



# Pretreatment of water-based seed coating wastewater by combined coagulation and sponge-iron-catalyzed ozonation technology

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## HIGHLIGHTS

- Sponge iron (SFe) was firstly used as catalyst in ozonation.
- Operating parameters of CS-SFe/O<sub>3</sub> process were optimized.
- Degradation behavior of pollutants was analyzed by UV–vis, EEM and GC/MS.

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## ABSTRACT

Coagulation–sedimentation combined with sponge iron/ozone (CS-SFe/O<sub>3</sub>) technology was applied to pretreat water-based seed coating wastewater (WSCW) from pesticide manufacturing. Coagulation with polyferric sulfate at a dosage of 1.5 g L<sup>-1</sup> and a pH of 8.0 was effective, with color and chemical oxygen demand (COD) removal rates of 96.8 and 83.4%, respectively. SFe/O<sub>3</sub> treatment further reduced the organic content in the effluents, especially concerning the degradation of aromatic pollutants, as demonstrated via ultraviolet–visible spectrophotometry (UV–vis), excitation–emission matrix (EEM) fluorescence spectrometry, and gas chromatography–mass spectrometry (GC/MS) analyses. The residual color and COD values of the effluent were 581.0 times and 640.0 mg L<sup>-1</sup>, respectively, under optimal conditions (ozone concentration of 0.48 mg L<sup>-1</sup>, SFe dosage of 20.0 g L<sup>-1</sup>, initial pH of 9.0, and reaction time of 30 min). Organic pollutants were also degraded by the high amounts of HO•, which may have been generated via the transformation of ozone into HO• on the SFe's surface and in the solution. Meanwhile, the biochemical oxygen demand (BOD<sub>5</sub>)/COD ratio of the WSCW increased, which indicates that the biodegradability improved significantly. The amount of iron leached from SFe particles was 4.5 mg L<sup>-1</sup>, which shows that the SFe catalyst has good stability. The operating cost of the combined CS-SFe/O<sub>3</sub> technology was estimated at approximately 2.79 USD t<sup>-1</sup>. The results of this study suggest that the application of the combined CS-SFe/O<sub>3</sub> technology in WSCW pretreatment can be beneficial for removing suspended solids, degrading recalcitrant pollutants, and enhancing biodegradability for the subsequent bioprocessing treatment.

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## 1. Introduction

With increasing freshwater source scarcity, eliminating pollutants from wastewater in order to recycle it is becoming critical. The quantity of wastewater from global pesticide manufacturing is significantly increasing with rapid population growth and the

continued development of modern farming practices. Pesticide usage is widespread, as it increases crop yields and eliminates pests and diseases. However, this contributes to the presence of pesticide residues in water supplies, which can lead to serious environmental problems if they remain untreated (Coupe et al., 2000; Wu et al., 2009; Bilinska et al., 2016). Water-based seed coating agents are widely used to coat seeds (e.g., those of wheat, peanuts, and cereals) for pest control. WSCW is primarily generated when bactericide-manufacturing companies clean their equipment. WSCW is highly complex and contains binders; dyestuffs; bactericides; and emulsifying, antifreeze, and film-forming agents. This

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wastewater is characterized by high color levels and high concentrations of organic refractory compounds. Therefore, biological processes cannot be applied directly to treat WSCW (Craigmill and Winterlin, 1988; Cheng et al., 2007). However, bioprocessing treatments would be ideal, as they are environmentally sustainable and cost-efficient. Therefore, to meet strict environmental discharge management legislation, simpler, cheaper, and safer technologies for pretreating WSCW should be developed urgently.

Over the past few decades, many studies have demonstrated that physicochemical processes can degrade and mineralize recalcitrant compounds in wastewater (Asghar et al., 2015; Hong et al., 2009; Zhu et al., 2014). Among the various pollutant treatment technologies, advanced oxidation processes (AOPs) are the most attractive, as they can degrade concentrated wastewater from a variety of industrial and domestic sources (Zhang et al., 2013; Zhou et al., 2013). To date, various AOPs, including photocatalytic oxidation, Fenton oxidation, electrocatalytic oxidation, and ozone oxidation, have been developed and studied extensively (Jafry et al., 2011; Guerreiro et al., 2016; Kaur et al., 2018; Wert et al., 2009). Ozonation has been frequently used as a water and wastewater remediation technology because it can remove organic compounds. A considerable number of studies have been published that show that the catalytic ozonation process is effective at enhancing the reactions between ozone and organic compounds in wastewater treatment (via ozone decomposition). During ozonation, the application of ozone significantly increases the removal of refractory compounds as ozone transforms into more reactive species (such as HO $\cdot$ ) through adsorption and the reaction between pollutants and the catalyst surface (Chen et al., 2014; Faria et al., 2009; Wu et al., 2015).

