

# Removal of COD and color from livestock wastewater by the Fenton method

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## Abstract

The Fenton method was applied to the removal of chemical oxygen demand using chromate (COD<sub>Cr</sub>) and color from high-strength livestock wastewater in which the initial COD<sub>Cr</sub> was 5000–5700 mg/L. The optimum ratio of H<sub>2</sub>O<sub>2</sub> (mg/L) to the initial COD<sub>Cr</sub> was 1.05 and the optimum molar ratio of H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> was 2. The optimum initial pH and the optimum reaction time were 3.5–4 and 30 min, respectively. Under optimal conditions, the removal ratios of COD<sub>Cr</sub> and color of the supernatant after static precipitation of the produced sludge were 88 and 95.4%, respectively. Addition of Fenton's reagents in several aliquots did not affect the efficiencies of COD<sub>Cr</sub> and color removal. © 2007 Elsevier B.V. All rights reserved.

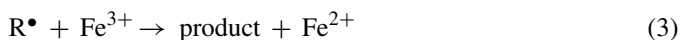
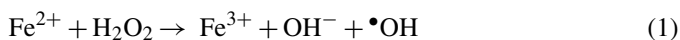
**Keywords:** Fenton method; Livestock wastewater; COD<sub>Cr</sub> removal; Color removal

## 1. Introduction

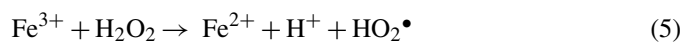
Livestock wastewater contains high-strength of COD, BOD<sub>5</sub>, color, nitrogen, phosphorus and suspended solids. Large volumes of livestock wastewater are produced, and the wastewater is a primary pollution source of surface and ground waters. Much attention is paid to these problems in many countries as treatment of livestock wastewater poses a difficult problem [1–4]. Issues include the intense odor, the possibility of pathogen dissemination and the content of structured polymers toxic to microorganisms [1,3]. Several treatment processes are therefore used to treat livestock wastewater. Among these are anaerobic digestion, aerobic digestion (e.g., the autothermal thermophilic aerobic digestion process (ATAD) [1]), anaerobic–aerobic digestion (e.g., anaerobic–aerobic sequencing batch reactor (SBR) [5]) and aerobic digestion–chemical treatment [6]. Since 2004, the law concerning the appropriate treatment and promotion of utilization of livestock manure has been put into effect in Japan, the advanced biological treatment methods of livestock wastes have been intensively pursued, including efficient composting, wastewater treatment using protozoa or sulfur-oxidizing denitrifiers, and methane fermentation incorporated

with membrane [7]. However, application of chemical treatments to livestock wastes is relatively small in number.

Recently, advanced oxidation processes (AOP) have been proposed to treat relatively low-strength industrial wastewaters containing non-biodegradable substances toxic to microorganisms. Among them, the Fenton method is cost-effective, easy to apply and effective with relatively low-strength wastewater containing organic compounds and has been applied to the decolorization of textile wastewater [8–12]. The Fenton method consists of four stages. First, pH is adjusted to low acidity. Then, main oxidation reaction takes place at pH value of 3–5. The wastewater is then neutralized at pH of 7–8 and finally precipitation occurs [13]. The oxidation mechanism in the Fenton process involves the reactive hydroxyl radical generated under acidic conditions by the catalytic decomposition of hydrogen peroxide, which reacts unselectively within 1 ms with organic substances (RH), which are based on carbon chains or rings and also contain hydrogen, oxygen, nitrogen, or other elements. The reaction mechanism is as follows [11,14–16]:



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Only a few applications of Fenton oxidation to high-strength wastewater such as effluent from a baker's yeast industry effluent or olive oil mill wastewater have been reported [17,18]. Therefore, purpose of this study was to apply the Fenton method to high-strength livestock wastewater, which is refractory to biological treatment unless COD has been reduced prior to the treatment. To find optimal conditions for removal of COD and color of livestock wastewater, we investigated the  $\text{H}_2\text{O}_2$  dosage,  $\text{Fe}^{2+}$  dosage,  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio, initial pH, reaction time and dosage method.

