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INNOVATIVE SLUDGE DISINFECTION APPROACH TO GENERATE CLASS A BIOSOLIDS FOR LAND APPLICATIONS

Luo, Jiaru[1,2], Elektorowicz, Maria[1,3]
[1] Concordia University, Department of Building, Civil and Environmental Engineering, Montreal, Canada
[2] **l_jiaru@encs.concordia.ca**
[3] **mariae@encs.concordia.ca**

Abstract: Wastewater treatment facilities generate a huge amount of sludge from their processes. One of the conventional method for sludge management is the land application as a fertile soil amendment which is an efficient way to recycle nutrients from human waste. However, since sludge also contains pathogens, it is necessary to be disinfected before its usage. Then, a novel system has been demonstrated for sludge disinfection using a treatment supported by electrical field. This paper mainly focuses on optimization of the system to find out the best operating conditions for their further implementations. Investigation of Fe^{2+}/H_2O_2 ratio, pH, current level, and amount of H_2O_2 were conducted to determine effective parameters for sludge disinfection through the Electro-Fenton method. The results showed log 7 reduction of *E. coli* within 30 minutes by applying selected parameters into sludge (WAS) in the electro-reactor.

1 INTRODUCTION

Municipal sludge management might reach up to 60% of the total operating costs in a wastewater treatment plant (Andreoli et al. 2007). Since it should be controlled in a harmless way toward the public, municipal sludge management usually includes dewatering, pathogenic disinfection and nutrient recycling if considered possible. The pathogenic reduction is the most important with respect to public health and environmental protection. Therefore, the regulations regarding the pathogen content in sludge are defined before the sludge land application. In order to satisfy the requirements for Class A biosolids, minimum 5 log reduction of fecal coliform should be achieved (EPA 1993, CCME 2012). The lime treatment, aerobic and anaerobic digestion and composting have been authorized as standard treatment methods. However, these methods require a long treatment time (from 20 hours to months), long retention time of the process, which lead to building facilities with an extensive space demand. Usually, the construction of such expensive facilities for an adequate sludge disinfection is a big financial issue for small wastewater treatment plants (WWTP). . Therefore, electrokinetic technology would be an ideal method to satisfy disinfection requirements because of its relatively low capital and operation costs.

Some previous work have already demonstrated that direct current (DC) applied to sludge generates electrokinetic stressors (Esmaeily et al. 2006), desiccation stressors (Huang et al. 2012), or ohmic heat stressors for disinfection of fecal bacteria (Daneshmand et al. 2012) as well as spores (Safaei et at. 2013). Furthermore, hydroxyl radicals ($^{\bullet}OH$) showed their high oxidization potential which could rapidly react with organic matrix as an effective disinfectant (Selvakumar et al. 2009). The previous study demonstrated that ($^{\bullet}OH$) can achieve 2 log *E. coli* reduction with the concentration of 0.8×10^{-5} mg/L.min (Cho et al. 2004). Generation of hydroxyl radicals can be done through Fenton reaction, Eq. [1-2], where ferrous iron, acting as a catalyst, reacts with hydrogen peroxide to generate ferric iron and hydroxyl radicals, while introduced hydrogen peroxide reacts with ferric iron to regenerate ferrous irons:

[1] $Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + H_2O + {}^{\bullet}OH$

[2] $Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2 \bullet\ + H^+$

Then, the reaction with (${}^{\bullet}$OH) leads to pathogens' inactivation. Fenton reaction has been used in different situations for disinfection, e.g. water. Recent studies on water and wastewater disinfection by using Fenton reaction focus more on Photo-Fenton reaction by applying extra UV light to accelerate the production of ferrous iron to generate more hydroxyl radicals (Polo-López et al. 2012; Ortega-Gómez et al. 2012). Since UV light cannot penetrate through the medium, photo-Fenton is not efficient for treatment of highly turbid water with a high organic load. In this case, Fered–Fenton oxidation process is considered as a substitute technology for the Fenton-based application. Similar to the Photo-Fenton reaction, ferrous iron reacts with hydrogen peroxide to produce hydroxyl radicals. However, reduction of ferric iron is conducted by electro-reduction. Equation [3] shows that ferric iron is reduced at the cathode in the presence of continuously applied hydrogen peroxide and produces hydroxyl radicals (${}^{\bullet}$OH) (Brillas et al. 2009).

