



Original Article

Treatment of Waste Sludge: A Comparison between Anodic Oxidation and Electro-Fenton Processes

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ABSTRACT

Background: Electrochemical methods, as one of the advanced oxidation processes (AOPs), have recently been applied to remove different contaminants from water and wastewater. This study compares the performance of anodic oxidation (AO) and electro-Fenton (EF) methods on waste sludge treatment.

Methods: This experimental study was performed on real sludge and the effect of operating parameters such as solution pH, operating time, current density, supporting electrolyte and hydrogen peroxide concentration were investigated in a batch reactor. For determination of oxidation and treatability of the sludge, chemical oxygen demand (COD) and total coliform (TC) removal were examined. Pb/PbO₂ and iron electrodes respectively for AO and EF were applied.

Results: Experimental data indicated for both AO and EF as the operating time and current density increased, COD removal increased. pH=4.0 and 3.0 and current density=1.75 and 2 A respectively for AO and EF and the concentration = 57.2 mMol of hydrogen peroxide for EF were measured as the optimum amounts of these variables. The removal efficiency of COD in AO and EF process was 76% and 72%, respectively. Of course, the efficiency of EF in TC removal was better and the percentage of TC removal in 60 min for AO and EF was 99.0% and 99.9%, respectively. The amounts of consumed electrical energy for AO and EF were 8.6 and 28.0 kWh kg⁻¹ COD, respectively.

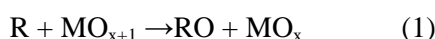
Conclusions: AO was more effective in treatment and mineralization of waste sludge and TC removal than EF in terms of environmental economical features.

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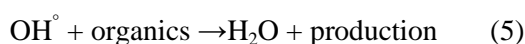
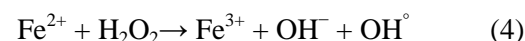
Introduction

Recently advanced oxidation processes (AOPs) have widely been applied to remove various pollutants particularly bio-refractory organic compounds^{1,2}; electro-Fenton (EF) and anodic oxidation (AO) are the two most common methods which have extensively been used to remove different pollutants from water and wastewater³⁻⁷. The two methods in which, in addition to direct oxidation of pollutants on the electrode's surface, pollutants are oxidized by indirect oxidation through free radicals in particular generated OH[°], as non-selective oxidants, have high oxidation power⁵⁻⁷. If the reactions are performed completely, organic matters are entirely destructed². In the case of AO, organic matters (R) are either directly oxidized on states oxides of the metal

(MO_{x+1}) or oxidized by means of adsorbed OH[°]; both mechanisms are dependent on the kind of electrode^{8,9} (Eqs. 1-3).



Hydrogen peroxide in EF is catalyzed by ferrous ion to generate OH[°] which is shown in Eq. 4^{10,11}.

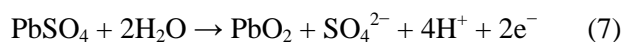


In recent years, on account of industrial and urban development and consequently the growth of wastewater treatment plants, a huge amount of sludge is produced; sludge must be treated because it contains pathogens and chemical contaminants as well as organic matters. Biological and chemical conventional techniques have some limitations as follows: They are affected by environmental conditions such as temperature, pH and so forth¹². They require a high detention time to complete degradation and microorganisms leave intact many persistent organic contaminants¹³. Some methods in which chemicals are added, not only they add extra chemicals to sludge economical, but also they lead to a high volume of sludge which is not reasonable in terms of environmental and disposal situations. Moreover, adverse by-products may be generated particularly when chlorine compounds are applied^{12,14,15}. However, on the other hand, in electrochemical ways the main reagents are electron and OH°. Moreover, they do not add and any adverse matter to solution and for the sake of environmental compatibility, versatility energy efficiency, safety, selectivity, amenability to automation, and cost effectiveness^{8,12,16} they are being addressed as alternative measures in sludge treatment; the AO and EF processes have enormous potential for this goal. To our knowledge, the application of the electrochemical method for treatment of sludge has not been seen before.

