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Comparison of classical Fenton and Fenton-like using μ-ZVI processes for the degradation of cresol in the wastewater

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ABSTRACT

The aim of this study was to investigate the applicability of a Fenton-like process using micron size zero valent iron (μ-ZVI) catalyst for the degradation of cresol in the wastewater, and the comparison of the degradation and the detoxification of cresol through this method and the classical Fenton process. The experiments were conducted in a batch reactor to examine the effects of pH, ZVI concentration, hydrogen peroxide concentration and temperature on the degradation of a wastewater containing 18.5 mM tricresol. The optimal reacting conditions were found to be pH = 2, [ZVI] = 5.35 g/L, [H_2O_2] = 472 mM and temperature = 30°C. The optimum conditions of the Fenton-like process were affected by corrosion phenomena and were different from the classical Fenton process. The evolutions of pH, cresol concentration, products concentration, dissolved iron concentration and chemical oxygen demand (COD) removal at the optimum conditions during the process were determined. At t = 120 min the efficiency of the degradation reaction reached at the maximum level (87.7%). The degradation efficiency obtained by the Fenton-like process is significantly higher in comparison with that achieved by the classical Fenton process (81.6%) at its optimum conditions. 85.8% of ZVI was recoverable by filtration after each experiment. The reusability of the recovered catalysts was also determined in 10 repetitive cycles (average degradation efficiency = 87.46%). Finally, bioassay tests for initial and treated wastewaters were conducted to evaluate their phytotoxicity on *Lepidium sativum* L. The phytotoxicity results were found to be in good accordance with the COD removals.

Keywords: Fenton-like process; μ-ZVI; Cresol; Degradation; Phytotoxicity

1. Introduction

Cresol or methylphenol (Fig. 1) is used as a starting material for the synthesis of herbicides, insecticides, resins, antioxidants and pharmaceuticals [1]. The major sources of cresol in the environment are wastewater from coal gasification, fractional distillation of coal tars, petroleum refineries and phenolic resin industries [2–4]. Cresol is a persistent compound and is toxic to a wide spectrum of organisms. In 1979, cresol was classified by the Environmental Protection Agency (EPA) as persistent, toxic and a priority [5]. In recent years, the EPA has classified cresols within group C: possible human carcinogens [6].

In the last two decades, different physical, biological and chemical processes were studied for the removal of cresol from wastewater. The most investigated physical method is the adsorption of cresol on the adsorbents [7,8]. Several microorganisms (bacteria and fungi) utilize cresol as a sole

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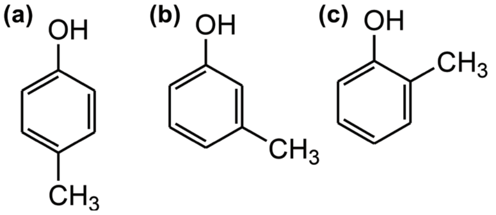

Fig. 1. Structure of (a) para-, (b) meta- and (c) ortho-cresol isomers.

carbon source in spite of its toxicity [9–11]. The chemical treatment methods include conventional oxidation [12] and advanced oxidation processes (AOPs). AOPs consist of near ambient temperature and pressure water treatment processes which involve the generation of hydroxyl radicals ($HO^•$) in sufficient quantity to affect water purification [15]. One of the most important AOPs is the Fenton process, which is based on the generation of hydroxyl radicals from hydrogen peroxide with ferrous (Fe^{2+}) ions acting as homogeneous catalysts at acidic pH and ambient conditions (Eqs. (1) and (2)) [13].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^• + OH^- \quad (1)$$

$$HO^• + \text{organics} \rightarrow \text{oxidized products} \quad (2)$$

Kavitha and Palanivelu [14] investigated cresol degradation by the Fenton process. They observed that the degradation efficiency for cresol isomers was 82% within 120 min at optimum conditions. Our previous study had assessed chemical, biological and ecotoxicological characteristics of cresol and its Fenton's degradation products [15].

