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# Effects of potassium ferrate oxidation on sludge disintegration, dewaterability and anaerobic biodegradation



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# ABSTRACT

Chemical oxidation is a common sludge pretreatment method. This paper studied the effects of potassium ferrate ( $K_2FeO_4$ ) oxidation on sludge disintegration, dewaterability and anaerobic biodegradation. Results showed that  $K_2FeO_4$  oxidation effectively disintegrated sludge, improved sludge dewaterability and enhanced sludge biodegradability. The suitable  $K_2FeO_4$  dosages for sludge disintegration, dewaterability and anaerobic digestion were all 500 mg/L. The maximum sludge disintegration degree ( $DD_{COD}$ ) reached 69% with a  $K_2FeO_4$  dosage of 500 mg/L. After  $K_2FeO_4$  oxidation, extracellular polymeric substance (EPS) concentration in the supernatant increased, and the increase of polysaccharides was larger than that of proteins.  $K_2FeO_4$  oxidation deteriorated the sludge settleability, but significantly improved the sludge dewaterability. Both sludge specific resistance to filtration (SRF) and viscosity decreased after  $K_2FeO_4$  oxidation especially the sludge SRF reduced by 85% with a  $K_2FeO_4$  dosage of 500 mg/L. For sludge anaerobic biodegradation, the cumulative biogas production remarkably increased with 500 mg/L  $K_2FeO_4$  pretreatment, and the final cumulative biogas production increased by approximately 44% compared to control.

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# Introduction

Facing with the severe water pollution, activated sludge process is the most widely used biological wastewater treatment technology (Qu and Fan, 2010). While a large amount of sludge produced in the wastewater treatment processes has become a serious problem, and the excess sludge, an unavoidable biowaste of wastewater treatment, must be properly disposed. The cost for sludge treatment and disposal may account for up to 65% of the total wastewater treatment plant operating costs (Liu, 2003). So sludge treatment and disposal is very important. Anaerobic digestion is well known as the biological process to treat the sewage sludge by degrading organic matters, generating bio-energy and killing pathogens. The conversion of organic matter to biogas occurs in a sequence of four steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Sanchez-Hernandez et al., 2013). However, the

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application of anaerobic digestion is limited by a long retention time of 20–30 days due to the rate-limiting step of hydrolysis (Pavlostathis and Gossett, 1986; Shimizu et al., 1993).

For this reason, different disintegration techniques have been applied to improve the sludge hydrolysis, thus enhancing solid removal and biogas production (Zhang et al., 2008; Park et al., 2013), including physical, chemical and biological, and combined methods. Ultrasonic treatment (Pham et al., 2009) is one typical physical disintegration method, and the study showed as much as 40% of improvement in sludge solubilization following ultrasonic pretreatment. Ozone oxidation is the most widely used chemical disintegration method (Magdalena et al., 2007), and an ozone dosage of 10 mg/(g MLSS  $\cdot$  d) for sludge disintegration resulted in a reduction of 50% sludge production (Kamiya and Hirotsuji, 1998). Alkaline treatment is normally used together with thermal treatment (Barjenbruch and Kopplow, 2003). Previous investigation reported the increase of soluble chemical oxygen demand (SCOD) and decrease of volatile suspended solid (VSS) even with a low dosage (<0.1 mol/L) alkaline treatment combining with thermal treatment (Cassini et al., 2006). Biological disintegration is normally achieved by adding microorganisms, microbial cell extract or metabolites of microbial cells into the sludge. However, these

techniques have disadvantages, such as affecting subsequent further processes due to chemical addition, high cost and energy requirements, and limitations in being scaled up for field application (Pilli et al., 2011; Lee et al., 2013).

 $K_2FeO_4$  is a strong oxidant that can oxidize organic compounds efficiently (Zhu et al., 2006). The oxidizability of  $K_2FeO_4$  is stronger than most traditional oxidizing agents and maintains high within the whole pH range.  $K_2FeO_4$  has been used for water disinfection, deodorization, and algae inhibition. Besides,  $K_2FeO_4$  is reduced to  $Fe^{3+}$  which is an excellent flocculant for most pollutants. Ye et al. (2012a) reported that  $K_2FeO_4$  oxidation was a feasible method for disintegration of excess activated sludge and the sludge solubility (SCOD/TCOD) reached 0.32 with a  $K_2FeO_4$  dosage of 0.81 g/g SS.

When sludge disintegration pretreatment is applied, microbial cells undergo lysis or death and then the intracellular contents are released, so the hydrolytic stage of anaerobic digestion can be shortened (Onyeche et al., 2002; Bougrier et al., 2005). With an alkaline pretreatment at pH 9–11, biogas production improved by 7.2%–15.4% (Shao et al., 2012). With a thermal pretreatment at 190 °C, methane production increased by 25%. With an ultrasound pretreatment, Neis et al. (2000) reported an enhancement of anaerobic digestion by 42.4% at an ultrasound intensity of 18 W/ cm<sup>2</sup>.