[3] $Fe^{3+} + e^- \rightarrow Fe^{2+}$

One of the advantage of Fered-Fenton and Photo-Fenton processes compared to traditional Fenton reaction is the feasibility of using smaller amount of ferrous iron at the beginning as a catalyst, which can be regenerated by the system. For example, Fered-Fenton process were applied for treatment of both leachate and industrial wastewater having obvious turbidity and high concentration of COD (Zhang et al. 2006; Li et al. 2010; Jahromi 2016). However, there is always a concern to find technological conditions which inactivate pathogens during a short retention time at low costs. Therefore, the aim of this study was to find such an electro-Fenton oxidation method which might transform waste activated sludge (WAS) into Class A biosolids during a short exposure period in order to advance the sludge management methods.

2 MATERIALS AND METHODS

Sludge samples were taken from the activated sludge facilities located in wastewater treatment plant in Saint-Hyacinthe (Quebec, Canada) and stored at 4℃. The sampling took place after thickening process where the coagulant (ferrous sulfate) was added before sludge dewatering. The initial characteristic of sludge included: pH, TAN, *E.coli,* Total coliform, Cl^-, conductivity, TS%, while values of these factors were as follow 6.4-6.8, 51mg/l, 4×10^9 cfu/100ml, 4.17×10^9/100ml, 14.5mg/l, 1200μS/cm, and 5.5%, respectively.

A series of batch experiments were conducted in an electro-reactor (12cm × 7.5cm × 10cm) where stainless steel electrodes were placed (Fig. 1). An amount of 1000ml of sludge solution was used for each experimental run. DI water (Milli-Q® Integral, Sigma Inc.) was supplied for sludge dilution to reach a required TS (total solids) content around 0.5-0.8%. The electro-reactor was operated at a constant current density using DC power supply (GENESYS™ 750W/1500W, TDK-Lambda Americas Inc.). To maintain a homogenous sludge solution, a magnetic stirrer (Fisher brand) was used for continuous mixing. Furthermore, a thermocouple was placed in the middle of the reactor for temperature monitoring (Agilent 3450a data logger), and a peristaltic pump (Cole-Parmer Instrument. LLC) was applied for sampling from a middle area of the electro-reactor.

The pH level was adjusted with 1M hydrochloric acid before applying the current as well as during the treatment process. The pH value was monitored by Accumet™ AB2000 pH/conductivity bench meter. Ferrous ammonia sulfate was used as a source of ferrous iron. Total ammonia (TAN) and chloride were measured by selective ammonia membrane probe (IntelliCAL™ Ammonia ISE) and Fisher ISE chloride probe, respectively.

Source of *E.coli,* used as a pathogenic indicator*,* was the wastewater in a poultry farm (Quebec, Canada). Collected bacteria were subjected to bioaugmentation to reach an adequate density of cells, and then, 1ml of prepare cultured LB was injected into 50ml fresh LB and incubated overnight to maintain the range of bacteria *E.coli* density around 10^{11} cfu /100ml. In order to identify the *E.coli*, MFC agar (BD Difco™) was applied for a standard membrane filtration method with a serial dilution, then, counting on the filters (APHA/AWWA/WEF 2012) was conducted. In this study, for simulating the density of bacteria in the sludge, 10mL of cultured LB broth was injected into the electro-reactor to achieve 10^{-9} cfu/100mL for the initial bacterial concentration.