The Fenton process is an old method for wastewater treatment. In the last few decades several modification of this method were proposed and studied by different researchers. One of the modified Fenton or Fenton-like processes is the use of zero valent iron (ZVI, elemental iron) instead of ferrous salts. In this method, the following additional reactions occur [16,17]:

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2 \quad (3)$$

$$2Fe^{3+} + Fe^0 \rightarrow 3Fe^{2+} \quad (4)$$

In acidic conditions, the surface of the metal iron corrodes giving rise to ferrous ions and hydrogen gas (Eq. (3)) then ferrous ions react with hydrogen peroxide (Eq. (1)) to produce hydroxyl radical and ferric ions. The ferric ions react with metal iron and regenerate ferrous ions catalyst (Eq. (4)). Particular advantages of this process are the cost-savings due to the use of metal iron rather than iron salts and it was proposed faster recycling of ferric iron at the iron surface through Eq. (4) [17,18].

The Fenton-like process using ZVI has been applied, in the last decades, on the remediation of several wastewaters such as olive mill phenolic effluents [19,20], textile industries wastewaters [21–24] and landfill leachates [25–28]. Lucking et al. [29] reported the oxidation of 4-chlorophenol using iron powder (70–100 μm) and hydrogen peroxide. Namkung et al. [16] and Bremmer et al. [17] developed a Fenton-like process for the degradation of phenol involving the oxidation of iron metal sheets (Fe^0) at acidic conditions generating in situ Fe^{2+}, which will promote hydrogen peroxide decomposition into hydroxyl radicals.

The objective of this work is to investigate the applicability of the Fenton-like process using micron size zero valent iron (μ-ZVI) catalyst for the degradation of cresol in wastewater and the comparison of the degradation and the detoxification of cresol through this method and the classical Fenton process.

2. Materials and methods

2.1. Materials

Technical grade tricresol (97%, mixture of isomers) and industrial grade iron powder (95%, 30 μm) were supplied by Sepahan Chime (Esfahan, Iran) and Ariapowder (Mashad, Iran), respectively. Technical grade hydrogen peroxide (35%), analytical grade sodium hydroxide (99%) and sulfuric acid (97%) were used. These reagents were supplied by Merck KGaA (Darmstadt, Germany). For pH adjustments, sulfuric acid and sodium hydroxide 20% solutions were used. Phytotoxicity tests were carried out with garden cress seeds (*Lepidium sativum* L.) from a local source.

2.2. Experimental procedure

To optimize the Fenton-like process, 1,000 mL solutions containing 2 g/L (18.5 mM) cresol in distilled water with proper pH were prepared in a 2,000 mL Pyrex glass beaker. Immediately after adding ZVI to these solutions, the reaction was initiated by adding the proper amount of hydrogen peroxide to the reactor that was equipped with a temperature-controlled water bath. Before starting the reaction, the solution was stirred by a mechanical stirrer (500 rpm). After 120 min (for optimization tests) or after predefined times, samples were withdrawn from the reactor and immediately quenched by adding sodium hydroxide solution (20%) to raise their pH to 12 and were heated at 40°C in a closed vial for 120 min to decompose any residual hydrogen peroxide and to avoid interference of hydrogen peroxide on the chemical oxygen demand (COD) measurements. The absence of sensible hydrogen peroxide was controlled prior to the COD measurements of treated samples. The samples for dissolved iron concentration did not quench to prevent precipitation of iron as iron hydroxides.

