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**Research article** 

# Effect of K<sub>2</sub>FeO<sub>4</sub>/US treatment on textile dyeing sludge disintegration and dewaterability



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#### ABSTRACT

The effect of potassium ferrate/ultrasonic (K<sub>2</sub>FeO<sub>4</sub>/US) treatment on the physicochemical features of textile dyeing sludge was studied. The soluble chemical oxygen demand (SCOD), deoxyribonucleic acid (DNA), sludge volume index (SVI), sludge viscosity, capillary suction time (CST) and particle size were measured to understand the observed changes in the sludge physicochemical features. The results showed that the combined K<sub>2</sub>FeO<sub>4</sub>/US treatment presented great advantages for disrupting the sludge floc structure over K<sub>2</sub>FeO<sub>4</sub> or ultrasonic treatments alone. The optimal parameters of sludge disintegration were found to be a K<sub>2</sub>FeO<sub>4</sub> treatment time of 60 min, a K<sub>2</sub>FeO<sub>4</sub> dosage of 0.5936 g/g SS, an ultrasonic time of 15 min and an ultrasonic intensity of 0.72 W/mL. The initial median diameter of the sludge particles was 15.24  $\mu$ m, and this value decreased by 35.89%. The CST was initially 59.6 s and increased by 231%, whereas the SVI was 97.78 mL/g and decreased by 25.89%. Scanning electron microscope (SEM) images indicated that the sludge surface was irregular and loose with a large amount of channels or voids during K<sub>2</sub>FeO<sub>4</sub>/US treatment. K<sub>2</sub>FeO<sub>4</sub>/US treatment synergistically enhanced the sludge solubilization and reached 668.67 mg/L SCOD, which is 31.81% greater than the additive value obtained with K<sub>2</sub>FeO<sub>4</sub> treatment alone (215.95 mg/L) or with ultrasonic treatment alone (240 mg/L).

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#### 1. Introduction

With the development of the textile dyeing industry in China, textile dyeing sludge, which has a strong color and a high concentration of suspended solids, was largely produced by the textile dyeing wastewater treatment process (dos Santos et al., 2007). Simultaneously, the management of large quantities of excess sludge generated by a conventional activated sludge treatment plant usually accounts for 50–60% of the total operating cost (Luo et al., 2013). In addition, sludge reduction is a promising approach in the textile dyeing industry for meeting the increasingly stringent environmental regulations. Some novel sludge reduction alternatives, including metabolic uncoupling, predation on bacteria, lysis-cryptic and other improved sludge reduction processes, have recently been used (Guo et al., 2013). The lysis-cryptic method may be a sustainable way to minimize the sludge yield because it could efficiently recycle material within the textile dyeing sludge

without releasing any harmful substances to humans or the environment. When a cell is lysed, huge amounts of the cell contents and nutrients are released into the aqueous phase, resulting in an increase in organic material, and then reused for metabolism in the aeration tank where biodegradation occurs (Mohammadi et al., 2011). However, because of the complex structure of sludge, hydrolysis is the rate-limiting step of sludge biodegradation. The release of substances inside the cells into the aqueous phase is the key for success (Xu et al., 2010).

Several methods, including physical (Tiehm et al., 2001), chemical (Kamiya and Hirotsuji, 1998) and biological methods (Wu et al., 2014), have been developed for the hydrolysis of sludge cells prior to sludge disintegration. Previous research has allowed the development of chemical methods to disintegrate excess sludge, such as ozonation (He et al., 2006), chlorination (Saby et al., 2002), hydrogen peroxide oxidation (Kim et al., 2009) and Fenton oxidation (He and Wei, 2010). Although ozonation is a more advanced method of sludge disintegration and had been successfully developed, its generation and application are too costly (Saby et al., 2002). To enhance the cost-effectiveness of sludge disintegration, chlorination has been used to replace ozonation. However,



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trihalomethanes (THMs) are produced during the chlorination process, and these may pose a serious threat to human beings (Guo et al., 2013). The biodegradation of refractory organics has been reported to be expensive (Sarria et al., 2002), and these compounds are non-readily degradable (Zhu et al., 2006) compared with other options. Ultrasound (US) treatment, which is one of the physical methods developed, is characterized as an energy-intensive, efficient and environmentally friendly method. As a result, US treatment has been widely used to disrupt sludge flocs and enhance the lysis of sludge bacteria during the lysis-cryptic growth process (Grönroos et al., 2005). During US treatment, acoustic cavitation induces different effects, including chemical reactions by radicals, pyrolysis, combustion and shearing (Ning et al., 2014). Tiehm et al. (2001) studied sludge disintegration by US treatment (3.6 KW, 31 KHz, 64 s) and demonstrated that the SCOD in the supernatant increased from 630 mg/L to 2270 mg/L.