The object of this study was to investigate the efficiency of AO and EF processes in waste sludge treatment. In this regard, the effects of current density, operating time, supporting electrolyte, pH and hydrogen peroxide concentration were studied. These two ways were compared in terms of the removal amount of chemical oxygen demand (COD), total coli form (TC) and energy consumption.

Methods

This experimental study was carried out during September 2011 to September 2012. The used sludge was taken from the disposed sludge of the sedimentation tank of Tioran Company's wastewater treatment plant. All samples were transferred to the laboratory with polypropylene containers at the temperature of 4°C. Measured properties of raw sludge samples are as follows: COD=7150±750 (mg L⁻¹), conductivity =1500±100 (µS cm⁻¹), TC=15×10⁶±1×10⁶ (MPN/100 mL), pH=7.1±0.8. A 800 ml polyvinyl chloride electrolytic cell was used to perform electrolysis (effective volume was 500 ml). In the case of AO, Pb/PbO₂ electrodes were used. In order to prepare Pb/PbO₂, rod Pb were placed in sulfuric acid (10%) and for each cm² of the electrode surface, a current density of 10mA was passed for 90 min at the temperature of 25°C- which at the end of the time PbO₂ layer was formed on the surface of the electrode (Eqs. 6 and 7)¹⁷.



In the case of EF, iron electrodes were used. At both cases, dimensions of each electrode were 10 cm×1 cm, the electrodes were used in pairs by a monopolar arrange in the reactor (10 electrodes of each one were placed in the reactor). Effective electrode surface area was 200 cm². The distance separating the electrodes was fixed at 1.5 cm. They were connected to terminals of a direct current power supply (Adak, ps_405. Hamadan Kit Co. Iran) which is characterized by the ranges 0–5 A for current and 0–25 V for voltage. A constant stirring speed of 300 rpm was applied during all experiments. The experimental setup of EF and AO are shown in Figure 1.

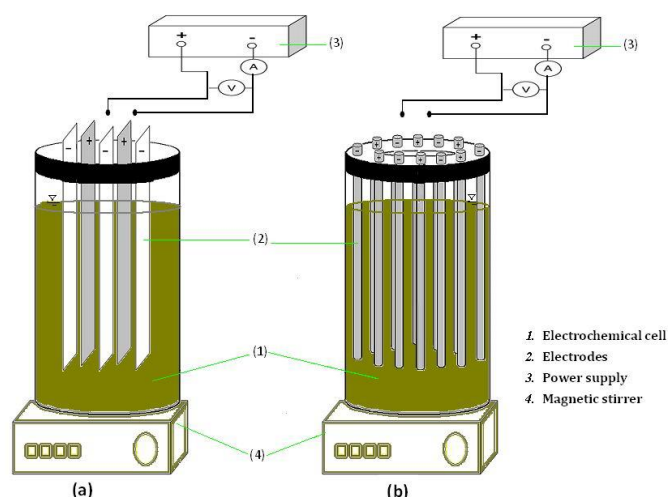


Figure 1: The experimental setup of electro-Fenton (a) and anodic oxidation (b) processes

Hydroxide sodium and sulfuric acid were used to adjust pH; NaCl (4, 6, 8, 10, 12, 14 and 16 g L⁻¹) and hydrogen peroxide (30% w/w) (22.9, 34.3, 45.7, 57.2, 68.6 and 80.1 mMol) were employed as supporting electrolyte and OH° source, respectively in EF. All chemicals were purchased from Merck Co. Germany. In this work, Fe²⁺ was electrically added to the solution as anode destruction. Sludge treatment was performed at the electrolytic cell with the following conditions: current density=0.5-3 A, pH=2-9 and operating time=15-120 min. A very important point to make here is that all parameters were optimized separately. So as to measure the efficiency of sludge treatment, the levels of COD and TC reduction were calculated by using Eq. 8¹².

$$R (\%) = ((X_i - X_t) / X_i) \times 100 \quad (8)$$

where, X_i and X_t are the concentrations of the COD (mg L⁻¹) and TC (MPN/100 mL) in the feed and the treated solutions, respectively.