2.3. Analysis

The initial pH of the solution was measured using WTW pH meter inoLab 720 (Germany). For COD analysis, all the samples were immediately quenched by adding sodium hydroxide solution (20%) as described above. The hydrogen peroxide concentrations in treated samples were monitored by iodometric method. The COD of the samples was measured according to standard methods of APHA [30]. Specific quantitative analysis of cresol was carried out in High Performance Liquid Chromatography using Knauer K2500 HPLC, with a C18 (250 × 4.6 mm) column and NB166 UV detector. Mixtures of water/methanol/acetic acid (59.4/39.6/1 v/v) were used as mobile phase at a flow rate of 0.8 mL/min. The quantitative determinations of the products (acetic acid and oxalic acid) were carried out in Metrohm Ion Chromatography 861 Advanced Compact (Switzerland) with a Metrosep A Supp 5 (150 mm long) as analytical column. The isocratic mobile phase consisted of 4.5 mM Na_2CO_3 and 1.5 mM $NaHCO_3$, operated at a flow rate of 0.8 mL/min. The retention times for the acetate and oxalate ions were detected at 4.00 and 11.86 min, respectively. The concentrations of the dissolved iron were analyzed using Perkin Elmer AAnalyst 800 Atomic Absorption Spectrometer.

Phytotoxicity tests were conducted in order to evaluate the phytotoxicity of cresol and its degradation products using an indirect acute toxicity bioassay on the dicotyledonous plant *Lepidium sativum* L. according to a standardized protocol [31]: 3 d of seed germination and root growth tests were conducted using the dicotyledonous garden cress *Lepidium sativum L.* as an indicator. The toxicity tests were conducted using 20 seeds each for germination on Petri dishes. The pH of the all samples was neutralized to 7 then 3 mL of these solutions and also the diluted solutions (1:10) were used to wet the paper supporting the seeds. The Petri dishes were kept at room temperature (25°C–30°C) for 72 h. The number of seeds germinated, stem length and root length were measured to determine the relative bioassays and germination index (GI).

3. Results and discussion

3.1. Degradation of cresol by the classical Fenton process (for comparison)

The overall degradation of cresol was monitored by COD measurements and degradation efficiency was calculated as follows:

$$\text{Degradation efficiency} = \frac{\text{COD}_{\text{initial}} - \text{COD}_{\text{sample}}}{\text{COD}_{\text{initial}}} \tag{5}$$

The classical Fenton process for the degradation of cresol was performed in a similar procedure as described by Kavitha and Palanivelu [14] except that technical grade tricresol (mixture of isomers) was used instead of analytical grade separated isomers of cresol and also 10 times more concentrated cresol solution was used (18.5 mM instead of 1.85 mM). Experimental conditions are presented in Table 1. In these conditions, we observed 81.6% degradation efficiency for cresol.

Table 1
Experimental conditions for the classical Fenton process (for comparison)

[Tricresol], mM	18.5
[H_2O_2], mM	315
[$FeSO_4.7H_2O$], mM	7.91
pH	3.0
T, °C	30
t, min	120

3.2. Optimization of the reaction conditions for the Fenton-like process

In this section, the effects of operating variables including ZVI concentration, pH, hydrogen peroxide concentration and temperature on the degradation efficiency of cresol for the Fenton-like process were determined.

3.2.1. Effects of ZVI concentration and initial pH

Both ZVI concentration and initial pH have strong effect on generation of ferrous ion (Fe^{2+}) in the solution, which has the catalyst role in the Fenton-like reactions. Therefore, in this paper ZVI concentration and pH effects were studied simultaneously. Four series of batch experiments were conducted to determine the effects of ZVI concentration and initial pH of solution on the degradation efficiency. In these experiments, cresol concentration, hydrogen peroxide concentration and temperature were 18.5 mM (2 g/L), 315 mM and 30°C, respectively. In this section, the molar ratio of hydrogen peroxide to cresol (315 mM/18.5 mM = 17) was selected due to theoretical reaction of cresol and hydrogen peroxide (Eq. (6)):

$$C_7H_8O + 17H_2O_2 \rightarrow 7CO_2 + 21H_2O \tag{6}$$

The effects of these parameters are shown in Fig. 2 (2D and 3D). The hydrogen peroxide to ZVI mass ratios (HPZR) are also specified in secondary horizontal axis in Fig. 2(a).

As illustrated in Fig. 2, the degradation efficiency increases with decreasing pH and with increasing ZVI concentration. Decreasing of pH leads to more violent corrosion rate of ZVI surface and increased concentration of ferrous in solution.

