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Treatment of landfill leachate by sonolysis followed by Fenton process

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ABSTRACT

The landfill leachate was treated by combined sonolysis and Fenton process. The effect of factors such as operation mode, pH, chemical reagents, initial concentration, and temperature on TOC removal was investigated. The results showed that the pre-treatment of leachate by sonolysis at the presence of Fe^{2+} was beneficial to Fenton process, and the highest TOC removal efficiency was achieved at four different operation modes by this treatment. The optimal reaction condition of Fenton was experimentally determined to be $[H_2O_2]_0 = 100$ mM, $[H_2O_2]/[Fe^{2+}] = 5$, pH = 3, and $[TOC]_0 = 600$ mg/L by taking both economy and efficiency into consideration in this study. The removal kinetics of leachate by Fenton process followed the first-order reaction kinetics and the kinetic equation of TOC degradation was established.

Keywords: Landfill leachate; Sonolysis; Fenton process; Operation mode; Kinetic

1. Introduction

Landfill leachate, which has been confirmed to be a potential danger by various test organisms such as white-toothed shrew [1], wood mouse [2], and Bioassays [3], is generated by physiochemical and biological decomposition of solid wastes and percolation of rainwater through compacted wastes [4]. The characteristics depend on the type of deposited waste and its landfill site, hydrological, humidity, and climate conditions, and landfill age factors [5]. Biological processes are demonstrated to be effective for the treatment of young leachate (landfill age less than 1–2 years), but not for moderate (landfill age range from 2 to 5 years) or mature leachate (landfill age more than 5–10 years), due to changes of five-day biochemical oxygen demand (BOD_5)/chemical oxygen demand (COD) ratios and types of molecular weight organics [6]. Therefore, advanced oxidation processes (AOPs) have been studied or used for the treatment of moderate or mature leachate [6–8].

Fenton process as one of AOPs has been extensively studied for leachate treatment [9] and considered to be an alternative to biological processes [10]. It is based on hydrogen peroxide catalyzed by ferrous ion to produce hydroxyl radicals (Eq. (1)), and followed chain reactions (Eqs. (2–4)) resulting in oxidation of organics [11].

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

$$Fe^{2+} + {}^{\cdot}OH \rightarrow Fe^{3+} + OH^- \quad (2)$$

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$$RH + {}^{\cdot}OH \rightarrow H_2O + R^{\cdot} \quad (3)$$

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

However, its high cost induced by the consumption of hydrogen peroxide hinders it from being widely used in practical applications. Current literatures focused on Fenton process coupled with sonolysis, which was named as sono-Fenton and found to be effective for reducing the dosage of chemicals and improving the removal efficiency of organics [12–16]. However, little research was conducted sequentially on the integration of sonolysis and Fenton process in the homogeneous solution. The object of this study focused on the sequentially integration of sonolysis and Fenton process for oxidation of landfill leachate. The effect of influencing parameters such as operation mode, initial pH, temperature, chemical reagents, and initial concentration of leachate were comprehensively investigated.

2. Materials and methods

2.1. Materials

All regents used in this work were of analytical grade obtained from Aladdin® (China). The raw landfill leachate was collected from an adjusting tank of the Huainan Landfill (China) which had been run since 2009. The pH of the samples taken were adjusted to less than 3.0 and preserved in refrigerator at 4°C according to the Standard Methods. The characteristics of the leachate used and the average values are given in Table 1.

