current density, only 76.1% of the initial $H_2S$ was removed from the headspace of the reactor, and the removal efficiency for TCOD was relatively low as well. As the applied voltage increased to 20 V, the $H_2S$ removal efficiency increased and almost complete oxidation was observed within the 30 min period. Nevertheless, the removal efficiency for TCOD was not improved at the applied voltage of 20 V. A significant increase in the TCOD removal was found when the applied voltage was further increased to 30 V. Consequently, this result suggests that an appropriate current density as a threshold value should be selected depending on the application purpose of the electrolytic system, that is, either for the $H_2S$ removal or the TCOD oxidation. In order to reduce the odour-causing potential only, the current density of 80 A/m$^2$ would be an optimum at this given condition.

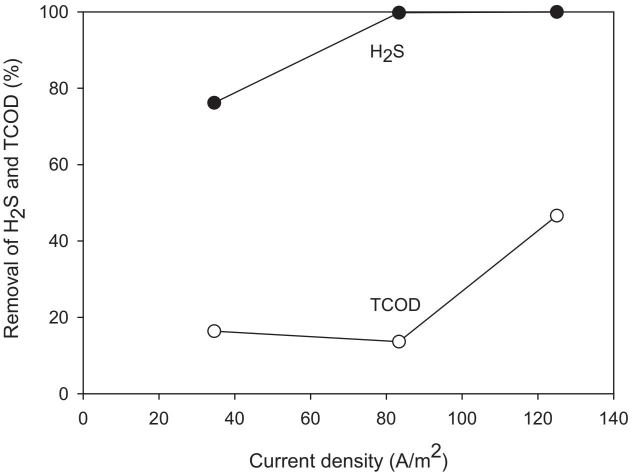

Fig. 5. Effects of the current density on the removal efficiencies of $H_2S$ and TCOD.

## 4. Conclusions

The electrolytic batch system was applied to oxidize and remove both organics and odourous sulphur compounds from septic tanks treating the excrement slurry. In this study, the DCs at fixed voltages of 10, 20 and 30 V, respectively, were applied to metallic electrodes submerged into the slurry. At all of the voltage conditions, active electrolytic oxidation took place in the excrement slurry without an addition of external electrolytes, and the predominant chemical reaction at the surface of the anode was assumed to be the production of the oxidation power. This reaction resulted in the simultaneous oxidation of the odourous and organic compounds and the increase in ORP from the absolute anaerobic state to the near neutral condition. At the applied voltages greater than 20 V, the concentration of $H_2S$ in the headspace of the reactor exponentially dropped within initial 30 min period, and its profile could be fitted into the pseudo-first-order kinetic equation. As the $H_2S$ concentration decreased in the headspace, the concentration of sulphate ion more substantially increased in the slurry phase, indicating that the sulphide was actually oxidized to its final oxidation product, and the electrolysis at these given conditions oxidized not only $H_2S$ but also other organic sulphur compounds in the excrement slurry. In addition, the TCOD in the slurry was gradually declined at all the voltage conditions, and a rapid removal of the TCOD was observed only when the fixed voltage of 30 V. The energy consumptions expressed as kWh/m$^3$, kWh/kg-$H_2S$, and kWh/kg-TCOD were calculated and compared with the experimental results herein and with values reported in the literature, showing that they varied greatly depending on the system configuration and the wastewater characteristics. An appropriate current density and energy consumption as a threshold value should be selected to optimize the system for the simultaneous removals of organics and reduced sulphur compounds. Consequently, the electrolysis can be an alternative over traditional methods for the control of odours emitted from the excrement slurry.

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