In all the pHs except for pH = 3, after a specific ZVI concentration, the degradation efficiency reached at the maximum level (~81%). This is probably due to the saturation of ferrous ion in the solution. This specific ZVI concentration in pH = 1.5 and 2 is 5.35 g/L and in pH = 2.5 is 10.7 g/L. According to these results, the optimal pH and ZVI concentration are 2 and 5.35 g/L, respectively, because in this condition the maximum degradation efficiency is reached, with the minimum required amounts of ZVI and the optimal pH are. Here, the ZVI concentration of 5.35 g/L is corresponded to HPZR of 2. As mentioned above, corrosion phenomenon eventually leads to a different optimal pH in the Fenton (optimal pH = 3 [14,32]) and the Fenton-like process using ZVI (optimal pH = 2) for cresol degradation.

3.2.2. Effect of hydrogen peroxide concentration

In order to determine the effect of hydrogen peroxide concentration, two series of batch experiments were conducted. In these experiments cresol concentration, pH and temperature were 18.5 mM, 2 and 30°C, respectively. In one series of experiments, the catalyst concentration was 5.35 g/L and in the other series the HPZR was 2. In two series of experiments, the concentration of hydrogen peroxide was varied from 157.5 to 630 mM (molar ratios of 8.5–34). The results are presented in Fig. 3.

According to Fig. 3, the degradation efficiency first increases with increasing hydrogen peroxide concentration

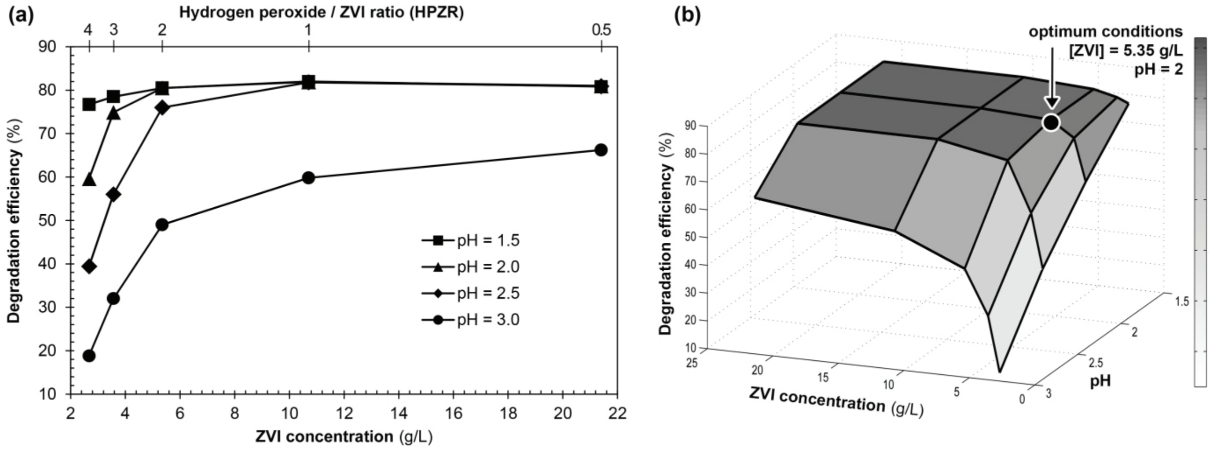

Fig. 2. The effects of catalyst concentration and initial pH on degradation of cresol (a) 2D and (b) 3D presentation (Conditions: [cresol] = 18.5 mM, [H_2O_2] = 315 mM, T = 30°C and t = 120 min).

and then declines after 472 mM (molar ratio of 25.5) for both series. Maximum degradation (~87.5%) occurred at 472 mM of hydrogen peroxide.