Potassium ferrate ( $K_2FeO_4$ ), a black-purple compound, is another powerful oxidant with a wide pH range and is considered to be an ideal alternative oxidant to replace ozonation and chlorination compounds in water and wastewater treatment (Li et al., 2005). It has a high redox potential, as determined through Eqs. (1) and (2):

$$FeO_4^{2-} + 8H^+ + 3e^- = Fe^{3+} + 4H_2O \quad E = 2.20 V$$
 (1)

$$FeO_4^{2-} + H_2O + 3e^- = Fe(OH)_3 + 5OH - \quad E = 0.70 \ V \equal (2)$$

During the process, ferrate (VI) ions are reduced to Fe (III) ions and simultaneously form a coagulant (Fe(OH)<sub>3</sub>). With a high oxidation capacity, K<sub>2</sub>FeO<sub>4</sub> is able to oxidize organic and inorganic materials by disrupting colloidal contaminants, such as chlorophenols (Graham et al., 2004), organosulfur compounds (Sharma et al., 2011) and antibiotics (Anquandah et al., 2011). Moreover, K<sub>2</sub>FeO<sub>4</sub> is a coagulant and can be used as a transformer of fine particles into large aggregates (Jiang et al., 2006), to promote the removal of heavy metals (Prucek et al., 2013) and as an environmentally friendly oxidants in water and wastewater treatment (Jiang et al., 2006).

It has been well known that the incorporation of chemical oxidation into sludge disintegration is more efficient than chemical oxidation alone due to its easy control ability, high operating flexibility, high performing stability and low cost (Wang et al., 2011). Therefore, US-assisted K<sub>2</sub>FeO<sub>4</sub> treatment may be a promising method for sludge disintegration. However, few studies have addressed the effect of K<sub>2</sub>FeO<sub>4</sub>/US technology on the physicochemical properties of textile dyeing sludge and the relationship between sludge dewaterability and disintegration.

This study aimed to investigate the effect of  $K_2FeO_4/US$  treatment on the physicochemical features of textile dyeing sludge. The mechanism underlying the changes observed in sludge disintegration and dewaterability is also discussed.

#### 2. Materials and methods

#### 2.1. Sludge and apparatus

The sludge samples used in this study were obtained from a textile dyeing wastewater treatment plant (Dongguan City, China), where the treating capacity was 9000 m<sup>3</sup>/d using an A/O activated sludge process. To acquire thickened sludge samples, the sludge was settled gravitationally for 24 h, and the supernatant was then poured out. The thickened samples were stored in plastic containers placed in a refrigerator at 4 °C until use. The average values of the sludge sample characteristics are the following: pH 7.06; moisture content, 98.63%; soluble chemical oxygen demand,

32.00 mg/L; protein 2.44 mg/L; polysaccharide, 1.01 mg/L; deoxy-ribonucleic acid, 0.01 mg/L; and capillary suction time, 59.60 s.

Potassium ferrate (purity  $\geq$  93%) was purchased from Zhenpin Chemical Company (Shanghai, China) and stored in a vacuum desiccator prior to use. All other chemicals were of analytical grade.

Sludge sample disintegration with ultrasonic irradiation was executed in a JY99-IIDN system (Xinzhi Biotechnology Ltd.) with a probe diameter of 25 mm, a frequency of 20 KHz and an ultrasonic energy ranging from 180 to 1800 W. To maintain the temperature of the sludge samples in the beaker to  $20 \pm 2$  °C, the sono-tank method was conducted using an ice water bath.

#### 2.2. Experimental procedure

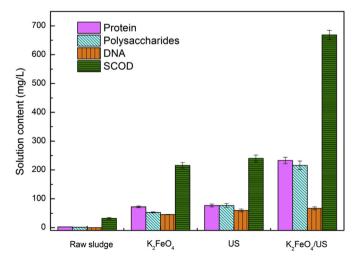
Sludge disintegration by  $K_2FeO_4/US$  was performed with a 500mL sludge sample in a series of bench-scale experiments. The sludge oxidation reaction was then initiated by dosing powdered  $K_2FeO_4$  into a 1000-mL beaker, and the mixture was then stirred at 120 rpm for 60 min using a six-joint stirrer (ZR4-6, Zhongrun, China) to ensure that the reaction was finished.

The K<sub>2</sub>FeO<sub>4</sub>-pretreated sludge (500 mL) was transferred to the ultrasonic reactor. During US treatment, the ultrasonic probe was immersed to approximately 1 cm below the surface of 500 mL of sludge in the middle of the beaker. The ultrasonic time was varied from 5 to 35 min and the density was ranged from 0.36 to 2.88 W/ mL to obtain the optimal digestion conditions.

The supernatant samples were collected by centrifugation at 4000 rpm for 10 min and filtered through a 0.45-µm-pore-size membrane before subsequent analysis. The filtered supernatant was used to measure the SCOD, protein, polysaccharide and DNA. Each measurement was performed in triplicate, and the average values were obtained.

#### 2.3. Analytical methods

After K<sub>2</sub>FeO<sub>4</sub>/US treatment, the SCOD was determined through the dichromate titrimetric method according to standard methods (Taras, 1971), and the SVI was utilized to evaluate the sludge settleability, which was defined as the sludge volume per gram of dry solid (DS) after 30 min of sedimentation. The sludge particle size distributions were assessed with an eye tech particle size and shape analyzer (Ankersmid Ltd. Co., Netherlands), which enabled the



**Fig. 1.** Comparison of the solution contents obtained after the sludge disintegration by  $K_2FeO_4$ , US and  $K_2FeO_4/US$  (pH 7.06,  $K_2FeO_4$  dose = 0.5936 g/g SS, US energy density = 0.72 W/mL, US time = 15 min).

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