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# Employing Fered-Fenton advanced oxidation process for waste-activated sludge stabilization and reuse

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## ABSTRACT

In this study, the Fered-Fenton advanced oxidation process is utilized for sludge stabilization and reuse. According to US EPA, reductions in volatile solids (VS) and fecal coliforms are considered as the criterion of sludge stabilization. VS removal efficiency of 78±5%, 60±5% and 55±5% can be achieved at different pH levels of 3.0, 5.0, and 7.0, under operational conditions as follows: [Fe]/[$H_2O_2$] = 0.60, [$H_2O_2$] = 1250 (mg/L), retention time = 240 (min), and current density = 600–800 (mA). Moreover, Fecal coliforms decrease significantly (more than 99.9%) and according to pathogen reduction requirements (US EPA), the stabilized sludge samples are classified as class A. This sludge can be used for agricultural and forestall areas, lawns and home gardens. The effect of the Fered-Fenton process on nutrients in sludge was investigated. The average values of carbon/nitrogen (C/N) ratio, nitrogen, phosphorus, ammonium, nitrate, potassium, calcium and sodium reductions are 21.1%, 55%, 41.7%, 61.4%, 66.4%, 7.3%, 30.6% and 3.8%, respectively. It is assessed that the stabilized sludge at pH 7.0 has higher fertilizing quality than the same at pH 3.0 and pH 5.0. The results of the Fered-Fenton process indicate that this system, not only has a good ability to stabilize the sludge but also the output sludge could be used as a fertilizer in agriculture.

*Keywords:* Waste-activated sludge; Fered-Fenton method; Agricultural land application; Nutrients preservation; Fecal coliforms inactivation; Fertilizer

## 1. Introduction

Treatment and safe disposal of hazardous materials (waste, excess sludge, leachate, etc.) is an imperative and inevitable task in preserving the environment. Nowadays, wastewater treatment systems are used widely by activated sludge method, which results in producing a great amount of biological excess sludge that is a threat to human health [1]. The increasing growth of biological excess sludge has made it one of the most important and most critical environmental issues [2]. Management, consolidation and disposal of the sludge are considered as the key and strategic objectives in wastewater treatment systems [3]. The wastewater sludge contains hazardous substances, such as chemical organic materials, heavy metals, and pathogens which will enter the food chain and endanger human health if they are not stabilized and safely disposed [4]. The overall objective of stabilizing sludge includes the removal of the organic matter, heavy metals, pathogens and reduction of the sludge volume as well as converting them into useful sources [5]. Stabilized sludge can be reused as a fertilizer. The most common way of using the stabilized sludge is for land application [6].

The inability of conventional methods (aerobic, anaerobic. compost) in sludge oxidation and stabilization has led to new investigations [7]. Recently, employing advanced oxidation processes (especially the Fenton process) has become a major concern in wastewater treatment [8]. The performance efficiency of the advanced oxidation processes in degradation of biological organic compounds, sewage sludge contaminants, and soil pollutants has promoted the

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application of such methods [9]. Hence, they have been adopted by researchers for pretreatment of conventional methods [10,11], the disintegration of waste activated sludge [12,13] and the release of sludge organic carbon [14,15]. These procedures produce adequate hydroxyl radicals [8]. The hydroxyl radical which is one of the most impressive oxidizing agents in water and environment reacts non-selectively [8] ($E^0$ = 2.87) [16]. One of the most common advanced oxidation methods is Fenton process with advantages such as low initial cost, easy operation, and production of non-toxic byproducts [17]. Fenton reaction is the combination of hydrogen peroxide ($H_2O_2$) with iron (II) that leads to radical hydroxyl generation (reaction 1). One of the drawbacks of the Fenton process is that, the consumption rate of $Fe^{2+}$ ions (reaction 1) is faster than its reduction (reaction 2) [18]. The velocity difference between reaction 1 and 2 causes decreasing efficiency of Fenton process because the velocity of Fenton reaction 2 is limited. Also, for reduction of $Fe^{3+}$ ions to $Fe^{2+}$ ions some of hydrogen peroxide is spent (reaction 2). In this work, to overcome this drawback, the Fered-Fenton method is adopted.