Hydrogen peroxide is the source of hydroxyl radicals that are responsible for the degradation of cresol. However, when in excess, hydrogen peroxide reveals a radical scavenging capacity. Excess of hydrogen peroxide reacts with hydroxyl radical (Eq. (7)) to form hydroperoxyl radical ($HO_2^\bullet$) which has weaker activity than hydroxyl radical toward organic degradation (Eq. (7)) [33].

$$H_2O_2 + HO^\bullet \rightarrow H_2O + HO_2^\bullet \qquad (7)$$

The optimal hydrogen peroxide concentration in the Fenton-like process using ZVI is affected by the corrosion phenomenon and is different from the classical Fenton process (optimal molar ratio for hydrogen peroxide in the Fenton process is 17 [14] and here is 25.5).

Li et al. [34] investigated the effects of hydrogen peroxide on iron corrosion and passivation in water and water-organic mixtures using cyclic voltammetry. They found at a neutral pH range, hydrogen peroxide controlled iron passivation and inhibited corrosion but at lower pH, cathodic current increased indicating increased corrosion at the cathodic sites. Thus, hydrogen peroxide can intensify the corrosion rate of ZVI surface and can increase the concentration of a ferrous catalyst in the Fenton-like process.

3.2.3. Effect of temperature

At optimal hydrogen peroxide concentration, ZVI concentration and pH, the effect of temperature on the degradation efficiency of cresol was studied. The results are illustrated in Fig. 4.

The degradation efficiency first increases sharply with increasing temperature and then decreases after 30°C. The optimal temperature obtained for the cresol degradation in the Fenton-like process is in accordance with the optimal temperature in the classical Fenton processes [14,32,35]. The decline in degradation efficiency below 30°C is due to slow reaction rate between hydroxyl radical and cresol. Above 30°C the decline is due to the formation of recalcitrant organic

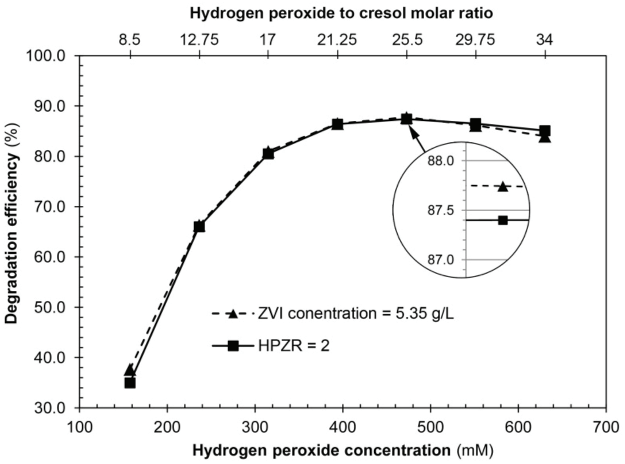

Fig. 3. The effects of hydrogen peroxide concentration on degradation of cresol (Conditions: [cresol] = 18.5 mM, pH = 2, T = 30°C and t = 120 min).

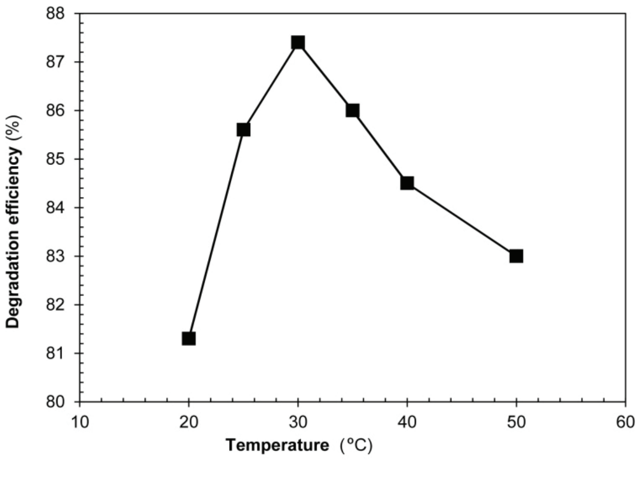

Fig. 4. The effects of change in temperature on degradation of cresol (Conditions: [cresol] = 18.5 mM, [H_2O_2] = 472 mM, [ZVI] = 5.35 g/L, pH = 2 and t = 120 min).

intermediates or thermal degradation of hydrogen peroxide prior to formation of hydroxyl radicals.