2.2. Procedure

All experiments were conducted in a cylindrical glass tube (500 mL). Leachate sample was diluted to the desired TOC strength with distilled water. Temperature and pH were adjusted to the desired values. A selected amount of ferrous sulfate was dissolved. The tube was fixed at the central of an ultrasonator (JK-5200DB, Jinnike®, China, operated at 80 kHz with a power of 200 W) and treated 30 min by sonication. Samples were taken and measured by TOC analyzer (Vcph-5000A, Shimadzu®, Japan). Then, a selected amount of hydrogen peroxide was added in a single step equipped with the magnetic stirrer maintaining same agitation speed to avoid dead zones. Reaction was timed as soon as the hydrogen peroxide was added. The surrounding temperature of tube was monitored during the reaction time and kept constant at desired value ±2°C. Samples were taken at pre-selected time intervals with syringe to a glass cylinder, neutralized to about 7.0 with power sodium hydroxide, and then adjusted to 8.0 ± 0.05 with 10 M sodium hydroxide and 1 M sulfuric acid. Following that they were dispensed and heated in water bath at 50°C (DK-S26, Jinhong®, China) for 30 min to remove any residual H_2O_2 in the solution. Finally, samples were settled for 30 min, and filtered with a 0.45 μm filter. TOC was measured by TOC analyzer and COD was measured by the colorimetric method at 610 nm using a spectrophotometer (Hach® DR/2000, China) according to the Standard Methods.

3. Results and discussion

3.1. Effect of operation mode

Effect of combined technology on TOC removal efficiency at a fixed H_2O_2 dosage of 200 mM at initial pH 3.0 with initial concentration of 600 mg L^{-1} was investigated by four modes at $[H_2O_2]/[Fe^{2+}]$ ratios of 3 and 5: (1) Fenton process and sonolysis run simultaneously; (2) Fenton worked after 10 min of sonolysis; (3) Fenton followed by sonolysis; and (4) operating only Fenton. In order to decrease the influence of reaction time for coupling system, the reaction time of sonolysis and Fenton was set to 30 min as shown in Fig. 1. It was found that operation mode significantly affected the TOC removal efficiency, and the order of the removal efficiency of different modes was: mode (3) > mode (1) > mode (2) > mode (4) for both $[H_2O_2]/[Fe^{2+}]$ molar ratios. Along with the treatment of mode (1), sonication, as an assistant technology, provides promotion of mixing, mass transfer, and a better homogenization that could be beneficial for chemical reaction [13,14]. At the same time, sonication induced a great amount of cavitation bubbles, which grow,

Table 1
Properties of the leachate used in experiments

Parameters	COD (mg L^{-1})	TOC (mg L^{-1})	DOC (mg L^{-1})	Chloride (mg L^{-1})	pH	Conductivity (ms cm^{-1})	Alkalinity (mg $CaCO_3$ L^{-1})
	3,930 ± 62	1,142 ± 53	1,037 ± 28	3,865 ± 31	7.89 ± 0.21	17.7 ± 0.2	3,751 ± 30

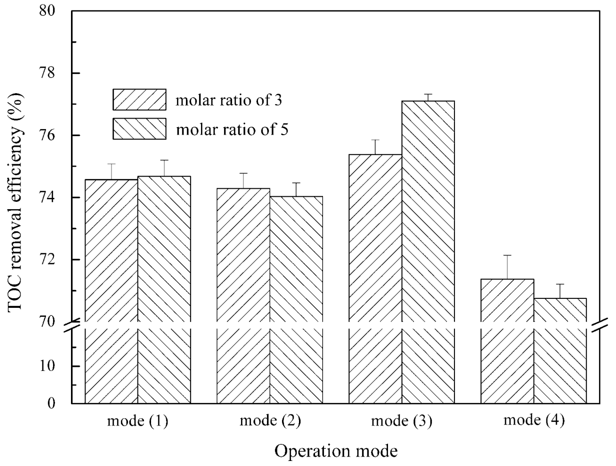

Fig. 1. TOC removal by various operation modes.

pulsate, and collapse, resulting in formation of a hot nucleus with high temperature (about 5,000 K) and pressure (up to 1,000 atm), and further leading to sonochemical effect taking place by pyrolytic decomposition or reduction and oxidation due to generation of hydroxyl radicals (Eq. (5)) [17]. Furthermore, sonication enhances the formation of hydroxyl radicals within the frame of the Fenton cycle [12,13] (Eqs. (6–8)) which was an aid to mineralization of leachate.