## 2. Materials and methods

### 2.1. Livestock wastewater

Livestock wastewater used was supplied by the Kanagawa Prefectural Livestock Industry Research Institute, Kanagawa, Japan. Raw livestock wastewater containing urine, washing water and feces was sieved using a No. 500 sieve (i.e., 500  $\mu\text{m}$  openings) to remove solid materials. This wastewater which is referred to as livestock wastewater was used in this study. Table 1 shows the characteristics of the livestock wastewater. The main characteristics of this livestock wastewater are that the pH was in the range of 8.4–8.7, the chemical oxygen demand using chromate (CODcr) was 5000–5700 mg/L. The maximum color absorbance at 287 nm was 2.1 and the color was dark grey [19].

### 2.2. Experimental procedure

The Fenton method was applied in 200 mL flasks containing 100 mL samples of livestock wastewater. All procedures were carried out at room temperature (22–25 °C) and at atmospheric pressure. First, the initial pH of the sample was adjusted to the desired pH value using 0.2N and 2N  $\text{H}_2\text{SO}_4$ . Then, a  $\text{H}_2\text{O}_2$  solution (34.5%, v/v) and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  powder were added to the flask and the mixture was vigorously stirred to dissolve the  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  powder for 1 min. The flask was then allowed to stand without stirring for 29 min. After that, the pH was neutralized to 7–8 (average 7.5) using 1N and 10N NaOH, and the precipitation was allowed to occur for 1 h in standing flasks. Experimental conditions were varied as follows. First, the initial pH, reaction time, and  $\text{Fe}^{2+}$  dose were kept constant while the  $\text{H}_2\text{O}_2$  dose was varied. Second, the initial pH, reaction time, and  $\text{H}_2\text{O}_2$  dose were kept constant while the  $\text{Fe}^{2+}$  dose was varied. Third, the reaction time,  $\text{H}_2\text{O}_2$  dose, and  $\text{Fe}^{2+}$  dose were

kept constant while the initial pH was varied. Fourth, the initial pH,  $\text{H}_2\text{O}_2$  dose and  $\text{Fe}^{2+}$  dose were kept constant while the reaction time was varied. Finally, all other conditions were kept constant while either  $\text{Fe}^{2+}$  dose or  $\text{H}_2\text{O}_2$  dose was given in several aliquots. The experimental procedures are shown in Fig. 1, where the initial pH was set at 4, the reaction time at 30 min, and the  $\text{Fe}^{2+}$  was given in either one or five doses and  $\text{H}_2\text{O}_2$  was given in either one or three doses.

### 2.3. Analytical methods

CODcr was measured by a closed reflux titrimetric method according to standard methods [20]. The pH values were measured with a pH meter (F-14, Horiba, Kyoto, Japan). The  $\text{H}_2\text{O}_2$  concentrations were measured using a  $\text{H}_2\text{O}_2$  sensor (DT-10D2 ABLE Co., Tokyo, Japan) in a response range of  $\geq 500$  mV (corresponding to  $\geq 100$  mg  $\text{H}_2\text{O}_2/\text{L}$ ) while test sticks (Quantofix, Germany) were used to measure  $\text{H}_2\text{O}_2$  concentrations in the range of 1–100 mg  $\text{H}_2\text{O}_2/\text{L}$ . Color intensities of samples were measured by absorption at 200–800 nm using a spectrophotometer (UV 2400, Shimadzu, Kyoto, Japan) after filtering the supernatant through 0.45  $\mu\text{m}$  filters. As the maximum absorbance of the samples was detected at 287 nm (see Fig. 3), the color removal ratio was calculated by dividing the difference between the initial and the final absorbances with the initial absorbance at 287 nm. Total solids (TS) were measured according to standard methods [20]. The reagents used were of analytical grade and were from the Kanto Chemical Co., Japan.