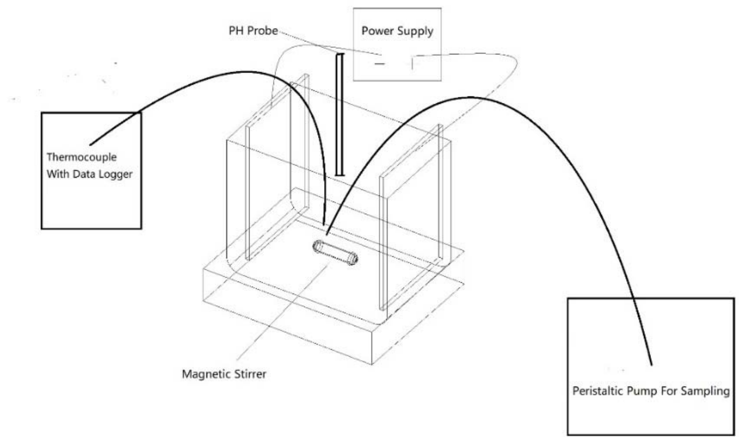

Figure 1 Schema of the experimental setup

Since this study aims to investigate an electro-Fenton like process, an effectiveness of multi-stressors on *E.coli* disinfection should be demonstrated. Subsequently, a series of experiments were conducted in the electro-reactor. Firstly, a disinfection efficiency was assessed in relation to various amounts of H_2O_2 (0ml, 1ml, 2ml), which is shown as cases A, B, C, respectively in Figure 3. Furthermore, Case D (Fig.3) considered the effect of Cl^- ion on *E. coli* inactivation; subsequently, 2.8g/L of KCl was added (at 1.3A) to the electro-reactor. Since hydrochloride was chosen for pH adjustment, Cl^- being oxidized to ClO^-/Cl^- on the anode might generate hypochlorite acid (HClO), which is considered as an effective disinfectant (Huang et al. 2016). Next series of experiments (Fig. 4) considered an effect of various amperage values (0.2A, 0.4A, 0.8A as cases A,B,C, respectively). Comparison of pH values of 4 and 7 (case A and B in Fig. 5) permitted to assess their impact on *E.coli* inactivation. Various ratio of Fe^{2+}/H_2O_2 (no H_2O_2, 1:12, 1:6, 1:3) were applied to demonstrate the Fenton reaction impact on *E.coli* disinfection efficiency (Fig. 6). Such methodological approach permitted to assess impacts of various combinations of stressors on bacteria inactivation and define the best conditions for the proposed disinfection method.

3 RESULTS AND DISCUSSION

3.1 Comparison of Different Applied Conditions on E. Coli Inactivation

The comparison of results from series A (impact of heat as a stressor), B (heat and Fenton reaction), C (stressors from electro-Fenton oxidation), are shown in Figure 2. As can be seen in case A, no significant *E.coli* inactivation during 30 minutes process was observed, which demonstrated that the combination of heat and acid environment was not the main bacteria inactivation factor for this system. However, in case B, an addition of ferrous iron and H_2O_2 permitted to reach 2 log reductions in 15 minutes and 5.2 log reduction in 30 minutes of treatment. This indicates that the combination of ferrous/ H_2O_2 created disinfection conditions, most probably by production of hydroxyl radicals. In such case, the system was exposed to Fenton reaction (Eq. [1]), which contributed to the *E.coli* inactivation. Furthermore, the case C showed that the implementation of electrical field significantly improved *E,coli* reduction by achieving 7.3 log within 30 minutes; while, the required log 5 reduction has been already reached after 15 min. Applied ratio of ferrous iron to hydrogen peroxide was 1:12. Since the ferrous iron concentration is a limiting factor for generation of hydroxyl radicals (•OH) in Fenton reaction, the application of current reduces ferric iron to ferrous iron by electro-reduction at the cathode. Therefore, in case C, ferrous iron was regenerated, additionally reacted with hydrogen peroxide and produced more hydroxyl radicals (•OH).