The energy consumption (kWh kg⁻¹ COD) was calculated from Eq. 9¹⁸.

$$\text{Energy - consumption} = \frac{VIt}{3600 \times 10^3} \times \frac{1}{\Delta C \times V_R \times 10^{-6}} \quad (9)$$

Where V is the average cell voltage (V), V_R is the solution volume (L), ΔC is the difference in COD in mg

L^{-1} , I is the applied current (A) and t is the electrolysis time (S).

The COD and TC were measured according to the standard methods for the examination of water and wastewater¹⁹. Conductivity was measured by a lab Hach type conduct meter and pH was measured with a lab pH-meter (from Hach Co. Germany) electrometric method.

Results

The variables were optimized as one at a timemethod¹². First, bufferic pHs were used to investigate

Table 1: Effect of pH on chemical oxygen demand and total coliform removal by anodic oxidation and electro-Fenton processes with NaCl =10g L⁻¹ and H₂O₂ = 57.2 mMol

Parameters	pH							
	2	3	4	5	6	7	8	9
Anodic oxidation								
Current Density (A)	1.75	1.75	1.75	1.75	1.75	1.75	1.75	1.75
Operating Times (min)	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0
COD removal (%)	70.6	70.3	74.7	68.8	67.5	69.4	65.5	66.2
TC removal (%)	99.9	99.9	99.5	80.8	76.0	73.9	73.3	69.0
Electro-Fenton								
Current Density (A)	2	2	2	2	2	2	2	2
Operating Times (min)	120	120	120	120	120	120	120	120
COD removal (%)	70.8	71.9	70.5	68.1	57.8	59.2	54.1	51.7
TC removal (%)	99.9	99.9	99.8	95.8	86.0	83.9	83.3	79.0

A rise in current density led to a rise in COD removal. Based on Figure 2 at the current density of 1.75 A in the AO process, COD was reduced from 7440 mg L⁻¹ to 1910 mg L⁻¹; at current densities more than this amount, there was not a more removal efficiency in COD removal and it raised only energy consumption. In contrast, in the case of the EF process, the highest COD removal was attained at the current density of 2 A, which COD was reduced to 2174 mg L⁻¹ and when current density exceeded this figure, the amount of COD removal decreased- at current density of 2.5 A COD reached 2311mg L⁻¹. At this current density and more than this iron was disposed from the anode's surface as brown sediment at the bottom of the reactor, which did not participate in the reaction and it wasted.

There was no need to an additive matter at the AO process by comparison with the EF process. NaCl was applied as a supporting electrolyte in the EF process. COD removal without the supporting electrolyte was only 45%. By 11.5 g L⁻¹ of NaCl, which was selected as

Table 2: The effect of NaCl on chemical oxygen demand removal by the electro-Fenton process with operation condition including: Current Density=2A, Operating Times=120 min, H₂O₂=57.2 mMol, and pH=3

Parameters	Tests							
	1	2	3	4	5	6	7	8
NaCl (g L ⁻¹)	4	6	8	9	10	12	14	16
COD-influent (mg L ⁻¹)	7780	7780	7780	7780	7780	7780	7780	7780
COD-effluent (mg L ⁻¹)	3925	3880	3010	2678	2350	2140	2125	2090

In view of the fact that in EF hydrogen peroxide was injected to the reactor manually, according to Table 3 its optimum content was obtained at 57.2 mMol. The results showed COD removal fell when hydrogen peroxide

the pH variable effects. In the application of EF and AO methods, the highest COD removals were achieved at pH values of 3 and 4, respectively (Table 1), which were 72% and 76%, respectively. In both processes, COD removal decreased dramatically when pH value was over 5 or less 2. In both EF and AO, TC removal occurred under acidic conditions; over 99% of TC was removed when pH solution was less than 5. Of course, the EF process was more effective in TC removal than the AO process.

the optimum amount, COD removal reached to over 72% (Table 2).