## 3. Results

### 3.1. Effect of $\text{H}_2\text{O}_2$ dose

It is important to optimize the amount of  $\text{H}_2\text{O}_2$  in the Fenton method because the main cost of the method is the cost of  $\text{H}_2\text{O}_2$  and excessive doses of  $\text{H}_2\text{O}_2$  trigger side effects [12]. The effects of  $\text{H}_2\text{O}_2$  doses in the range of 2993–17955 mg/L on CODcr and color removal ratios (%) were investigated at an initial pH of 4, a reaction time of 30 min,  $\text{Fe}^{2+}$  dose of 6554 mg/L and initial CODcr of 5700 mg/L. The result is shown in Fig. 2. The removal ratios of CODcr and color depended on the initial  $\text{H}_2\text{O}_2$  dose. The maximum CODcr removal ratio was 87.7% at 7980 mg/L of  $\text{H}_2\text{O}_2$  dose, when the color removal ratio was 92.9%. The maximum color removal ratio was 95.4% at 5985 mg/L of  $\text{H}_2\text{O}_2$  dose. At 11970 and 17955 mg/L of  $\text{H}_2\text{O}_2$  dose, CODcr removal ratios decreased to 85 and 81.3%, respectively, presumably because residual  $\text{H}_2\text{O}_2$  affected CODcr reduction. The spectra of the initial sample and the sample after 30 min of treatment are shown in Fig. 3. It is obvious that the peak at 287 nm was significantly reduced by addition of  $\text{H}_2\text{O}_2$ .

The degree of reduction depended on the doses of  $\text{H}_2\text{O}_2$ . The change in residual  $\text{H}_2\text{O}_2$  concentrations was measured for two different initial  $\text{H}_2\text{O}_2$  concentrations, 5985 and 11970 mg/L, when  $\text{Fe}^{2+}$  was supplied in two different ways. Namely, in one case, total amount of  $\text{Fe}^{2+}$  of 6554 mg/L was added at one time and in another case, 2185 mg/L (1/3 of 6554 mg/L) of  $\text{Fe}^{2+}$  was added at three divided times. The result is shown in Fig. 4. At an

Table 1  
Characteristics of livestock wastewater

Items	
pH	8.4–8.7
CODcr	5000–5700 mg/L
Total solid (TS)	5126–5900 mg/L
Color <sub>287</sub> <sup>a</sup>	2.1

<sup>a</sup> Absorbance at 287 nm.

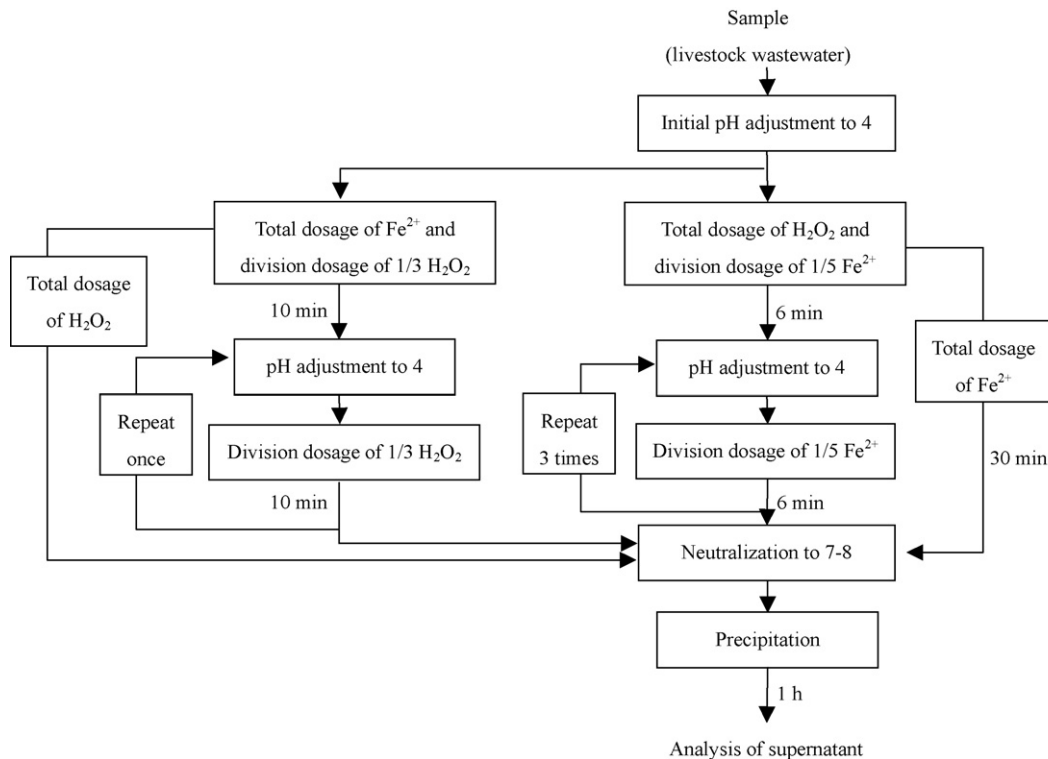


Fig. 1. Experimental procedures in this study including a single H<sub>2</sub>O<sub>2</sub> dosage and three division H<sub>2</sub>O<sub>2</sub> dosages and a single Fe<sup>2+</sup> dosage and five division Fe<sup>2+</sup> dosages (initial pH 4, reaction time = 30 min).