$$H_2O \xrightarrow{sonication} H^{\cdot} + {}^{\cdot}OH \tag{5}$$

$$Fe^{3+} + H_2O \rightarrow [Fe(OH)]^{2+} + H^{+} \tag{6}$$

$$[Fe(OH)]^{2+} \xrightarrow{sonication} Fe^{2+} + {}^{\cdot}OH \tag{7}$$

$$Fe(OOH)^{2+} \xrightarrow{sonication} Fe^{2+} + HO_2^{\cdot} \tag{8}$$

On the other hand, the organics were oxidized by hydroxyl radicals into CO_2, which induced free radicals by sonolysis [17]. These factors were related to reaction time, thus mode (1) achieved better removal efficiency than (2) and (4).

On comparing mode (3) with (1), leachate was pre-treated by sonolysis in the presence of Fe^{2+} without adding hydrogen peroxide. The TOC removal efficiency of mode (3) was higher than (1). This was caused by the recombination of hydroxyl radicals and perhydroxyl at the interface of the bubbles yielded hydrogen peroxide (Eqs. (9–10)) with concentration increasing along reaction time [18], which can oxidize organics in leachate catalyzed by Fe^{2+}. Moreover, the by-product, oxygen, was generated to produce more hydroxyl radicals (Eqs. (11–12)) resulting in improved TOC removal. On the other hand, the properties improved by sonolysis were beneficial to the following Fenton process to mineralize leachate. Mode (3) was selected for further experiments.

$${}^{\cdot}OH + {}^{\cdot}OH \rightarrow H_2O_2 \tag{9}$$

$${}^{\cdot}OOH + {}^{\cdot}OOH \rightarrow H_2O_2 + O_2 \tag{10}$$

$$O_2 \rightarrow 2O \tag{11}$$

$$O + H_2O \xrightarrow{sonication} 2{}^{\cdot}OH \tag{12}$$

3.2. *Effect of reaction time*

Fig. 2 shows TOC and COD removal as a function of reaction time. The trends of COD and TOC removal were the same, and COD removal was higher than TOC. COD and TOC removal increased rapidly to 79.1% and 72.4% during the first 5 min, 85.8% and 78.8% at 30 min, and afterward gradually reaching 86.9 and 79.8% in 120 min, respectively. According to the classification of degradation by Lyman et al. [19], COD will change throughout three degradation stages, while TOC decreases to ultimate degradation and may keep constant during the primary and acceptable degradation, which resulted in higher removal of COD than TOC. The result demonstrated that most COD and TOC removal occurred in the first 30 min, and the change of residual COD and TOC became insignificant after 30 min.

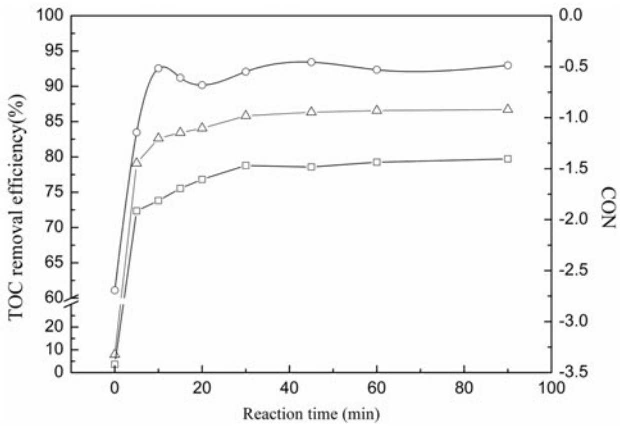

Fig. 2. Effect of reaction time on COD, TOC, and CON of leachate by Fenton process.

The average carbon oxidation number (ACON), which depicts carbon mineralization of organics, was calculated by the following equation:

$$\text{CON} = 4\left(1 - \frac{\text{COD}}{\text{TOC}}\right) \tag{13}$$

where TOC is in mol of C/L and COD is in mol of O_2/L. As shown in Fig. 2, the CON was rapidly reaching −0.55 from −2.69 during the first 30 min, afterward ranged from −0.45 to −0.53. It demonstrated that the organics were rapidly mineralized to carbon dioxide during the first 30 min. The reaction time was determined to be 30 min for further experiments.