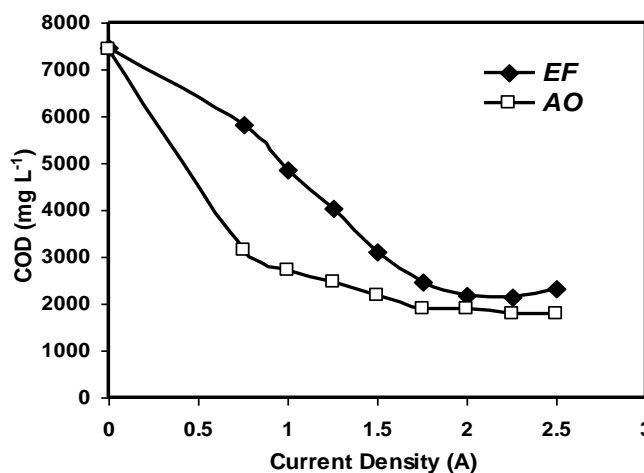


Figure 2: The effect of current density on chemical oxygen demand removal by anodic oxidation and electro-Fenton processes; charged passed= 3600 C for anodic oxidation; and charged passed=7900 C, NaCl=10g L⁻¹ and H₂O₂ = 57.2 mMol for EF process

the electro-Fenton process with operation condition including: Current

concentration was over 57.2 mMol. Therefore, other experiments were conducted at current densities of 1.75 A for AO and 2 A for EF and NaCl=11.5 g L⁻¹ and hydrogen peroxide=57.2 mMol only for EF processes, to

investigate the variables of operating time and energy consumption. As Table 4 shows, the AO process was better in COD removal than the EF process; the highest removal efficiency was achieved at operating times of 75 and 120 min, respectively for AO and EF. It must be pointed that both methods were entirely effective in TC removal (99.9%). Similarly, the use of operating times longer than the optimum amount it wasted in energy consumption and did not cause an increase in the removal efficiency and was not economical as well. After all variables were optimized, consumed energy was calculated by using equation 7 for both AO and EF processes (Figure 3). The results illustrated the AO process had both higher removal efficiency and less energy consumption compared to the EF process. The

amounts of consumed energy to reach the highest content of COD removal were 8.6 and 28.5 kWh kg⁻¹ COD, respectively for AO and EF. Of course, this amount of energy was enough to remove TC completely.

Table 3: The effect of H₂O₂ on chemical oxygen demand removal by the electro-Fenton process with operation condition including: Current Density=2A, Operating Times=120 min, NaCl=11.5g L⁻¹, and pH=3.

Parameters	Tests					
	1	2	3	4	5	6
H2O2 (mMol)	22.9	34.3	45.7	57.2	68.6	80.1
COD-influent (mg L ⁻¹)	7800	7800	7800	7800	7800	7800
COD-effluent (mg L ⁻¹)	4336	3245	2515	2213	2145	2101

Table 4: Effect of operating time on chemical oxygen demand and total coliform removal by anodic oxidation and electro-Fenton processes with operation condition including: Current Density=1.75 A and pH=4 for AO, Current Density=2A and pH=3, NaCl=11.5g L⁻¹ and H₂O₂=57.2mMol for EF

Parameters	Test							
	1	2	3	4	5	6	7	8
Anodic oxidation								
Operating Times (min)	15	30	45	60	75	90	105	120
COD removal (%)	18.3	39.8	59.8	72.7	76.2	76.1	77.3	77.5
TC removal (%)	84.4	89.8	95.1	99.0	99.8	99.9	99.9	99.9
Electro-Fenton								
Operating Times (min)	15	30	45	60	75	90	105	120
NaCl (g L ⁻¹)	11.5	11.5	11.5	11.5	11.5	11.5	11.5	11.5
COD removal (%)	11.1	28.3	38.8	49.8	59.3	68.2	71.9	72.3
TC removal (%)	98.8	99.1	99.5	99.9	99.9	99.9	99.9	99.9