initial H<sub>2</sub>O<sub>2</sub> dose of 11970 mg/L, residual H<sub>2</sub>O<sub>2</sub> concentration leveled off at ca.700–800 mg/L after 30 min. H<sub>2</sub>O<sub>2</sub> acts as a scavenger of the highly potent hydroxyl radical to produce the hydroperoxyl radical, HO<sub>2</sub><sup>•</sup> as shown in Eq. (6) [12] but the HO<sub>2</sub><sup>•</sup> radical has much lower oxidation capabilities than the hydroxyl radical [21,22]:



A high concentration of residual H<sub>2</sub>O<sub>2</sub> therefore resulted in reduced COD<sub>Cr</sub> removal. This was reflected in the results of Fig. 2 in that the removal ratios of COD<sub>Cr</sub> and color leveled off at dose of H<sub>2</sub>O<sub>2</sub> more than 5985 mg/L. This means that at the optimum H<sub>2</sub>O<sub>2</sub> dose of 5985 mg/L, the H<sub>2</sub>O<sub>2</sub> dose (mg/L)/COD<sub>Cr</sub> concentration (mg/L) was 1.05 and this ratio may be used as a criterion to determine the H<sub>2</sub>O<sub>2</sub> dose for optimum COD<sub>Cr</sub> concentration.

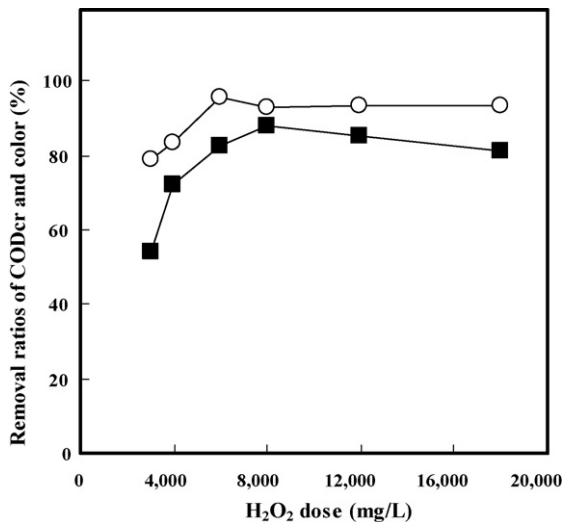


Fig. 2. Removal ratios of COD<sub>Cr</sub> (■) and color (○) with different initial H<sub>2</sub>O<sub>2</sub> doses (22 °C, initial pH 4.0, reaction time = 30 min, Fe<sup>2+</sup> dose = 6554 mg/L, COD<sub>Cr</sub> = 5700 mg/L).

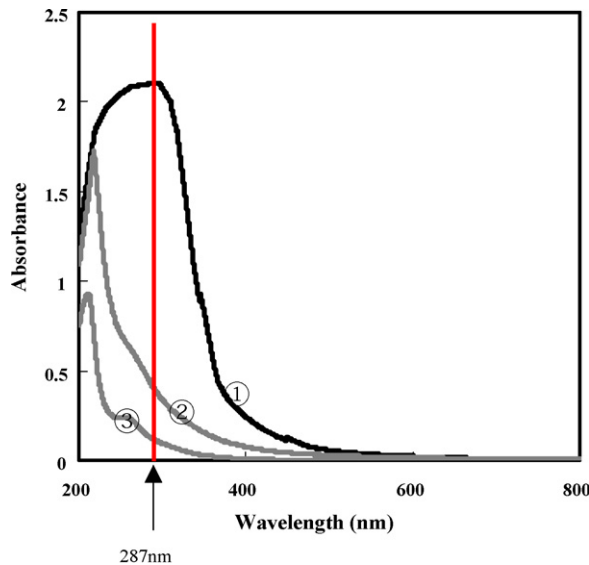


Fig. 3. Spectra of initial wastewater (①) and wastewater after 30 min treatment under the reaction conditions of pH 4, Fe<sup>2+</sup> dose of 6554 mg/L and H<sub>2</sub>O<sub>2</sub> doses of 2993 mg/L (②) and 5985 mg/L (③).