3.3. Effect of initial pH

The pH, which is one of the major factors in treatment of wastewater by sonolysis and Fenton process, affects the activity of the oxidant and substrate, the speciation of iron, and the stability of hydrogen peroxide [12,19]. The influence of pH was investigated and the results are shown in Fig. 3. It showed the TOC removal efficiency by sonolysis at the presence of Fe^{2+} was minimal; ranged from 2.3 to 4.0%. The maximum TOC removal efficiency of 78.8% was obtained at pH 3 by coupling system, and no significant difference in TOC removal at pH 2.0 and 4.0 was observed. Those were due to the higher hydroxyl radical product yields in the range of pH 2.0–4.0; where either hydrogen peroxide is regenerated or reaction rates are increased. Furthermore, below optimal pH has been found to be inhibited oxidation, above optimal pH also hinders Fenton oxidation [20]. Thus, pH 3 was selected for further experiments.

3.4. Effect of temperature

The leachate was treated by sequent coupling system at different temperatures of 12, 25, and 35˚C, and the results are shown in Fig. 4. The removal at 35˚C was 79.1% higher than at 25˚C and 12˚C, which indicated that temperature had a slight effect on the removal efficiency of leachate. The removal was accelerated with the increase of temperature because high temperature are likely to facilitate cavitation bubble formation with an increase in the equilibrium vapor pressure [16]. Furthermore, higher temperature can improve the rate of generation of oxidation species such as ·OH or high-valence iron species throughout by accelerating the reaction between hydrogen peroxide and ferrous ions [21,22]. But the improvement of TOC was slight. The temperature of further experiments was uncontrolled to decrease operating costs. According to the Arrhenius equation (Eq. (14)):

$$K = A\exp\left(-\frac{E_a}{RT}\right) \tag{14}$$

where K is the reaction rate constant, A is the frequency factor that depends on the probabilities and orientation of molecular collision, E_a is the activation energy (kcal/mol), R is the universal gas constant (kcal/mol K), and T is the absolute temperature (K). The E_a was equal to 7.13 by calculating the TOC degradation rate under different temperature and A was equal to 0.076.

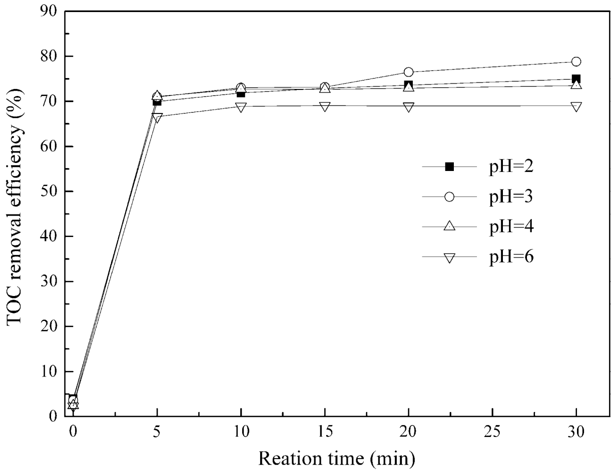

Fig. 3. Effect of initial pH on TOC removal with respect to time by Fenton process.

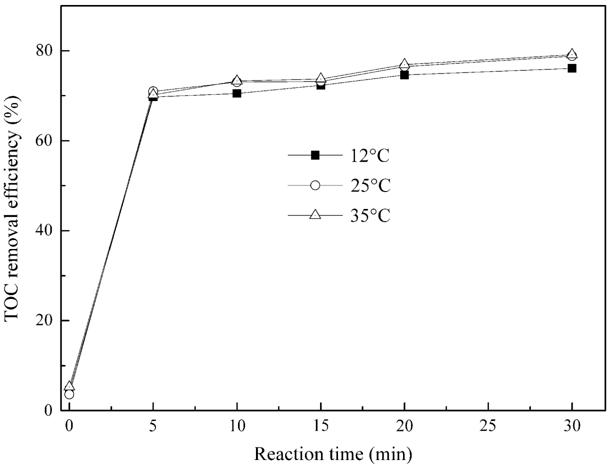

Fig. 4. Effect of different temperature on TOC removal efficiencies with respect to time by Fenton process.