The Fered-Fenton method is the combination of the electrochemistry and Fenton procedures. Reaction 1 in the Fered-Fenton process is similar to that of the Fenton. In the Fered-Fenton method, $Fe^{3+}$ ions are reduced to $Fe^{2+}$ ions in a cathodically through electrochemistry (reaction 3). Decreasing the consumption of iron and hydrogen peroxide and the increasing rate of the reaction are the advantages of the Fered-Fenton method compared to Fenton. This method has been adopted in many studies to eliminate the organic pollutants. Zhang et al., 2012, Wu et al., 2014 and Ye et al., 2016 utilized the Fered-Fenton process for removal of the organic loading from the landfill leachate [19–21].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\bullet} + OH^{-} \qquad 53 - 76 M^{-1}S^{-1} \tag{1}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H\,O_2^{\bullet} + H^{+} \qquad 0.01 - 0.02\,M^{-1}S^{-1} \tag{2}$$

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

Assessing the effect of the Fered-Fenton advanced oxidation process on sludge stabilization is the first objective of this study. Here, the VS and fecal coliform removal are chosen as a criterion for stabilizing the sludge and the effects of important parameters in the Fered-Fenton process (including pH, time, current density, the ratio of [$Fe^{2+}$/$H_2O_2$] and the concentration of $H_2O_2$), are assessed. Investigating the fertilizing value and the reuse possibility of the stabilized sludge with the Fered-Fenton advanced oxidation process is the second objective of this study.

## 2. Material and methods

### 2.1. Experimental setup

The assessments were run in a laboratory scale setup. The reactor cell is made of Plexiglass with a volume of 0.9 L. Graphite electrodes (i.e. two anodes and two cathodes) are chosen because of their chemical inert and decomposable properties. The electrodes of size 1 mm × 140 mm × 60 mm, were placed at a about 1.5 cm from each other [22] and 100 mm deep into the sludge with the contact surface of 100 mm × 60 mm. The sludge is continuously mixed using an electric mixer (Zheng, zs-ri, 6(V), DC, 300 rpm). A digital laboratory model power supply (Mps, DC-3003D, 0-3 (A), 0-30 (V)) is used for the amperage adjustment.

### 2.2. Materials

The chemicals consumed in this process consist of hydrogen peroxide (35%) as Fenton reagent and ferrous sulfate ($FeSO_4{\cdot}7H_2O$) as well as concentrated sulfuric acid and sodium hydroxide (soda) for pH adjustment. The chemicals are the products of the Merck® Co and Whatman® filter papers are used for filtering the treated samples (no. 42).

### 2.3 Raw sludge sample

The sludge samples were collected from Mahallati wastewater treatment plant in north of Tehran, Iran. The overall characteristics (the annual averages) of the samples are presented in Table 1.

### 2.4 Procedures and measurements

The pH values of the samples are adjusted by adding the adequate dosages of sulfuric acid and sodium hydroxide. Then, ferrous sulfate, hydrogen peroxide, and Fenton's reagents ($FeSO_4$) and hydrogen peroxide ($H_2O_2$) are added to the reactor cell. Thereafter, graphite plate electrodes are connected to the laboratory model DC power supply system and voltage and current are adjusted. Stabilized WAS samples are analyzed by means of analytical standards methods for the examination of water and wastewater [23].