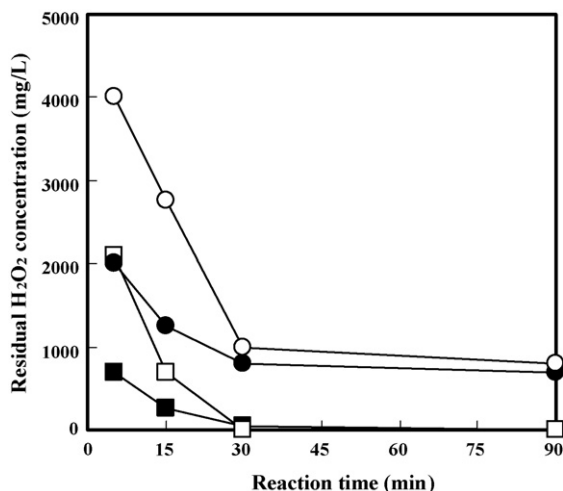


Fig. 4. Changes in residual H<sub>2</sub>O<sub>2</sub> concentrations with two different initial H<sub>2</sub>O<sub>2</sub> doses when Fe<sup>2+</sup> was added either once or in three aliquots: (■), H<sub>2</sub>O<sub>2</sub> dose = 5985 mg/L and Fe<sup>2+</sup> dose = (6554 × once) mg/L; (□), H<sub>2</sub>O<sub>2</sub> dose = 5985 mg/L and Fe<sup>2+</sup> dose = (2185 × 3 times) mg/L; (●), H<sub>2</sub>O<sub>2</sub> dose = 11970 mg/L and Fe<sup>2+</sup> dose = (6554 × once) mg/L; (○), H<sub>2</sub>O<sub>2</sub> dose = 11970 mg/L and Fe<sup>2+</sup> dose = (2185 × 3 times) mg/L (24 °C, initial pH 4.0).

### 3.2. Effect of the Fe<sup>2+</sup> dose and the H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio

The effect of the Fe<sup>2+</sup> dose on the removal ratios of CODcr and color are shown in Fig. 5, where the data derive from a sample at an initial pH of 4, a reaction time of 30 min, a H<sub>2</sub>O<sub>2</sub> dose of 5780 and 5500 mg/L of CODcr (H<sub>2</sub>O<sub>2</sub> dose/CODcr = 1.05). The Fe<sup>2+</sup> dose was varied between 1230 and 9486 mg/L and the H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio thus was varied from 1 to 8. The maximum removal ratios of CODcr and color were 88 and 95%, respectively at a Fe<sup>2+</sup> dose of 4743 mg/L (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup>

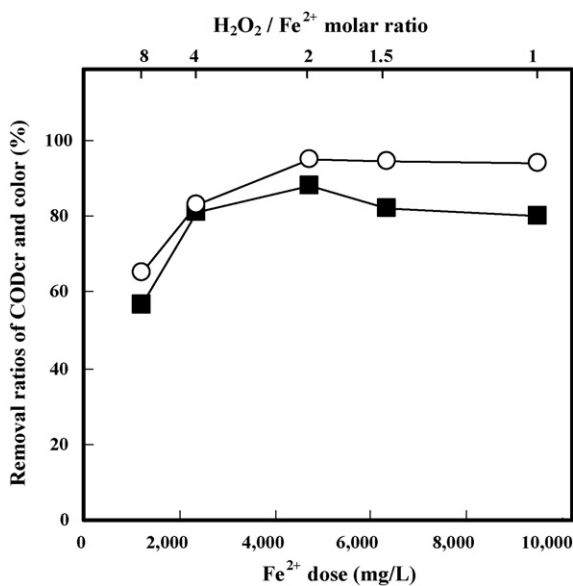


Fig. 5. Removal ratios of CODcr (■) and color (○) with different Fe<sup>2+</sup> doses (23 °C, initial pH 4.0, reaction time = 30 min, H<sub>2</sub>O<sub>2</sub> dose = 5780 mg/L and CODcr = 5500 mg/L).