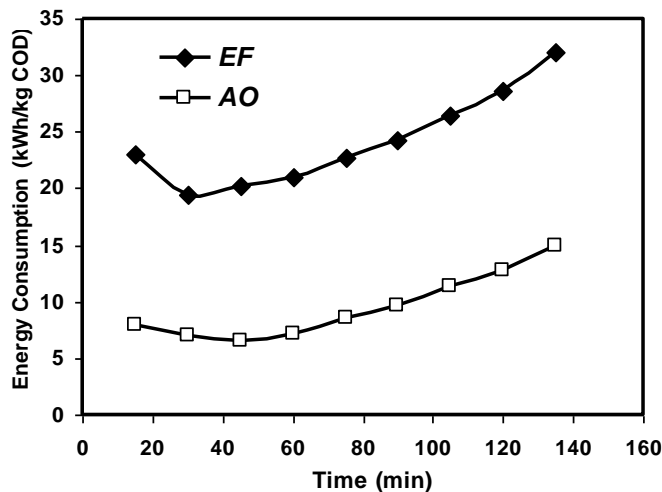


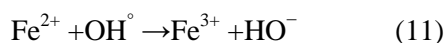
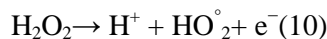
Figure 3: The energy consumption for anodic oxidation and electro-Fenton processes; operation condition: Current Density=1.75 A and pH=4 for anodic oxidation, Current Density=2A, pH=3, NaCl=11.5g L⁻¹ and H₂O₂=57.2 mMol for EF

Discussion

Under acidic conditions, both EF and AO had better removal efficiency to mineralize and treatment sludge. Different studies have suggested pH=3 for EF^{2,10,20}. According to Babuponnusami et al., an increase in efficiency in acidic pHs is on account of the fall in oxidation potential of HO[•] with a rise in pH. HO[•] is a weaker oxidant at near neutral pH than at acidic pH. When pH was raised beyond 7, HO[•] was quickly

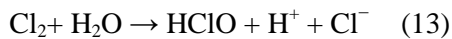
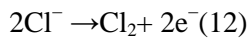
converted into its conjugate base O⁻ and it reacted more slowly than HO[•]¹⁰. Moreover, in the case of EF, the use of high pHs caused that Fe²⁺ transformed into Fe³⁺, which precipitated as brown sediment at the floor of the reactor and wasted without entering the reaction. This resulted in a decrease in removal efficiency. Under acidic conditions (pH value between 2.5 and 3.5), the degradation of hydrogen peroxide in order to generate OH[•] happens better²¹. However, the efficiency decreased at very low pH values (<2) which is because of the fact that hydrogen ion acts as HO[•] radical scavengers¹⁰.

Obviously, a rise in current density causes a rise in efficiency in accordance with Faraday law²², which is due to acceleration in direct oxidation of sludge on electrode's surface and more production of oxidant agents in the solution oxidizing sludge indirectly. However, if it exceeds the optimum amount (1.75 and 2 A for AO and EF, respectively), it will waste energy and consequently is not economical^{23,24}. This efficiency decline in high current densities is because of hydrogen peroxide oxidation on the surface of electrodes and the reaction between Fe²⁺ and OH[•] which Fe³⁺ is generated (see Eqs. 10 and 11)^{2,10,20,25}; in this case efficiency decreases.