3.3. Evolution of the parameters during Fenton-like process

At optimum conditions a batch experiment was conducted to determine COD, cresol concentration and the products evolution during Fenton-like process. Immediately after sampling, all the samples were quenched by adding sodium hydroxide solution. The evolution of the cresol concentration during the process is presented in Fig. 5(a), and Fig. 5(b) shows the evolution of the products during the process. The results of the COD removal during the process are shown in Fig. 5(c).

Another batch experiment was also conducted to determine the evolution of dissolved iron concentration and pH of solution during the process. These samples were immediately filtered through Whatman no. 42 filter paper to separate from non-soluble and elemental iron without any quenching – and then were analyzed using AAS method for dissolved iron concentrations. The concentrations of dissolved iron and pH of solution are illustrated in Fig. 5(d).

As presented in Fig. 5 during the initial stages of the process, cresol disappeared rapidly but the COD removal of the solution and the generation of the products occurred in a continuous and slower trend and after 120 min they reached the maximum level. This is most likely due to the rapid generation of some intermediates from cresol that finally degraded to acetic and oxalic acid. We could not detect these intermediates by HPLC or IC methods.

The trend of the COD removal in the Fenton-like process using μ-ZVI (this study) was substantially different from the trend in the classical Fenton process [15, Fig. 1]. The rate of the COD removal in early stages of the Fenton-like process was very higher than the rate in the classical process presumably due to adsorption of cresol on the heterogeneous catalyst surface in the Fenton-like process prior to destruction.

During the first 30 min of the reaction time, dissolved iron concentration and pH of the solution increased simultaneously specifically between 20 and 30 min of the reaction time. After 30 min of the reaction time, the pH of the solution reached a steady value (~5.2) but a slow decay in the dissolved iron concentration was detected afterwards – indicating that dissolved iron was slowly transformed into a non-soluble form. This phenomenon is in accordance with a similar study [28]. We propose that the dissolved iron was precipitated by the organic products of the degradation of cresol (e.g., oxalate) as in this period of time (30–120 min) the pH of the solution did not change and the precipitation in the form of iron hydroxides was not feasible.

3.4. ZVI reuse

For a more detailed investigation of this process, at the optimum conditions (5.35 g/L fresh ZVI Fig. 5(a)) a batch experiment was conducted without any sampling. After the completion of the process (120 min) all the reactor contents were quantitatively transferred to a filter and filtered through Whatman no. 42 filter paper. The residual solids on the filter were washed with distilled water. Eventually 4.84 g/L dried ZVI (Fig. 6(b)) was recovered (purity = 90.1%).