molar ratio = 2). CODcr removal ratio decreased at Fe<sup>2+</sup> dose of ≥ 6324 mg/L. This is because hydroxyl radical may be scavenged by the reaction with excess Fe<sup>2+</sup> (Eq. (4)) [23]. In addition, the Fe<sup>3+</sup> formed can react with H<sub>2</sub>O<sub>2</sub> (Eq. (5)) to generate Fe<sup>2+</sup> and hydroperoxyl radicals (HO<sub>2</sub>·) in solution. This also results in a decrease in CODcr removal due to consumption of H<sub>2</sub>O<sub>2</sub> [11,23]. Thus, the optimum Fe<sup>2+</sup> dose was 4743 mg/L and the optimum H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio was 2.

### 3.3. Effect of initial pH

The effects of initial pH on removal ratios of CODcr and color are shown in Fig. 6. The initial CODcr concentration of livestock wastewater was 5000 mg/L and thus the H<sub>2</sub>O<sub>2</sub> dose (mg/L) was fixed at 5250 mg/L by using that the H<sub>2</sub>O<sub>2</sub> dose/CODcr should be 1.05. Then, the Fe<sup>2+</sup> dose was set at 4312 mg/L using that H<sub>2</sub>O<sub>2</sub>/Fe molar ratio should be 2. As the pH of the reaction mixture dropped by about 0.5–1.5 units after addition of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub>, the initial pH values were selected between 3.5 and 7. As can be seen in Fig. 6, maximum removal of CODcr and color were obtained at pH values of 3.5–4, and they decreased with increases in pH, under which conditions Fe<sup>2+</sup> is transformed to Fe<sup>3+</sup>. Then, the Fe<sup>3+</sup> has a tendency to produce ferric hydroxo complexes with H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> is unstable and readily decompose to oxygen and water [11]. It is reported that stable hydroxyl radicals are produced at pH values of 3–5 [8,9,24] and that high oxidizing potential was exhibited in this pH range [25].

### 3.4. Effect of reaction time

The reaction time was varied between 10 and 60 min. The initial pH, the H<sub>2</sub>O<sub>2</sub> dose, the Fe<sup>2+</sup> dose and the initial CODcr

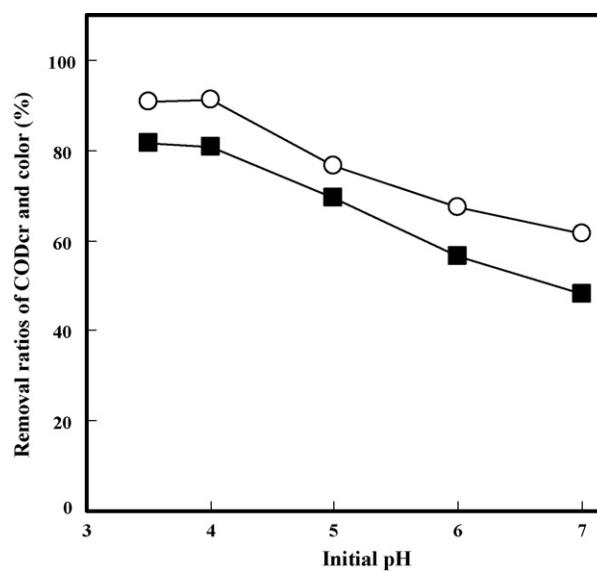


Fig. 6. Removal ratios of CODcr (■) and color (○) with different initial pH values (temperature = 23 °C, reaction time = 30 min, H<sub>2</sub>O<sub>2</sub> dose = 5250 mg/L, Fe<sup>2+</sup> dose = 4312 mg/L (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2) and CODcr = 5000 mg/L).