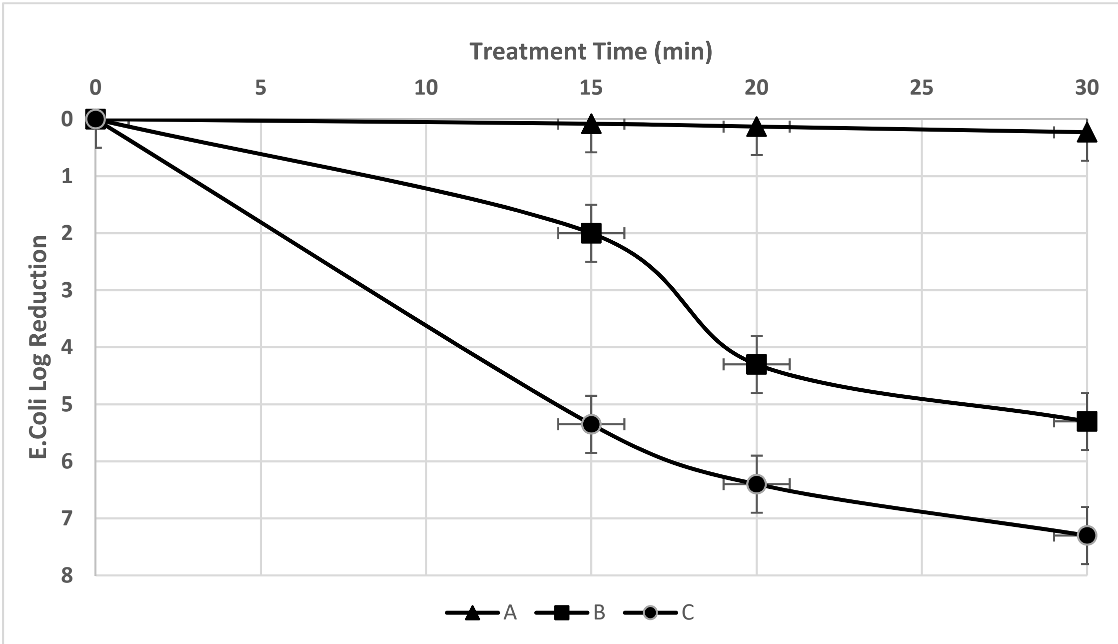

Figure 2 E.coli reduction vs. treatment time and different stressors (A, B, C)

Note: Electro-reactors operated under pH=4, and TS = 0.8%; A: heat as a stressor only; B stressors: heat as well as ferrous iron and H_2O_2 (Fe/ H_2O_2 = 1:12); C stressors: ferrous iron and H_2O_2 as well as a constant current density

The assumption that hydrogen peroxide as a strong oxidant, might also contribute to inactivation of *E. coli* was excluded since other study (Ortega-Gómez et al. 2012) demonstrated that H_2O_2 does not have negative effect on *E.faecails* survival rate. Therefore, it was concluded that hydrogen peroxide acted as a hydroxyl radicals' generator but it was not the main disinfectant.

3.2 Effect of H_2O_2

The experiment B (Fig. 3) shows that the addition of 1ml of H_2O_2 permitted to reach 2 log reduction of *E.coli* in 15 minutes and almost 4 log reduction in 30 minutes compared to 0.2 log reduction without H_2O_2. The application of twice higher amount of H_2O_2 can achieve twice higher inactivation (4 log reduction) in 15 minutes. A higher amount of H_2O_2 generates a greater *E.coli* log reduction due to presence of an organic matter which also reacts with hydroxyl radicals (•OH). The abstraction of protons can be further oxidized to CO_2 and others organic forms (Yoon et al. 2001). Therefore, a high amount of the organic matter might compete with the bacteria inactivation process with respect to (•OH) reactions, and subsequently, might cause a decrease of the disinfection efficiency. Figure 3 does not show an obvious *E. coli* reduction (less than 1 log) in case D for both 15 and 30 minutes. It may be concluded that chlorite is not the main disinfectant in this system. In order to satisfy the conditions of anodic oxidation, the electrode material should have a high oxidation potential. Formation of chlorite on electrode must have higher potential than 1.36V (Martínez-Huitle and Andrade 2011). However, iron electrode achieves 0.77V oxidation potential only, then, no inactivation of *E. coli* can be expected with such low formation of ClO^-/Cl_2. This conclusion was also generated in other tests when additional KCl and seven times higher (1.3A) current than in cases B(0.2 A) and C(0.2 A) were applied. The results still had much lower *E.coli* log reduction (1.3 log) by the end of 30 minutes than in the case C (5.2 log reduction) (Fig. 3). Besides, a poor E.coli reduction might be caused by the presence of ammonia in the medium. Huang et al., 2016 demonstrated that under the presence of ammonia, free chlorine generated in the anode will be converted to chloramine, which has much lower disinfection efficiency for eliminating MS2 (bacteriophage) in wastewater.