With the sludge disintegration pretreatment, not only the sludge hydrolysis can be accelerated by the increase of dissolved components, but the sludge biodegradability, dewatering and reduction of pathogens and foaming can also be improved (Rani et al., 2013). Sludge dewaterability has been reported as correlating with many factors, such as particle size distribution, extracellular polymeric substances (EPS) contents, colloidal particle amount, surface charge, etc. (Kopp et al., 1997; Xu et al., 2011). Zhang et al. (2012) reported that K<sub>2</sub>FeO<sub>4</sub> oxidation enhanced sludge dewaterability when pH was 3, while caused deterioration of sludge dewaterability at pH of 4–8. Ye et al. (2012b) reported K<sub>2</sub>FeO<sub>4</sub> oxidation improved sludge settleability, and slightly reduced water content of dewatered cake, but sludge capillary suction time increased after K<sub>2</sub>FeO<sub>4</sub> oxidation.

 $K_2FeO_4$  is a strong oxidant with a high potential for effective sludge disintegration, thus to change anaerobic digestion performances and sludge dewaterability. However, researches about sludge disintegration with  $K_2FeO_4$  oxidation are limited; the effect of  $K_2FeO_4$  oxidation on sludge dewaterability is unclear; and there is no research on sludge anaerobic biodegradation. Therefore, this paper deals with the effects of  $K_2FeO_4$  oxidation on sludge disintegration, dewaterability and anaerobic biodegradation systematically. Sludge oxidation feasibility was analyzed with the change of SCOD/TCOD, particle size and Fe concentration in supernatant. Sludge disintegration was represented by DD<sub>COD</sub> and EPS concentration in the supernatant. Sludge dewaterability was evaluated by specific resistance to filtration (SRF) and viscosity. Sludge anaerobic biodegradation was evaluated with biogas production during sludge digestion.

#### Materials and methods

#### Materials

Excess activated sludge was collected from Qinghe sewage treatment plant in Beijing, China. In this plant, about 75,000 m<sup>3</sup>/ d of municipal wastewater was treated using anaerobic/anoxic/oxic process. The collected sludge samples were transferred to laboratory within 60 min and then stored in a refrigerator at 4 °C. Seed sludge for sludge digestion was obtained from an UASB bioreactor in the laboratory, which was cultivated for six months. The

Table 1
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Characteristics of excess sludge and seed sludge.

	TS (g/L)	VS (g/L)	TCOD (mg/L)	SCOD (mg/L)	рН
Excess sludge	9.6 ± 0.3	7.1 ± 0.4	10,427 ± 36	93 ± 5	$6.86 \pm 0.14$
Seed sludge	10.1 ± 0.5	6.9 ± 0.3	11,500 ± 20	500 ± 8	$7.25 \pm 0.32$

characteristics of excess activated sludge and seed sludge are shown in Table 1.

# Sludge disintegration

A series of bench-scale experiments were conducted with a sample volume of about 500 ml. Sludge sample was preheated to the room temperature. Then sludge oxidation was initiated immediately by adding different  $K_2FeO_4$  dosages. Two or three replicates for each experimental condition were performed with the sample analysis in triplicate to ensure the reproducibility of results. The average values were reported.

Sludge disintegration degree ( $DD_{COD}$ ) was defined as Eq. (1),

$$DD_{COD} = \frac{SCOD - SCOD_0}{TCOD - SCOD_0}$$
(1)

where  $SCOD_0$  means the SCOD of sludge sample without  $K_2FeO_4$  oxidation, and TCOD was the total COD of untreated sludge.

### Anaerobic sludge digestion

After filling with 30 ml of seed sludge and 100 ml of excess activated sludge into 250 ml serum bottles, the serum bottles were flushed with N<sub>2</sub> to create an anaerobic environment. A blank bottle was prepared, containing 30 ml of seed sludge and 100 ml of distilled water. All bottles were placed in a shaking bath that was controlled at 35 °C. The final biogas production was the value that was subtracted from the control. Each sample was tested with three experimental runs.

# Analysis

APHA standard methods (Eaton et al., 2005) were used to test the total solids (TS), volatile solids (VS), COD. The pH was tested with a pH meter (PHS-3C, Leici Co., China). Particle size distribution was assessed with an Eye Tech Particle Size and Shape Analyzer (Ankersmid Ltd. Co., Netherlands) with a measurement range from 0.1 to 300.0  $\mu$ m. Fe concentration in the supernatant was tested with an atomic absorption spectrophotometer (A & E Laboratory Ltd. Co., China). Protein and polysaccharide contents were determined spectrophotometrically using a double beam ultraviolet—visible spectrophotometer (Beckman Coulter DU 640, USA). Proteins were determined by modified Lowry method (Frølund et al., 1995), and polysaccharides were measured by anthrone method (Riesz et al., 1985).

Viscosity was determined using a rotational viscosity meter (NDJ-5S, China) at room temperature. Sludge SRF was measured following the method of Feng et al. (2009). Sludge settleability was evaluated by carrying out a SV<sub>30</sub> test according to APHA standard method (Eaton et al., 2005). Biogas production was measured every day by the liquid displacement method (acidified saturated NaCl solution).

Tukey's test was adopted to analyze the significance of the values to ensure the level of significance above 95% (P < 0.05).

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