Therefore, combined catalytic ozonation with zero-valent iron (ZVI) technology has been used as both pretreatment and post treatment for removing a variety of pollutants from wastewater, including omethoate (Qiang et al., 2013) and *p*-nitrophenol (PNP) (Xiong et al., 2016a), and for treating wastewater produced through dyeing and finishing (Wu et al., 2016), automobile coating (Xiong et al., 2017), and so on. Bare nanoscale ZVI (nZVI) or microscale ZVI (mZVI) particles are inclined to either react immediately with their surrounding medium (such as dissolved oxygen or water) or aggregate into larger-scale flocs, which reduces their reactivity and dispersity in the solution and limits further application. To overcome these obstacles, we selected sponge iron (SFe) as a catalyst to improve the performance of ozonation and develop a cost-effective pretreatment for WSCW. SFe has unique properties, including greater particle size, porosity, and structural stability in basic media, which enhance its catalytic activity during oxidation.

In addition, the predominant fractions of WSCW such as hydrophobic organic substances could form suspended solids owing to the presence of a variety of surfactants. Hence, coagulation–sedimentation (CS) is often applied prior to ozonation with ZVI. CS is widely employed in wastewater treatment for removing colloids, color, and organic pollutants (Maleki et al., 2009; Verma et al., 2012; Van Haute et al., 2015). Thus, the purpose of this study was to apply CS combined with SFe/O $_3$  technology to pretreat WSCW and ensure that the effluent meets the requirements of subsequent biochemical treatment to ultimately meet the updated environmental standards. Based on the WSCW characteristics, a suitable coagulant type and dosage were determined, and the influence of settling time on the coagulation effect for color and COD removal also was evaluated. The effluent produced via CS was then treated using the subsequent SFe/O $_3$  technology. Meanwhile, experiments were conducted to determine the optimal conditions and mechanism of organic matter removal for the combined CS-SFe/O $_3$  technology. Furthermore, variations in organic pollutant composition were characterized during each

stage of the integration process via UV–vis, EEM, and GC/MS analyses. This study demonstrates that the combined CS-SFe/O $_3$  technology is more efficient than CS or SFe–O $_3$ ; therefore, it could be applied more extensively in wastewater treatment.

## 2. Materials and methods

### 2.1. Wastewater and chemicals

WSCW samples were obtained from a pesticide manufacturing plant in Northern China. The physicochemical characteristics of the WSCW are presented in Table 1. The wastewater was stored at 4 °C prior to use, and sampling was conducted every 7 days.

Polyaluminum chloride (PAC), polymeric ferric sulfate (PFS), FeCl $_3$ , and polyacrylamide (PAM) were purchased from the Fuchen Chemical Reagent Company, China. The SFe (mean diameter of 3.0–5.0 mm) was provided by the Tianjin Liuying Water Purification Company, China. The SFe particles had a specific surface area of 80.0 m $^2$  g $^{-1}$  and an iron content of 98.0%. Deionized water was used to prepare all solutions used in this study.

### 2.2. Experimental procedure

#### 2.2.1. Coagulation experiments

Jar tests were conducted at room temperature (25 °C) using a jar test apparatus (ZR4-6, China), with PFS, PAC, and FeCl $_3$  as coagulants. A set dosage of the coagulants was added to 200 mL of WSCW, and the pH was adjusted to 8.0 by 0.1 M NaOH or 0.1 M H $_2$ SO $_4$ . Samples were mixed by an electric stirrer at 300 rpm for 1 min to thoroughly mix the coagulants with the WSCW. They were then mixed at 60 rpm for 5 min to allow flocs to grow and subsequently allowed to stand for different times for floc settlement. Supernatant was collected from 1 cm below the water surface and filtered through 0.45  $\mu$ m membranes for further analysis. PAM was also added to the CS process and the effluent of CS process could be named as ECS. The optimal experimental conditions were based on preliminary experiments.

#### 2.2.2. SFe/O $_3$ experiments

In this study, ozone with a concentration of approximately 5.3 mg L $^{-1}$  was generated using a corona discharge ozone generator (SW-005-10G, Qingdao Weiste Co., Ltd., China) with air as the feed gas. The gas produced by the ozone generator (O $_2$  and O $_3$  gas mixture) was then sparged into the bottom of the reactor at different flow rates using diffuser stones. A dissolved ozone tester (OZS 30, CLEAN, USA) was used to monitor the aqueous ozone concentration. The residual ozone in the off-gas was absorbed by a KI solution.

All SFe/O $_3$  treatments were performed in a semi-batch environment. The required dosage of SFe was added into a 1000 mL glass reactor containing 500 mL of ECS. After pH adjustment, the ozone was injected into the ECS by continuously bubbling the ozone gas, and then the reaction continued for a specific period.

**Table 1**  
Characteristics of WSCW and the effluent after different processes.

Parameters	WSCW	ECS <sup>a</sup>	After CS-SFe/O $_3$ process
Color	Dark purple red	Light pink	yellowish
Color values (times)	31390.0 $\pm$ 100	1004.0 $\pm$ 86	581.0 $\pm$ 35
COD (mg L $^{-1}$ )	5420.0 $\pm$ 125	900.0 $\pm$ 42	640.0 $\pm$ 26
BOD $_5$ (mg L $^{-1}$ )	1410.0 $\pm$ 52	342.0 $\pm$ 25	332.0 $\pm$ 36
BOD $_5$ /COD	0.26	0.38	0.52
pH	6.0 $\pm$ 0.03	7.13 $\pm$ 0.34	6.87 $\pm$ 0.62

<sup>a</sup> The effluent produced by CS.

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