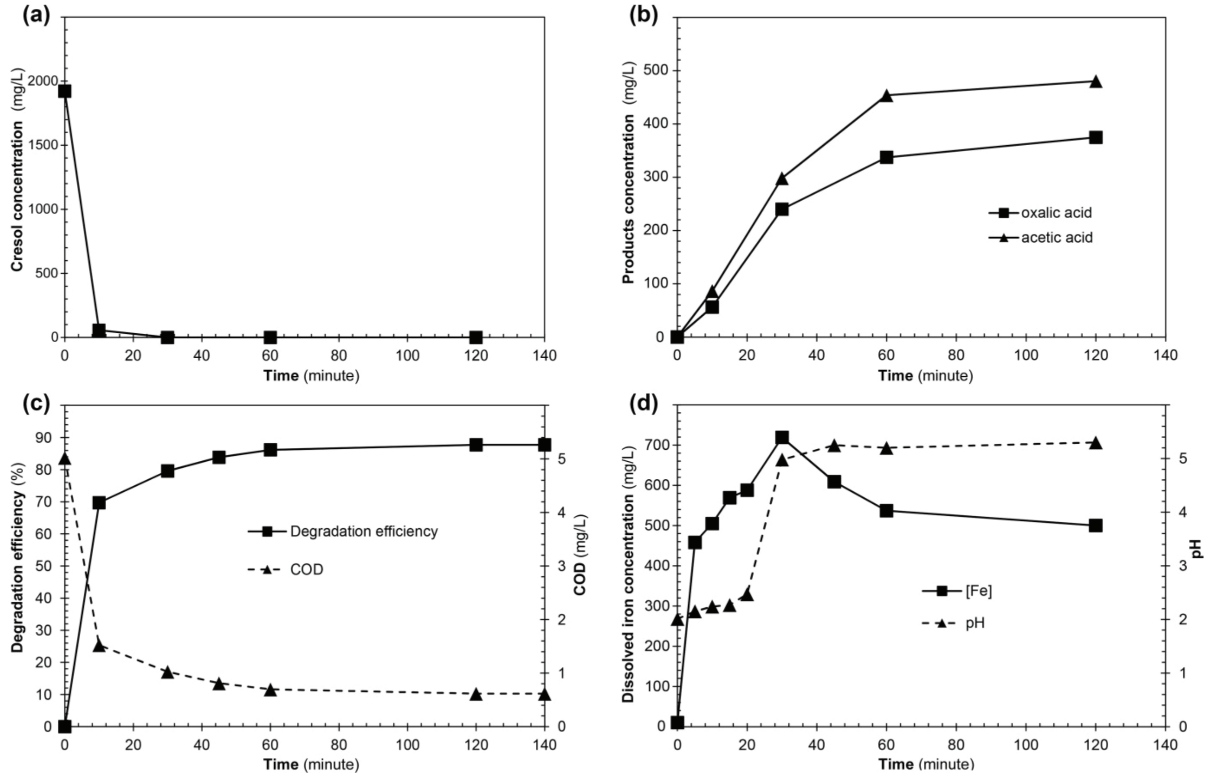

Fig. 5. Evolution of (a) [cresol], (b) [products], (c) COD and (d) dissolved iron and pH during Fenton-like process (Conditions: [cresol] = 18.5 mM, [H_2O_2] = 472 mM, [ZVI] = 5.35 g/L, pH = 2 and T = 30°C).

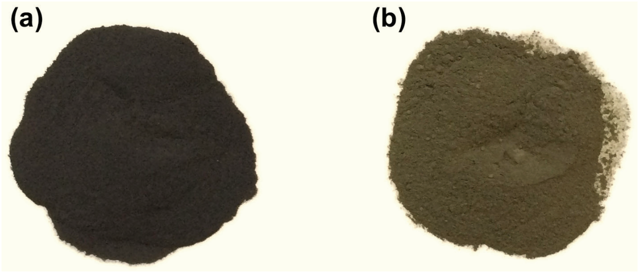

Fig. 6. ZVI (a) before and (b) after Fenton-like process.

According to the weight and purities of the ZVI samples after the process 85.8% of initial ZVI was recovered as elemental iron. Thus, to check ZVI behavior along reuses, 10 repetitive cycles were performed. After any batch experiments, the remaining ZVI was washed with distilled water, dried in an oven and weighed. Subsequently, an appropriate amount of fresh ZVI was added to it to maintain total ZVI concentration at 5.35 g/L for the next batch. The average fresh ZVI in these batches was 21% of the total ZVI. The results of these batches are presented in Fig. 7.