3.5. Effect of chemical reagents

The effects of both absolute chemical reagents (hydrogen peroxide and ferrous ions) and molar ratios ($[H_2O_2]/[Fe^{2+}]$), which determined the operation costs as well as efficacy, were fully investigated. Firstly, in order to investigate the effect of molar ratios on the degradation, experiments were conducted at four different values ranging between 3 and 20 along with 200 mM H_2O_2. As shown in Fig. 5(a), at a low molar ratio of 3, the removal efficiency was 77.8% at 30 min reaction time, whereas for 5, 10, and 20, the removal efficiencies were 78.8, 69.2, and 60.7%, respectively. The TOC removal by coagulation was 12.4, 10.8, 10.4, and 9.6% at 30 min for molar ratios of 3–20. These results were induced by the changed mechanism [19] for high molar ratio and hydroxyl radicals scavenged by excessive Fe^{2+} for low molar ratio [23], leading to a lower removal at fixed dosage of H_2O_2. Molar ratio of 5 was selected in the following experiments to control the cost of operation.

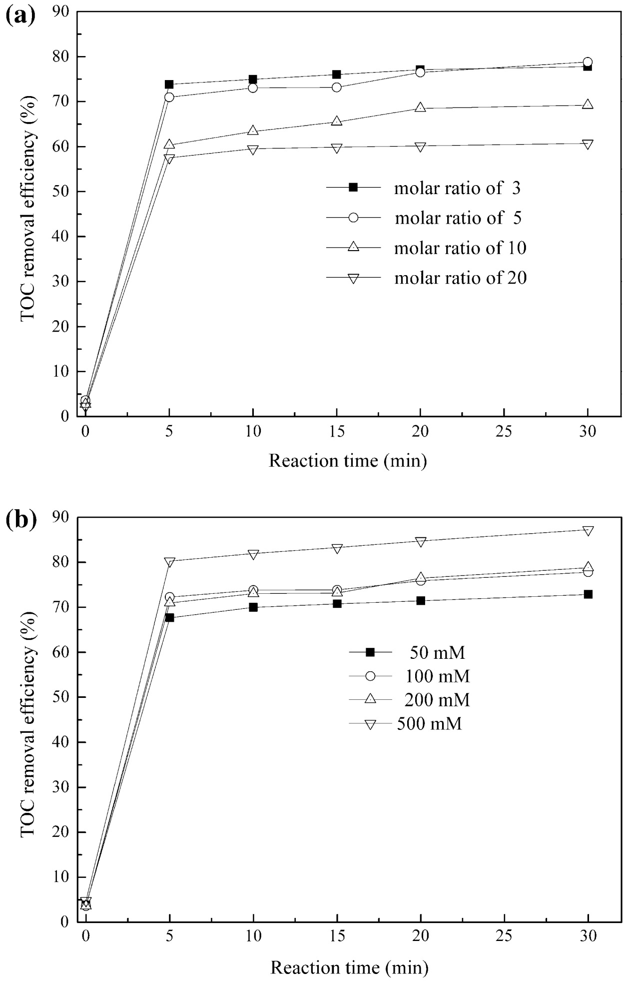

Fig. 5. (a) Effect of different $[H_2O_2]/[Fe^{2+}]$ molar ratios on TOC removal efficiencies with respect to time by Fenton process. (b) Effect of different dosage of H_2O_2 on TOC removal efficiencies with respect to time by Fenton process.