Table 1
The characteristics of the raw sludge samples

| Parameter | Range |
|---|---|
| pH | 6.53–7.31 |
| Temperature (°C) | 13.5–15.7 |
| Chemical oxygen demand (COD) (mg/L) | 5768–10985 |
| Soluble chemical oxygen demand (SCOD) (mg/L) | 38–67 |
| Volatile solids (VS) (mg/L) | 3136–6682 |
| Total solids (TS) (mg/L) | 4375–8825 |
| VS/TS | 0.73–0.77 |
| Fecal coliforms (MPN per gram) | $(4–4.5)\times10^{6}$ |
| Total coliforms (MPN per gram) | $(3.9–4.1)\times10^{7}$ |
| $N\text{-}NO_3$ (mg/kg) | 756–945 |
| $N\text{-}NH_4$ (mg/kg) | 5991–7012 |
| Total phosphor (TP) (% of dry matter) | 2.5–3.5 |
| Total nitrogen (TN) (% of dry matter) | 2.2–3.1 |
| Total carbon (TC) (% of dry matter) | 38.9–50 |
| Potassium (K) (% of dry matter) | 0.45–0.6 |
| Sodium (Na) (% of dry matter) | 0.63–0.92 |
| Calcium (Ca) (% of dry matter) | 6.1–6.7 |

In order to measure COD, Na, Ca, K, TP, TN, $NH_4^+$, $NO_3^-$, solids (VS and TS), fecal and total coliforms, towards standard methods, the 5220D, 3500-Na, 3500-Ca, 3500-K, 4500-P, 4500-N, 4500-$NH_3$, 4500-$NO_3^-$, 2540 and 9221 are applied, respectively. All experiments were repeated three times.

## 3. Results and discussion

### 3.1. Effective parameters on VS removal

pH is one of the most important and efficient parameters in Fenton and Fered-Fenton processes. In this experiment, the oxidation processes are studied under different pH conditions (1.0 up to 8.0). The reactors laboratory assessments show that the maximum efficient VS removal of up to 68% happens at pH of 3 (Fig. 1 diagram a). Furthermore, it is reported that the efficiency of the system will decrease with the pH of less than 3, so that with pH =1 it drops to 54%. At the lower values of pH (pH <3), the iron species form stable complexes with $H_2O_2$, and as a result, the neutralization of iron catalysts occurs and the efficiency of the oxidation decreases considerably [22].

The other factor which makes the VS removal less efficient is the consistency of the hydrogen peroxide in the form of $H_3O_2^+$ [24], which results in the disinvolvement of the $H_2O_2$ in the reaction 1 and less hydroxyl radical being generated. On the other hand, by increasing pH from 3 to 5, the efficiency of the laboratory system drops to 58% in pH 5.0. Further, with the pH of more than 5.0, because of the unstable situation of hydrogen peroxide and its transformation to water and oxygen, the efficiency of the system reduces even faster. So that, at pH 7.0, the VS removal efficiency decreases to 49% [25]. pH 3.0 is chosen because the maximum removal of VS happens at this level of pH. pH 5.0 is a good choice because of the possibility of using it in the natural situation and pH 7.0 can be easily used in the oxidation process since it does not require additional chemical matters to adjust the pH level.

Increasing the electric current density results in the transformation of iron $Fe^{3+}$ to iron $Fe^{2+}$. Thus, Fered-Fenton process will be more effective and the reaction 3 takes place [26]. As it is shown in Fig. 1 diagram b, when there is no current in the laboratory system, in practice only Fenton process exists and the efficiency of the system will be 22% (at pH 3.0), 17% (at pH 5.0) and 15% (at pH 7.0). After applying electric current in the system the efficiency increases and within the range of 600–800 mA the VS removal efficiency reaches to 72% in the best situation. On the other hand, increasing the current density to more than 800 mA leads to the reduction of the hydrogen on the cathode electrodes' surface and prevents the reduction of iron, thus, the VS removal deteriorates. The reason that the range of 600–800 mA was chosen for the current density is related to the sludge electrical resistance, which depends on the sludge VS itself. In addition, by changing VS, the range of the current density which is directly related to the electrical resistance will change subsequently. The VS removal efficiency is optimum at 600–800 mA of current density. In the optimum conditions the current density is 800 mA but over time the amount of the current density reduces and by the end of the process it reaches to 600 mA.