3.5. Bioassay tests

Previously, we reported the applicability of the germination of *Lepidium sativum* L. for the evaluation of cresol phytotoxicity and its Fenton's degradation products [15]. In this study, the phytotoxicities of the initial wastewater ([cresol] = 18.5 mM), a Fenton treated wastewater and a Fenton-like treated wastewater (both of them at optimum conditions) and their diluted solutions (1:10) were assessed by germination of *Lepidium sativum* L. In all the germination tests, the percentage of relative seed germination (RSG), relative root growth (RRG) and GI were calculated as follows [15,36]:

$$\text{RSG} = \frac{\text{Number of seeds germinated in sample}}{\text{Number of seeds germinated in blank}} \times 100 \tag{8}$$

$$\text{RRG} = \frac{\text{Mean root length in sample}}{\text{Mean root length in blank}} \times 100 \tag{9}$$

$$\text{GI} = \frac{\text{RSG} \times \text{RRG}}{100} \tag{10}$$

Fig. 8 presents the results of the germination of seeds in the samples.

In this study, the GI improvement after the classical Fenton process for 18.5 mM solution of cresol is in accordance with the results of our previous study [15]. In that study, the GI of a 1.85 mM solution of cresol was improved from 33.1% to 59.7% during a classical Fenton process. On the other hand, the GI of the Fenton-like treated wastewater is better in comparison with the classical Fenton process results. The phytotoxicity results in the two treated samples were proportional to their COD removal.

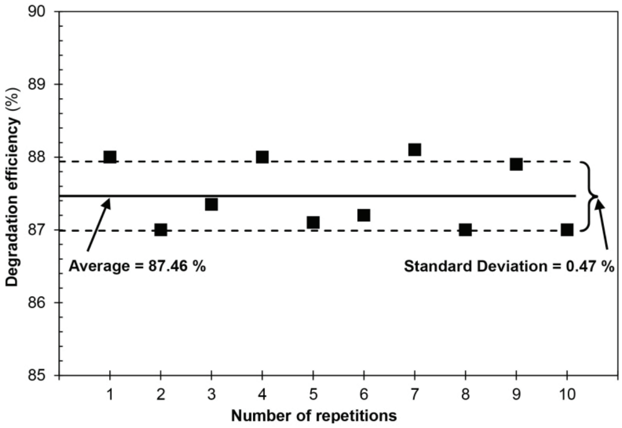

Fig. 7. Degradation efficiencies for repetitive ZVI (Conditions: [cresol] = 18.5 mM, [H_2O_2] = 472 mM, [ZVI] = 5.35 g/L (from previous batch + fresh), pH = 2, *T* = 30°C and *t* = 120 min).

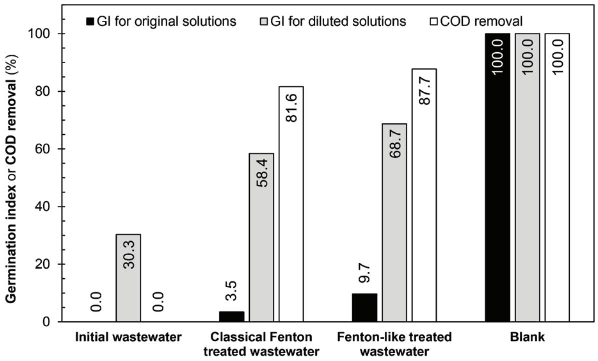

Fig. 8. Germination index and COD removal for the initial wastewaters, the classical Fenton and the Fenton-like treated wastewaters.

4. Conclusions

The classical Fenton and the Fenton-like processes portray different optimal conditions for the degradation of cresol. In the Fenton-like process, optimum initial pH is 2 – which is lower in comparison with the optimum pH of the classical Fenton process (3). Moreover, optimum H_2O_2/cresol molar ratio in the Fenton-like process (25.5) is significantly higher in comparison with the classical Fenton process (17). Accordingly, the Fenton-like process using ZVI prefers more acidic and oxidative conditions.

The efficiency of the Fenton-like process using μ-ZVI for the degradation of cresol (87.7%) was significantly higher in comparison with the classical Fenton process (81.6%). The μ-ZVI catalyst shows appropriate reusability in 10 repetitive cycles.

According to this study, the Fenton-like process using μ-ZVI can be used as a treatment method for the degradation of cresol in wastewaters. The degradation efficiency and the detoxification of this method are higher in comparison with the classical Fenton process.

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