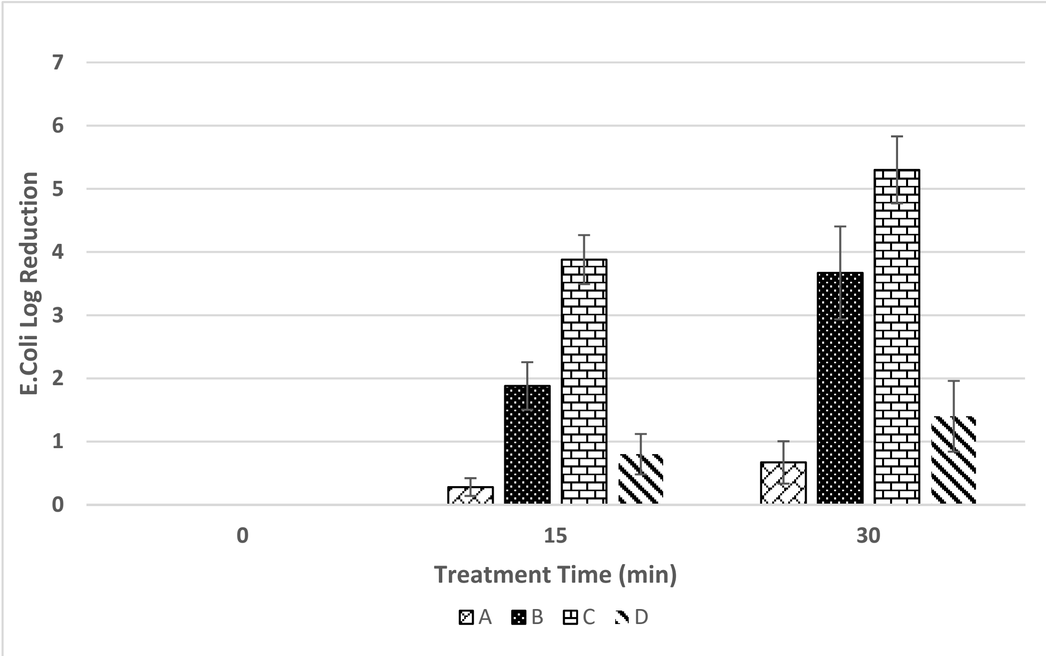

Figure 3 E. coli reduction vs. time of treatment and different dosage of H_2O_2 with additional test applying KCl

Note: All tests operated under 0.2A, TS = 0.8%, pH=4, additional ferrous iron and H_2O_2 in the ratio of Fe/ H_2O_2 = 1:12, while H_2O_2 were 0ml, 1ml, 2ml in case A,B,C, respectively; case D was operated under pH=4 with 1.3A current, 2.8g KCl /L, (pH was adjusted with H_2SO_4)

3.3 Effect of current

Figure 1 has already shown that the highest inactivation of *E. coli* could be achieved when an adequate Fe/ H_2O_2 ratio and DC field were applied. Figure 3 indicates that under constant input of H_2O_2 more ferrous iron is present in the system, thus, better inactivation is achieved. The same observation was presented by others (Brillas et al. 2009). Then, the application of current, which regenerate ferrous, is directly related to disinfection efficiency. Figure 4 shows the relationship between current and *E.coli* log reduction. However, since applied current can also cause ohmic heat effect, higher current comes along with higher temperature in the system; this might be another environmental stressor for bacteria inactivation (Safaei et al. 2013).