The dosage of the Hydrogen peroxide is another factor involved in the process of oxidation. Fig. 1 diagram c shows the impact of hydrogen peroxide on VS removal. By increasing the concentration of the hydrogen peroxide according to the reaction 1, hydroxyl radical is produced and VS removal increases. This process continues for the concentration of up to 1250 mg/L which eventually reaches a removal efficiency of 74% (at pH 3.0), 60% (at pH 5.0) and 53% (at pH 7.0). Subsequently, the efficiency of the laboratory system decreases by increasing the hydrogen peroxide concentration. This process is due to the consumption of hydroxyl radicals by the extra hydrogen peroxide in the system which reduces the VS removal efficiency [27].

The effect of $Fe^{2+}/H_2O_2$ ratio in the efficiency of VS removal is also considered. By increasing the $Fe^{2+}/H_2O_2$ ratio, the amount of $Fe^{2+}$ in the media increases and the hydroxyl radicals are produced. Thus, the VS removal happens. The maximum VS removal efficiency will occur at $Fe^{2+}/H_2O_2 = 0.6$. However, the excessive increase in the ratio of $Fe^{2+}/H_2O_2$ results in the oxidation of $Fe^{2+}$ by the hydroxyl radicals and the efficiency of the VS removal decreases consequently (Fig. 1 diagram d).

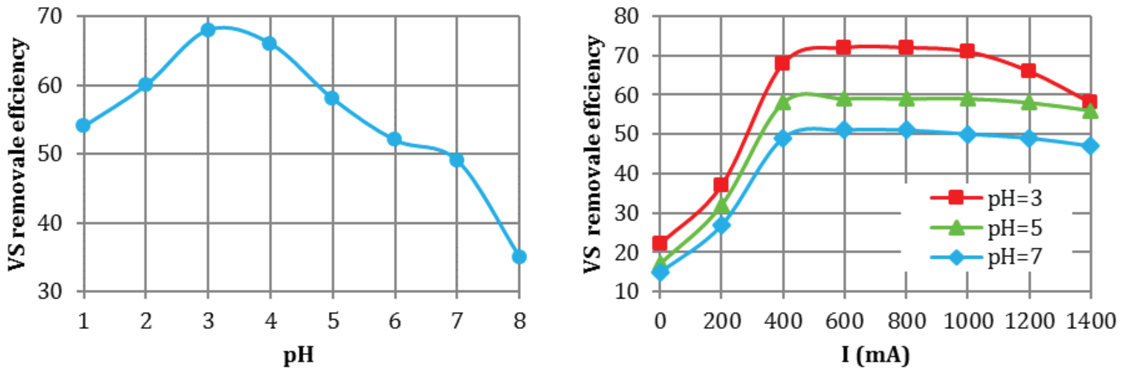
The retention time, due to more energy consumption in the Fred-Fenton system, is also of great importance and optimizing it lowers the level of energy consumption. As Fig. 1 diagram f shows, the maximum rate of VS removal efficiency will be reached within the first two hours of the test, which is approximately up to 50%. This is due to the high concentration of the biodegradable organic matters in the laboratory system. The efficiency of the laboratory system in the next two hours reaches to 78% (at pH 3.0), 61% (at pH 5.0) and 55% (at pH 7.0), and after that, no significant changes are observed. Fered-Fenton process is more powerful than Fenton process in terms of oxidation, and its efficiency in complex organic compounds removal is much more than Fenton's [28]. In this study, it was observed that sludge VS removal efficiency in Fered-Fenton process is three times more than that in Fenton process.

### 3.2. Fertilizing property preservation

The efficiency of the Fered-Fenton oxidation process in preserving the fertilizing property of the sludge is assessed through the total carbon (TC) and inorganic elements concentrations like: $NO_3^-$, $NH_4^+$, TN, TP, Na, K, and Ca. The TC concentration is considered as an indicator of the existing organic material.