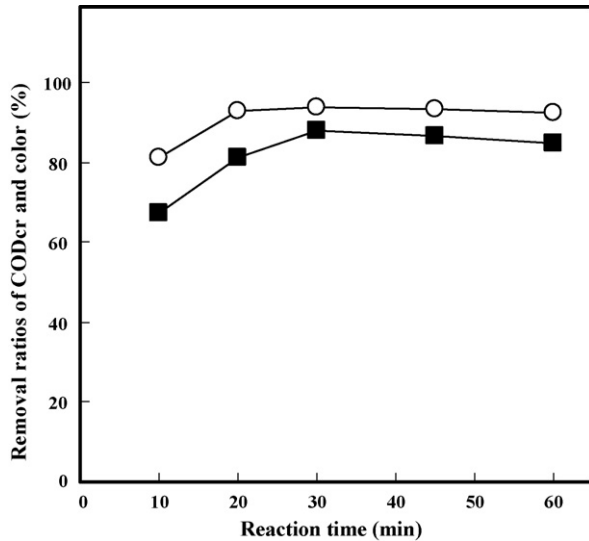


Fig. 7. Removal ratios of CODcr (■) and color (○) with different reaction times (23 °C, initial pH 4, H<sub>2</sub>O<sub>2</sub> dose = 5250 mg/L, Fe<sup>2+</sup> dose = 4312 mg/L (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2) and CODcr = 5000 mg/L).

(H<sub>2</sub>O<sub>2</sub>/CODcr = 1.05) were kept constant at 4, 5250, 4312 mg/L (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2) and 5000 mg/L, respectively. The result is shown in Fig. 7. After 10 min, the removal ratios of CODcr and color were significantly lower, but after 30 min removal ratios of CODcr and color were 86 and 94%, respectively. Thus, the optimum reaction time was determined to be 30 min. In 30 min, the supplied H<sub>2</sub>O<sub>2</sub> was probably almost completely consumed as shown in Fig. 4.

### 3.5. Effect of the dosage method

The effects of H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> dosage methods on removal ratios of CODcr and color are shown in Figs. 8 and 9, respectively. In these experiments, the initial pH was 4 and the reaction time was 30 min. The amounts of H<sub>2</sub>O<sub>2</sub> added and Fe<sup>2+</sup> added were 5780 and 4743 mg/L, respectively (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2), but the H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> dosages were divided into 1–3 aliquotes and 1–5 aliquotes, respectively. The initial CODcr concentration of livestock wastewater was 5500 mg/L and the H<sub>2</sub>O<sub>2</sub> dose/CODcr concentration ratio was fixed at 1.05. As shown in Figs. 8 and 9, there were no significant differences in CODcr and color removal under the conditions examined. This indicates that one-time addition of the Fenton's reagents permits maximum removal of CODcr and color.

## 4. Discussion

When high-strength wastewater such as piggery wastewater is treated in a microbial system such as activated sludge, dilution of the raw wastewater is required because excessive loads of carbon and nitrogen in the sludge reduce degradation rates. However, the dilution of raw wastewater requires a large amount of water, making pre-treatment of anaerobic digestion of high-strength wastewater an option. However, the volume of the anaerobic digester needs to be rather large due to the slow

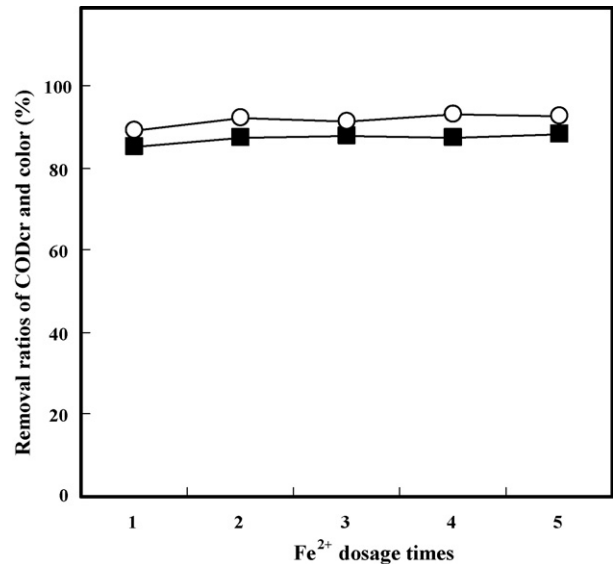


Fig. 8. Removal ratios of CODcr (■) and color (○) with different Fe<sup>2+</sup> dosage regimens (24 °C, initial pH 4, H<sub>2</sub>O<sub>2</sub> dose = 5780 mg/L). The total Fe<sup>2+</sup> dose was 4743 mg/L and this was delivered either as 4743 mg × once, 2372 mg × twice, 1581 mg × 3 times, 1186 × 4 times or 949 mg × 5 times (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2).