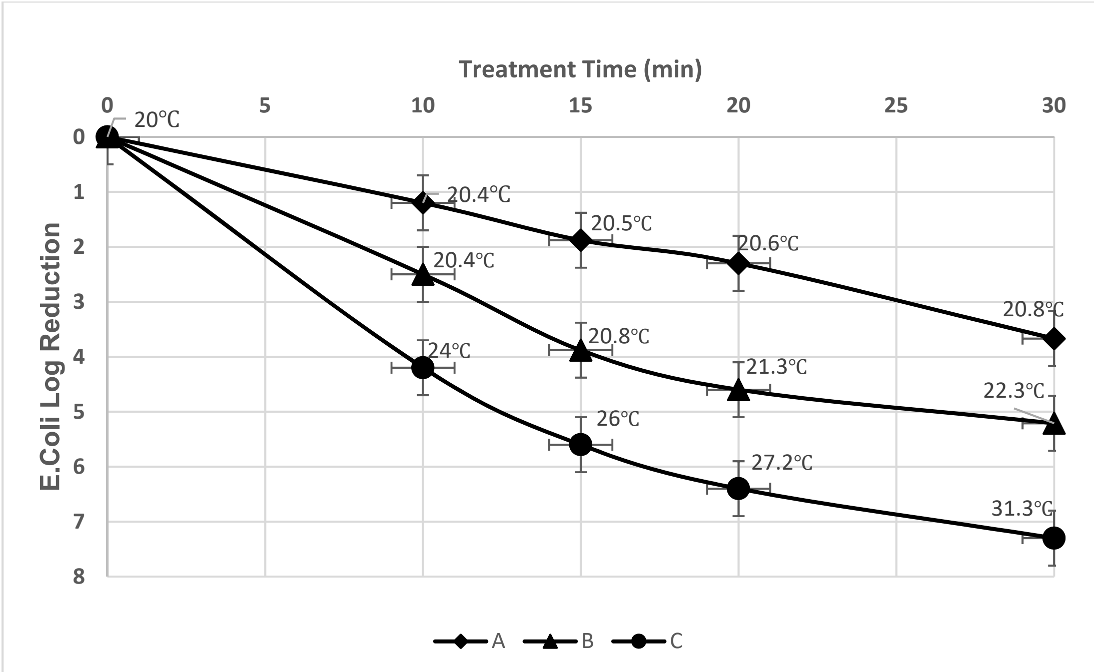

Figure 4 E. coli reduction vs time of treatment for different DC current.

Note: Experiments were conducted under: pH=4, TS: 0.8%; additional ferrous iron and H_2O_2 (1ml) in the ratio of Fe/ H_2O_2 =1:12, while current 0.2A, 0.4A, 0.8A were applied into cases A, B, C, respectively; Temperature was also recorded in different treatment periods

To avoid impact of a heat, the experiments were conducted at a low current level to minimise the temperature effect. Current between 0.2A and 0.4A kept temperature around 20-22 ℃. However, the bacteria inactivation was obviously different; the *E.coli* reduction of 3.5 log for 0.2A, and 5.5 log reduction for 0.4A (after 30 minutes) were observed. The current increase to 0.8A did not change significantly the bacteria reduction, although temperature has been simultaneously increased by 6 ℃. This proved that ohmic heat was not the main stressor for *E. coli* inactivation. Though, some of the other published articles declared that ohmic heat can also be used for disinfection (Yin et al. 2018). They demonstrated that by using ohmic heat, when temperature went up to 50 ℃, the 4.5 log reduction could be achieved, while 0.68 log reduction only for temperature of 28℃. Their results confirmed the outcome of this study. Although raising current density could have a higher reduction rate and regenerate more ferrous iron, however, it would use more energy to produce ohmic heat, thus, it does not seem to be necessary for an adequate inactivation of *E. coli* in thickened WAS. Some investigations on cost-effectiveness should be conducted in the future.