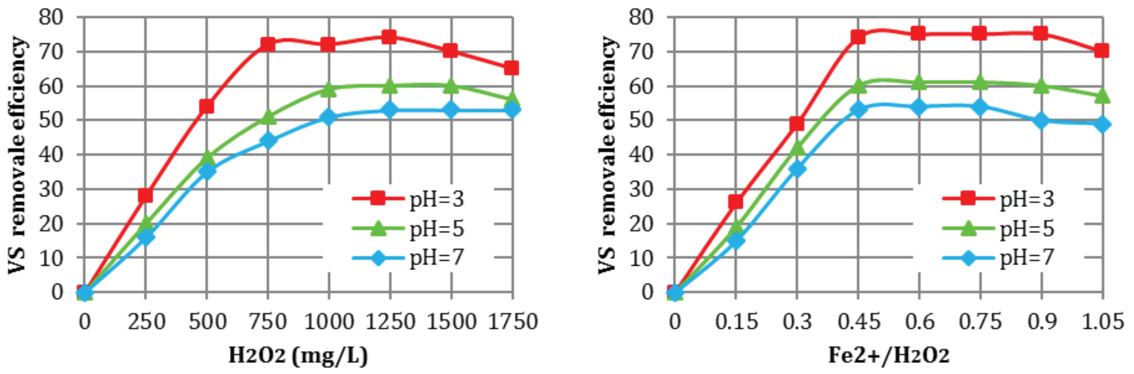
The existing organic matters in the soil improve the physical, chemical, and biological properties which are necessary to ensure the soil fertility [6]. The main agent of the organics oxidation is the generated hydroxyl radicals (OH·) through the Fenton reaction [Eq. (1)]. It is shown by several studies that the most efficient Fenton process occurs at pH 3.0 [22]. In this study, the maximum removal efficiency is 79.6% at pH 3.0. The removal efficiency decreases with an increase in the pH level (e.g. 45.7% at pH 7.0). This can be justified with respect to a decline in OH· production during Fenton reaction (Fig. 2).

In this study, the nitrogen content of the raw and treated waste-activated sludge has been considered as nitrate ($NO_3^-$) and ammonium ($NH_4^+$) ions. The maximum removal of the nitrate (i.e. 83.8%) and ammonium (i.e. 73%) happens at pH



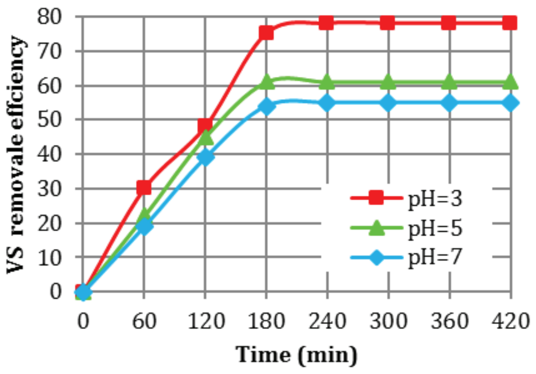
**(a).** The relationship between pH and VS removal efficiency, constants: a hydrogen peroxide concentration of 1000 mg/L, a current density of 400 mA, the ratio of [$Fe^{2+}/H_2O_2$]=0.45, a retention time of 180 min.

**(b).** The relationship between current density and VS removal efficiency, constants: a hydrogen peroxide concentration of 1000 mg/L, the ratio of [$Fe^{2+}/H_2O_2$]=0.45, a retention time of 180 min.



**(c).** The relationship between peroxide hydrogen concentration and VS removal efficiency, constants: a current density of 600-800 mA, the ratio of [$Fe^{2+}/H_2O_2$]=0.45, a retention time of 180 min.

**(d).** The relationship between [$Fe^{2+}/H_2O_2$] ratio and VS removal efficiency, constants: a current density of 600-800 mA, a hydrogen peroxide concentration of 1250 mg/L, a retention time of 180 min.