As shown in Fig. 5(b), the removal efficiencies increase from 74.8 to 87.2% as a consequence of increasing H_2O_2 dosage from 50 to 500 mM at $[H_2O_2]/[Fe^{2+}]$ of 5 at 30 min of the reaction. And the TOC removal by coagulation was 9.5, 10.5, 10.8, and 13.6% at the same condition. The improvement was induced by increasing $^{\cdot}$OH amount generated from the decompose of H_2O_2 [24,25]. However, once the concentration of $[H_2O_2]$ exceeds 100 mM, TOC removal efficiency did not improve significantly and got less 9% improvement for 500. This phenomenon explained that hydroxyl radical were scavenged by hydrogen peroxide to produce perhydroxyl radical leading to the degradation of utilization ratio and oxidation capacities and the excessive hydrogen peroxide was decomposed by itself [25]. So, the dosage of H_2O_2 was selected for 100 mM for further experiments.

3.6. Effect of initial concentration

The effects of different concentrations of leachate were conducted and the results are shown in Fig. 6. As the concentration of leachate increases from 120 to 600 mg/L employing the $[H_2O_2]/[TOC]$ molar ratio of 1, $[H_2O_2]/[Fe^{2+}]$ of 5 and pH 3 were previously determined. The removal efficiency of leachate increased from 67.9 to 77.8% within 30 min of reaction time. Increase in the concentration of leachate implied that leachate molecules per molar ratio will be easily

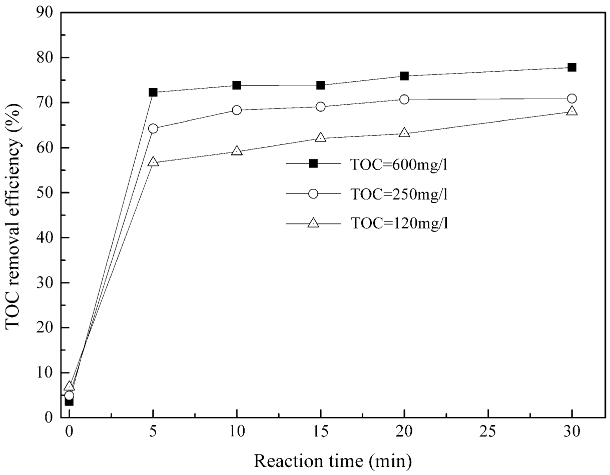

Fig. 6. Effect of different initial concentration of leachte on TOC removal efficiencies with respect to time by Fenton process.

oxidized by the same amount of ·OH, which leads to a decrease in ·OH scavenged by hydrogen peroxide or itself and an increase in the mineralization of organic matter.

4. Kinetic model

The process of Fenton reaction was commonly used to depict with kinetic models [21,22,26]. In this study, first-order kinetic reactions were used to simulate the removal of leachate by Fenton process. Regression analyses based on first-order kinetic reactions are shown in Table 2.

The results showed the values of the regression coefficients for first-order reaction ranged from 0.44 to 0.99 with an average of 0.88. Furthermore, the regression coefficients for $\text{pH} = 3$ were beyond 0.90 except for $[H_2O_2]/[Fe^{2+}]$ molar ratio of 20; for that Fenton's reaction mechanisms changed [19]. These results indicated the TOC removal of the leachate via Fenton process described by the first-order kinetic reaction. According to the above research, the kinetic constants were affected by the dosage of H_2O_2, $[Fe^{2+}]$ and temperature. The kinetic model was proposed using the following equation:

$$-\frac{\mathrm{d}[\mathrm{TOC}/\mathrm{TOC}_0]}{\mathrm{d}t} = 0.076\exp\left(-\frac{7.13}{RT}\right) \times [H_2O_2]^m[Fe^{2+}]^n[\mathrm{TOC}/\mathrm{TOC}_0] \qquad (16)$$

where TOC_0, $[H_2O_2]$ and $[Fe^{2+}]$ are the initial concentration of leachate or Fenton reagents in mg/L, and TOC was the concentration of leachate at pre-selected reaction time in mg/L. As described by Xu [26], the $m = 0.17$ and $n = -0.63$ were obtained using the curvilinear regressions of $\mathrm{Ln}(-\Delta\frac{\mathrm{TOC}}{\mathrm{TOC}_0}/\Delta t)$ vs. $\mathrm{Ln}([H_2O_2])$ and $\mathrm{Ln}(-\Delta\frac{\mathrm{TOC}}{\mathrm{TOC}_0}/\Delta t)$ vs. $\mathrm{Ln}([Fe^{2+}])$. As a result, the kinetic model for landfill leachate treated by Fenton process was expressed as follows:

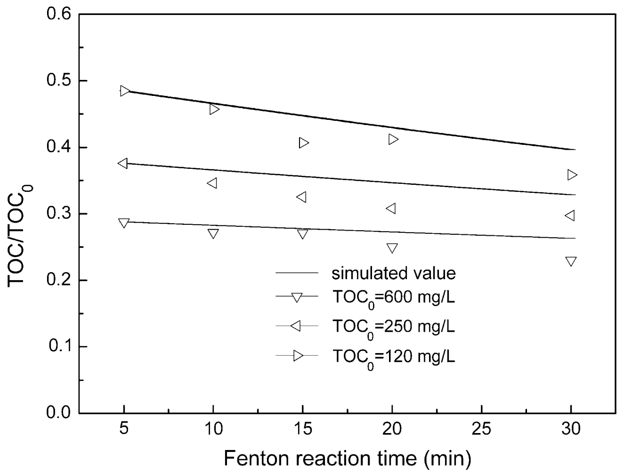

Fig. 7. Predicted value vs. experimental value.

$$-\frac{\mathrm{d}[\mathrm{TOC}/\mathrm{TOC}_0]}{\mathrm{d}t} = 0.076\exp\left(-\frac{7.13}{RT}\right)[H_2O_2]^{0.17} \times [Fe^{2+}]^{-0.63}[\mathrm{TOC}/\mathrm{TOC}_0] \qquad (16)$$

As shown in Fig. 7, the results showed excellent agreement between simulation by Eq. (16) on different initial concentrations of leachate and the experiment.

Table 2
The first- and second-order kinetic rate constants for the removal of landfill leachate at different reaction conditions

No.	[TOC] (mg/L)	$[H_2O_2]_0$ (mM)	$[Fe^{2+}]_0$ (mM)	pH	Temp (℃)	First-order	
						k_1 (min^{-1})	R^2
1	600	200	40	2.0	25	0.0070	0.96
2	600	200	40	3.0	25	0.013	0.96
3	600	200	40	4.0	25	0.0029	0.74
4	600	200	40	6.0	25	0.0023	0.44
5	600	200	67	3.0	25	0.0067	0.95
6	600	200	20	3.0	25	0.010	0.90
7	600	200	10	3.0	25	0.0027	0.77
8	600	50	10	3.0	25	0.012	0.90
9	600	100	20	3.0	25	0.0089	0.96
10	600	500	100	3.0	25	0.017	0.99
11	600	200	40	3.0	12	0.010	0.96
12	600	200	40	3.0	35	0.014	0.97
13	250	42	8	3.0	25	0.093	0.91
14	120	17	3	3.0	25	0.012	0.94

5. Conclusion

Degradation of landfill leachate was investigated by sonolysis followed by Fenton process. The removal efficiency was affected by the type of operation mode, pH, the values of Fenton reagent, initial concentration of leachate, and temperature. The optimal reaction condition was experimentally determined to be $[H_2O_2]_0 = 100\,\text{mM}$, $[H_2O_2]/[Fe^{2+}] = 5$, $\text{pH} = 3$, and $[TOC]_0 = 600\,\text{mg/L}$ with a balance of cost and efficiency in this study. The removal kinetics of leachate by Fenton process followed the first-order reaction kinetic and the kinetic equation of TOC degradation was established and expressed as follows:

$$-\frac{\mathrm{d}[\mathrm{TOC}/\mathrm{TOC}_0]}{\mathrm{d}t} = 0.076\exp\left(-\frac{7.13}{RT}\right) \times [\mathrm{H_2O_2}]^{0.17}[\mathrm{Fe}^{2+}]^{-0.63}[\mathrm{TOC}/\mathrm{TOC}_0].$$

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