3.4 Effect of pH

As Eq. [1] demonstrated that Fenton reaction should be conducted under acidic environment in order to generate more hydroxyl radicals (●OH). Therefore, pH might directly affect the generation of oxidants. Significant different results of bacteria inactivation were obtained (Fig. 5), under pH=7 and pH=4, where 0.5 log and 5.5 log reduction in 15 minutes were achieved, respectively. No further significant change for pH=7 was observed even when the exposure time was extended to 30 minutes. This demonstrated that contact time in the system was not the main factor affecting disinfection. However, the amount of produced hydroxyl radicals (●OH) defined *E. coli* disinfection. This also was showed by others (Diagne et al. 2007), who removed methyl parathion under pH=3-4 by using hydroxyl radicals (●OH) while a different removal they obtain under pH=7. Furthermore, another explanation (as it has been suggested by Pang et al., 2011) may be attributed to the formation of surface bonding hydroxyl radicals. It was assumed that hydroxyl radicals can be generated even under neutral environment, but they bond with the surface of insoluble metal species (e.g. ferric hydroxides). Subsequently, hydroxyl radicals cannot unselectively oxidize *E.coli*.

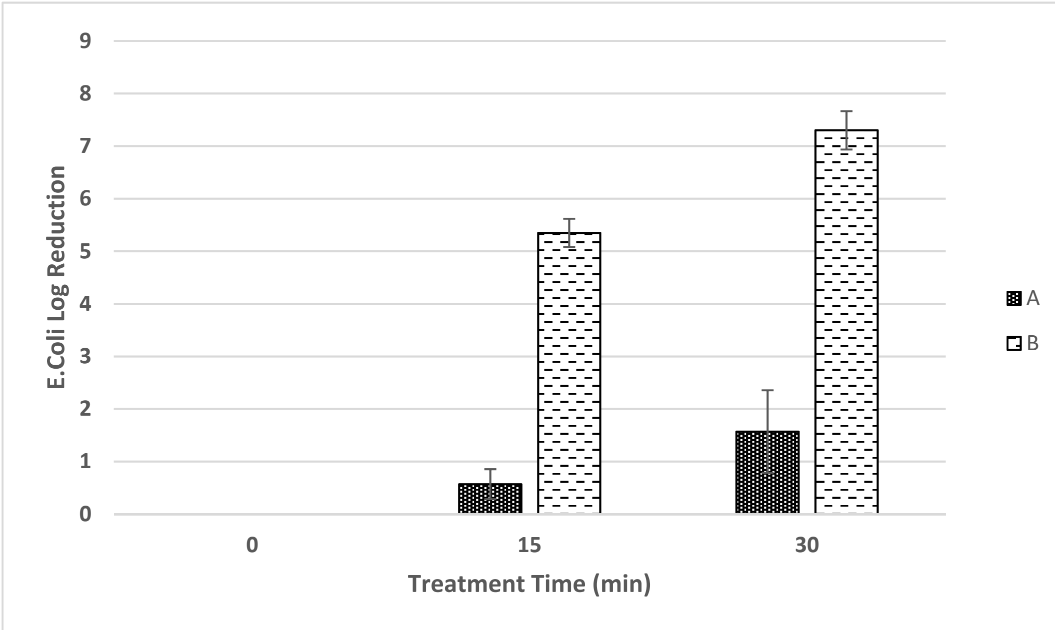

Figure 5 E. coli reduction vs treatment time for different pH

Note: Cases A and B were operated under current 0.8A, ratio of Fe^{2+}/ H_2O_2 =1:12, case A was under pH=7, case B was under pH=4