**(f).** The relationship between retention time and VS removal efficiency, constants: a current density of 650 mA, the ratio of [$Fe^{2+}/H_2O_2$]=0.60, a hydrogen peroxide concentration of 1200 mg/L.

Fig. 1. The relationship between VS removal efficiency and the parameters affecting the operation of the Fered-Fenton system.

3.0. An increase in the pH value (up to 7.0) causes a reduction in the nitrate and ammonium ions removal efficiency (i.e. 52.3% and 51.1%) (Figs. 2 and 3). The main cathodic reactions by the electrochemical reduction of nitrate ions to nitrogen gas and ammonia in acidic solution have been investigated by Paidar et al. [29], Kim et al. [30] and Mook et al. [31].

The initial phosphorus content in raw sludge was 2.97 ± 0.1 % of dry matter. The maximum removal (26–35%) happens at pH 3.0 by adding appropriate quantities of sulfuric acid for pH adjustment. This could be explained by P precipitation in the form of ferric phosphate, thus, the generation of $Fe^{3+}$ due to the Fenton reaction [Eq. (1)] at high pH values results in enhancing the sludge phosphorus content (Fig. 2). The phosphate in the sludge is coagulated by iron. Some amounts of the coagulated iron sediments on the electrodes' surface and removes from the sludge. Then, the remaining coagulated phosphate in the sludge cake is measured.

The initial content of the micronutrients, (i.e. Na, K, and Ca) in the raw sludge was 0.87 ± 0.05, 0.59 ± 0.02, and 6.40 ± 2% of dry solids, respectively. A limited removal of the Na and K ions occurs during the treatment process (i.e. 2.3-5.7% for Na and 11.8–13.5% for K), while Ca is significantly removed (i.e. 23.4–37.3%) (Fig. 4).

The C/N ratio, which is an important parameter to assess the total nutrient balance, is considered as one of the parameters to represent the fertilizing property of the stabilized waste-activated sludge samples [6]. The C/N ratio of the treated sludge must be well-balanced in order to assure its stability once applied to the crop fields. The stability threshold ranges between 10 and 20 according to the several studies conducted in considered wastes [32]. In this study, C/N ratios indicates a clear decrease in the raw sludge (i.e. 14.5) in comparison to the treated sludge at different pH values, essentially due to the efficient TC removal by the means of the Fered-Fenton process. However, at pH value of 3, the ratio decreases to 8.47. The maximum C/N ratio, (i.e. 13.7) is achieved at pH 7 (Fig. 2), suggesting an appropriate fertilizing volatilization for agricultural land applications.

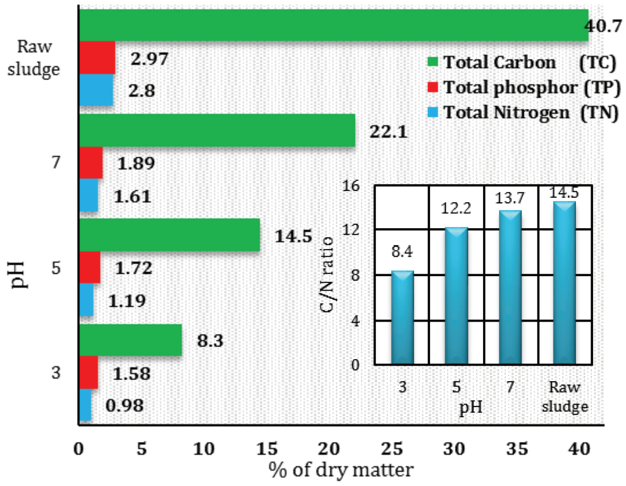


Fig. 2. Total carbon, total phosphorus, and total nitrogen content, and C/N ratio of the stabilized waste-activated sludge at different operating pH values. $[Fe^{2+}]/[H_2O_2] = 0.60$, retention time = 240 min, $[H_2O_2] = 1250$ mg/L, current density = 600–800 mA.