In this study, the AO process was performed by Pb/PbO₂ electrodes, which are from the electrodes of dimensionally stable anodes (DSAs) and a "non-active"

anode⁸; the upside of using this electrode is cheapness and easy preparation. The process has a very good efficiency without using any extra chemicals. In contrast, in the EF process in order to improve the efficiency, NaCl, as a supporting electrolyte, is used to produce active chlorine that as OH[•] participates in organic matters oxidation (Eqs. 12 and 13)^{2,8}. It is required to optimize the concentration of hydrogen peroxide because the use of the amount more than the optimum amount not only does not take part in the reaction, but also reacts with OH[•] which hydroperoxyl radical (HO₂[•]) is generated that has low oxidizing ability^{2,11}. Applied hydrogen peroxide concentration depends on sludge concentration; the study Virkutyte et al. in which was found that the concentration of hydrogen peroxide is associated to wastewater quality²¹. The lack of pollutant removal after an optimal hydrogen peroxide concentration may be expressed by the competing reaction between hydrogen peroxide and OH[•] radicals when the hydrogen peroxide concentration is in excess²⁶.



There are different mechanisms involving in the two methods, but the main mechanism in both methods is sludge oxidation on the anode surface by direct oxidation and indirect oxidation through creating oxidants; in AO organic matters are oxidized by absorbed OH[•] and in EF they are oxidized by free hydroxyl radicals. Besides, over the reactions in the processes many intermediate reactions occur which are effective in sludge oxidation. Active chlorine ions are examples of these reactions that play a role in the oxidation^{8,11}.

By comparison with EF, AO consumed less energy for sludge treatment and COD removal. It is owing to the fact that sludge is treated in a less time (optimum operating time=75 min) and a lower current density (1.75 A) compared to EF (optimum operating time=120 and current density=2 A); in general, the AO had a better efficiency. However, in terms of TC removal both processes have a very good efficiency. Of course, it should be pointed the performance of EF is better which is because of the use of low pHs, production of active chlorine types that play an important role in germicide and the application of higher current densities.

In conventional systems (like perchlorine, electromagnetic wave, UV, and so forth) only disinfection takes place, but electrochemical processes are multiple because in addition to sludge oxidation they can remove microbes²². Previous studies have reported that these methods are entirely effective microbe removal^{22,27,28}. In the case of electro coagulation, microbe removal occur by means of adsorption and trapping among flocks and as well as potential difference originated from current density²⁷. Both AO and EF, in which there is potential difference and hydroxyl radical is

produced, have a noticeable efficiency in microbe removal.

There is the issue of Pb corrosion and its entrance into sludge. However it should be noted PbO₂ is resistance against corrosion; in this study the pure Pb was covered by a PbO₂ layer which raised the resistance. Corrosion is usually more at high current densities, but the range of which is between in which corrosion is little. Pb remained in the sludge lower than the standard value (≤ 0.005 mg/L). The application of the PbO₂ electrode was due to the following reasons: inexpensive material, commercially available and prepared easily and rapidly and as well as it has low resistivity, good chemical stability and a large area^{29,30}. Moreover, this electrode belongs to of dimensionally stable anodes (DSAs) electrodes; an example from this kind of electrodes is Boron doped diamond (BDD) having higher efficiency and lower corrosion, but this electrode is more expensive than the PbO₂ electrode.

Conclusion

The results of the study are indicative of the fact that either EF or AO can be applied to treat wastewater works sludge and both processes have an approximately similar efficiency even though AO has a slightly better efficiency. Moreover, the AO process does not require extra chemical matter and is less dependent on operating conditions. On the other hand, EF needs an appropriate supporting electrolyte such as NaCl and quite a few parameters are effective in treatment efficiency. Variables of current density, hydrogen peroxide content and pH value must be optimized precisely; otherwise, there is a decrease in efficiency, for example, in the case of AO the use of current density over the optimum amount does not result in a higher efficiency and in the case of EF it has reverse results and the efficiency goes down. The bottom line is that AO is preferable to EF because it results in higher mineralization, and consumes less energy; in addition to these, variables can comfortably be controlled.

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Conflict of interest statement

The authors declare that they have no conflicts of interest.

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