3.5 Effect of Fe/ H_2O_2 Ratio

Different ratios of Fe/ H_2O_2 were investigated to assess their impact on *E. coli* reduction. No regeneration of ferrous iron was considered in this experiment - no current was applied (Fig. 6). Since the initial thickened sludge has already contained the ferrous sulfate coagulant, the process achieved almost 2 log reduction of *E. coli* without iron additional. The results were improved with the addition of ferrous into the system. This is because (Eq. [1,2]) ferrous was the limitation factor for *E. coli* disinfection when Fe/ H_2O_2 ratio was smaller than 1 (Zhang et al. 2012; Neyens and Baeyens 2003). In such case, an excess of ferrous iron is converted to ferric iron and forms ferric hydroxo-complexes, which aggregate suspend solids and precipitate. The results from other research on Fenton method (Juarez et al. 2010) showed that disinfection efficiency increased as the ratio of Fe/H_2O_2 increases. It was assumed that the inactivation of MS2 was mediated by iron colloids. Therefore, when an amount of ferrous iron increases, and H_2O_2 is kept constant (ratio 1:3), the bacteria reduction reaches 5.5 log within 30 minutes of treatment. It could be expected that when the ratio increases to 1:1.5, a 7.3 log reduction can be achieved. Therefore, without current application, the ratio Fe/ H_2O_2 should be greater than 1:3 to reach 7.3 log reduction. Otherwise, electro-reduction should be used to regenerate more ferrous to compensate insufficient ferrous iron in the system.

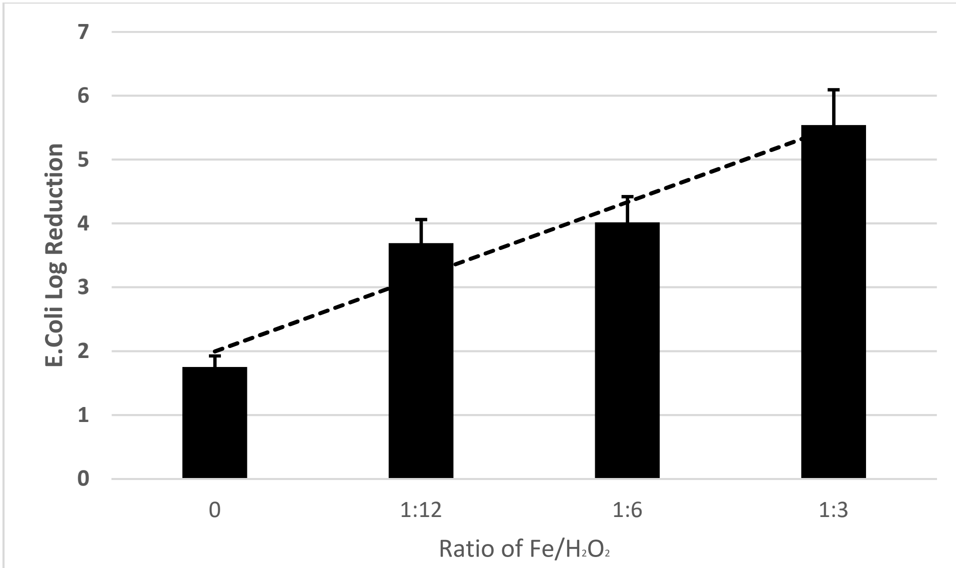

Figure 6 E. coli reduction vs different ratio of Fe^{2+}/H_2O_2 (after 30 min exposure time)

Note: Experiments operated under: pH=4, TS=.8% , addition of ferrous iron and H_2O_2, without current and heat.

4 CONCLUSIONS

- The novel electro-disinfection system was able to achieve log 7 reduction of *E. coli* in thickened sludge (WAS) containing 0.8% TS;
- Under pH=4, current 0.8A, while the ratio Fe^{2+}/ H_2O_2 = 1:6 (contained 1ml H_2O_2), the system could effectively inactivate *E.coli* within 30 minutes;
- Main stressors are in-situ generated hydroxyl radicals (•OH) which interfere with *E. coli* cells*;* other stressors such as ohmic heat, pH, ClO- ions are minor stressors within this system;
- Higher Fe^{2+}/ H_2O_2 ratio supported by current generate sufficient hydroxyl radicals (•OH) due to regeneration of an adequate amount of ferrous iron in the system to proceed with electro-Fenton like oxidation;
- The proposed method can be applied to enhance sludge management and generate class A biosolids.

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