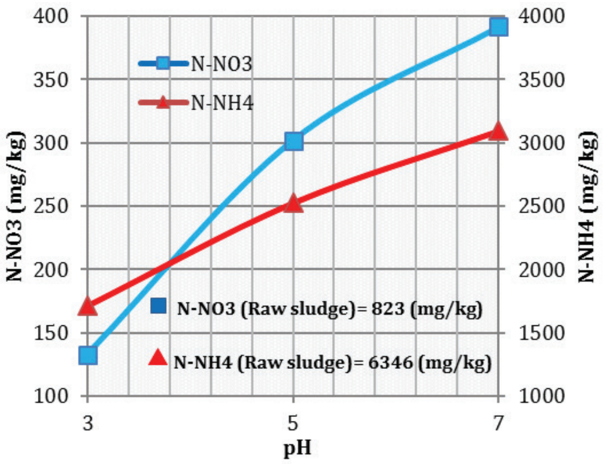


Fig. 3. Variations of Nitrate and ammonium under different operating pH values (3, 5 and 7), $[Fe^{2+}]/[H_2O_2] = 0.60$, retention time = 240 min, $[H_2O_2] = 1250$ mg/L, current density = 600–800 mA.

### 3.3. Fecal coliforms removal

The spread of diseases as a consequence of using pathogenic bacteria contaminated waste sludge in agricultural land is a serious limiting factor in this regard [33]. According to the US EPA (40 CFR Part 503) regulations, Class A bio-solids contain a fecal coliforms density less than 1,000 most probable number (MPN) per gram total

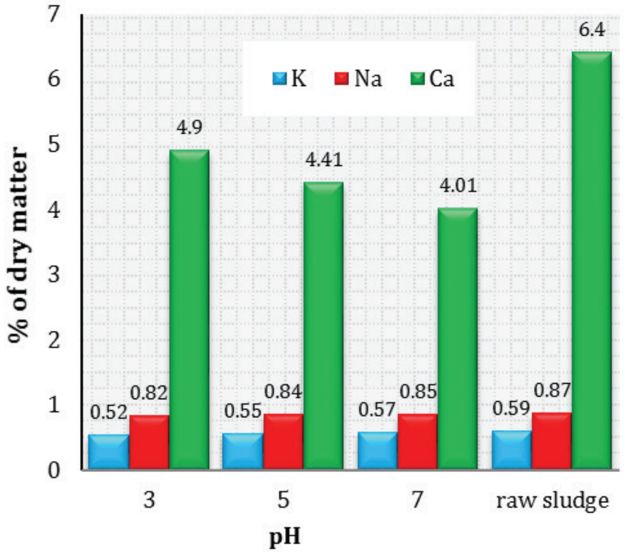


Fig. 4. Micronutrients content of the stabilized waste-activated sludge samples at different operating pH values $[Fe^{2+}]/[H_2O_2] = 0.60$, retention time = 240 min, $[H_2O_2] = 1250$ mg/L, current density = 600–800 mA.