digestion rate. To reduce the volumes of treatment facilities, a first-stage chemical treatment to precipitate organic matters, followed by microbial treatment of the reduced organic matters loads is one possibility. The hydroxyl radical, one of the most powerful chemical oxidation reagents with a high oxidation potential ( $E^\circ = 2.8$  V), is capable of reacting with many organic species through a series of chain reactions [26]. In order to utilize the hydroxyl radical efficiently, the doses of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> should be optimized to the initial CODcr. Under optimized condition, the maximum removal ratios of CODcr and color were 88

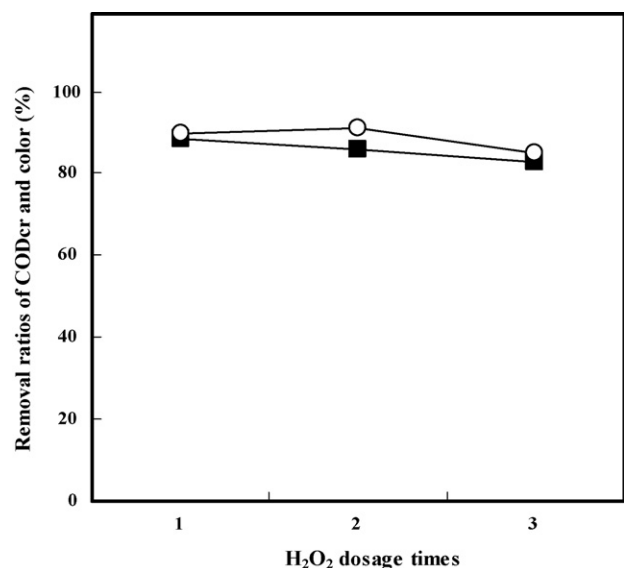


Fig. 9. Removal ratios of CODcr (■) and color (○) with different H<sub>2</sub>O<sub>2</sub> dosage regimens (25 °C, initial pH 4). The total H<sub>2</sub>O<sub>2</sub> dose was 5780 mg/L and this was delivered either divided dosages as 5780 mg × once, 2890 mg × twice or 1927 mg × 3 times. The Fe<sup>2+</sup> dose was 4743 mg/L (H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio = 2).

and 95.4%, respectively. From optimum  $\text{H}_2\text{O}_2/\text{COD}_{\text{Cr}} = 1.05$ , less than 50%  $\text{COD}_{\text{Cr}}$  could be oxidized theoretically. However, the actual removal of  $\text{COD}_{\text{Cr}}$  was significantly higher than 50%, and this indicates contribution of coagulation. After the Fenton treatment, the residual low-molecular compounds formed (residual low strength COD) may be subjected to biological treatment because microorganisms can decompose low-molecular-weight compounds. One of disadvantages of the Fenton method is that the sludge produced is high in iron [25]. One possible method for treatment of Fenton sludge is anaerobic digestion and this method is under investigation in our laboratory.

## 5. Conclusions

When the Fenton method of treating high-strength livestock wastewater is to be used, the following criteria may be helpful.

1. The  $\text{H}_2\text{O}_2$  dose (mg/L) should be 1.05 that of the initial  $\text{COD}_{\text{Cr}}$  concentration (mg/L).
2. The optimum  $\text{Fe}^{2+}$  dose should be ca. 4700 mg/L.
3. The initial pH should be 3.5–4, and the reaction time should be 30 min.
4. The  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio should be 2.

Under optimal experimental conditions, removal ratios of  $\text{COD}_{\text{Cr}}$  and color were  $\geq 80$  and  $\geq 95\%$ , respectively. Delivery of the Fenton reagents in aliquots did not affect  $\text{COD}_{\text{Cr}}$  and color removal. A single addition of the Fenton reagents is therefore satisfactory. The sludge produced may be subjected to anaerobic digestion and the treated wastewater may receive microbial treatment.

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