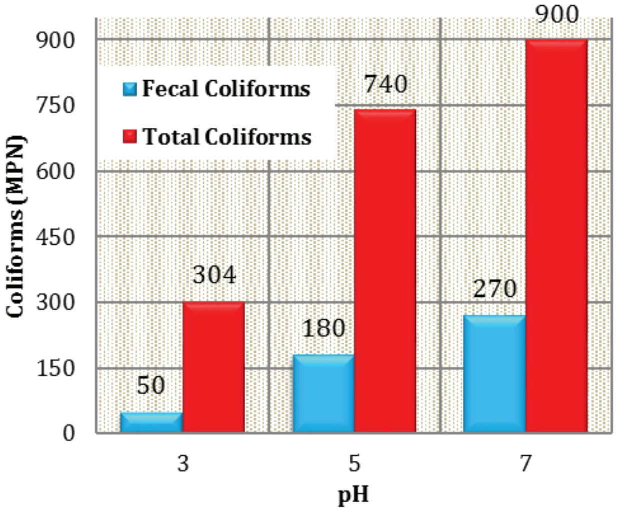


Fig. 5. Efficiency of removal Coliforms at constant conditions: pH values (3, 5 and 7), $[Fe^{2+}]/[H_2O_2] = 0.60$, retention time = 240 min, $[H_2O_2] = 1250$ mg/L, current densit y= 600–800 mA. Initial fecal coliforms concentration in the raw WAS = $4.25 \times 10^6$ MPN per gram total solids, initial total coliforms concentration = $4 \times 10^7$ MPN per g total solids.

solids [34]. The removal efficiency (i.e. more than 99.9%) of the assessed Fered-Fenton reactor regarding fecal coliforms and total coliforms present in the waste-activated sludge samples are shown in Fig. 5. The obtained results at different operating pH levels of 3.0, 5.0, and 7.0 correspond to the class A requirement for sludge land application purposes. The generated hydroxyl radicals Eq. (1) constitute the main affecting agent related to the inactivation of a wide range of bacteria and viruses [35].

### *3.4. Fertilizer quality of stabilized sludge*

The fertilizer quality of the stabilized sludge samples in optimum conditions and at the different levels of pH 3.0, 5.0 and 7.0 are studied. Here, the fertilizer quality is considered based on the nitrogen and applying Eq. (4) [36]. For this purpose, it is assumed that the sludge which is introduced to the agricultural land is dewatered ($k_v = 0.75$) and the agricultural land is located in a warm climate ($f_m = 0.4$). The amount of the available nitrogen for the plant from the stabilized sludge at different pH levels (3.0, 5.0 and 7.0) is equal to 3.32, 3.64 and 5.07 g/kg dry matter respectively (Table 2). We find out that the stabilized sludge as the result of natural conditions is of higher fertilizing quality than other stabilized sludge samples.

$$N_{PA} = [(NH_4^+)k_v + NO_3 + (N_o)\,f_m]F \tag{4}$$

where $N_{PA}$ = plant available nitrogen in the application year, g/kg; $NH_4^+$ = ammonia nitrogen in biosolids, %; $k_v$ = volatilization factor for ammonia, = 0.5 for surface-applied liquid biosolids, = 0.75 for surface-applied dewatered biosolids, = 1.0 for injected liquid or dewatered biosolids; $NO_3$ = nitrate nitrogen in biosolids, %; $N_o$ = organic nitrogen in biosolids, %; $f_m$ = mineralization factor for organic nitrogen, = 0.5 for warm climates and digested biosolids, = 0.4 for cool climates and digested biosolids, = 0.3 for cold climates and composted biosolids; F = conversion factor, 1000 g/kg of dry solids.

Table 2
Fertilizer quality of stabilized sludge

| Sludge | $NH_4^+$ (%) | $NO_3$ (%) | $N_o$ (%) | $N_{PA}$ g/kg of dry solids |
|---|---|---|---|---|
| pH = 3 | 0.17 | 0.013 | 0.797 | 3.32 |
| pH = 5 | 0.25 | 0.03 | 0.91 | 3.64 |
| pH = 7 | 0.31 | 0.04 | 1.26 | 5.04 |

## 4. Conclusions

In this article, the effect of the Fered-Fenton process on stabilizing wastewater sludge is assessed. It is found that the Fered-Fenton process has an efficient ability to stabilize the sludge and remove the volatile solids at different pH levels of 3.0, 5.0 and 7.0. Moreover, the quality of the stabilized sludge produced through this process is very suitable in terms of fecal coliform. Assessing the fertilizer quality of the stabilized sludge by this method shows that the stabilized sludge at pH 7.0 contains the most amounts of its nutrients and has a better fertilizing value compared to the other pH levels. It should be mentioned that, not only the Fered-Fenton process has the ability to stabilize the sludge but also the stabilized sludge could be reused as a high-quality fertilizer in the natural conditions.

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