



Effects of ultrasound assisted Fenton treatment on textile dyeing sludge structure and dewaterability

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HIGHLIGHTS

- US/Fenton treatment was applied to textile dyeing sludge.
- US/Fenton treating presented disintegration advantages over Fenton treating alone.
- The changes of sludge dewaterability were dependent on sludge disintegration.
- The synergetic effect of US/Fenton was confirmed in term of the formation of $\cdot\text{OH}$.

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ABSTRACT

The effect of ultrasound assisted Fenton (US/Fenton) treatment on the physicochemical properties of textile dyeing sludge was investigated in this paper. The sludge particle size, total organic carbon (TOC), extracellular polymeric substance (EPS) concentration and sludge disintegration degree (DD_{SCOD}) were measured in an attempt to understand the observed changes in sludge physicochemical properties. The results showed that the US/Fenton-treated sludge presented obvious advantages over the Fenton-treated sludge for disrupting sludge floc structure. The suitable condition for sludge disintegration was found to be 0.12–0.16 W/ml (ultrasonic density) and lower pH (≤ 3), under which the TOC in soluble phase increased obviously by releasing organic substances of sludge flocs. The changes of sludge dewaterability were dependent on sludge disintegration during US/Fenton treatment. The optimal EPS concentration and DD_{SCOD} to obtain maximum sludge dewaterability were found to be 90–100 mg/l and 4–6%, respectively. After 10 min of Fenton and US/Fenton treatment, the hydroxyl radical ($\cdot\text{OH}$) was 2.71 $\mu\text{mol/l}$ and 2.90 $\mu\text{mol/l}$, respectively, which confirmed that US/Fenton treatment had a synergetic effect.

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1. Introduction

With the expansion of textile dyeing industry in China, textile dyeing wastewater treatment, which includes physicochemical processes (e.g., coagulation/flocculation) and biodegradation, produces large quantities of textile dyeing sludge. Large amounts of sludge are generated from wastewater treatment process because textile dyeing wastewater is characterized by strong color and high concentration of suspended solids [1]. Treatment and disposal of sludge generated in the process may account for up to 60% of the total operation expenses [2]. To lower the cost of sludge transportation and disposal, it is imperative to reduce the moisture content of the raw sludge. However, sludge is hard to be dewatered due to its biological gel structure.

Several methods have been investigated to enhance sludge dewaterability, which include the addition of acids and surfactants

[3], calcined aluminum salts [4], fungal treatment [5], Fenton's reagent pretreatment [6,7], ultrasonication [8,9], microwave conditioning [10,11]. Most of these processes are dependent on the disruption of extracellular polymeric substances (EPS), and as a result sludge dewaterability is improved. EPS, one of the major components of the activated sludge flocs, has a significant affinity for water [12,13]. The water entrapped in the EPS-structure is bound mainly by polysaccharides and proteins which are the main compositions of EPS [14]. Considering the highly hydrated property of EPS, it is believed that sludge dewatering efficiency can be promoted by degrading EPS.

Ultrasound is well known for disrupting sludge flocs and microbial cell walls [12], and it can enhance sludge dewaterability via the disintegration of cell structure and the release of bound water [15]. Ultrasound induces most of its physical and chemical effects in the reaction system through the phenomenon of cavitation [16]. The physical effect of cavitation is the generation of intense convection in the medium through the phenomena of microturbulence and shock waves [17], whereas the chemical effect of cavitation is the generation of radical species such as oxygen (O^\cdot), hydroxyl

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($\cdot\text{OH}$) and hydroperoxyl ($\text{HO}_2\cdot$) through the dissociation of solvent vapor during transient collapse of cavitation bubbles [18,19]. Tiehm et al. [20] observed that ultrasound could disintegrate sludge and release organic substances into liquid phase. They also found that sludge disintegration was most significant at low ultrasonic frequencies. Feng et al. [8] reported that approximately 400–500 mg/l of EPS concentration was the optimum value during ultrasonic treatment to yield maximum sludge dewaterability. Further increase in EPS concentration resulted in the rapid increase in capillary suction time (CST) and specific resistance to filtration (SRF).

Fenton technology is one of the most effective advanced oxidation processes. The oxidation mechanism of Fenton's reagent is based on the generation of $\cdot\text{OH}$ which results from the use of ferrous iron (Fe^{2+}) to react with hydrogen peroxide (H_2O_2) under acidic conditions [21]. As for waste activated sludge, the degradation of EPS, which represent up to 80% of the mass of activated sludge [22], may attribute to the $\cdot\text{OH}$ generated from Fenton's reagent with powerful oxidizing abilities. For decades, Fenton's reagent application on the improvement of sludge dewatering property has been extensively investigated. For instance, Buyukkamaci [23] investigated the optimization of Fe^{2+} and H_2O_2 dosages to improve biological sludge dewaterability during Fenton reaction process. Tony et al. [6] used Fenton's reagent ($\text{Fe}^{2+}/\text{H}_2\text{O}_2$) to condition aluminium-based sludge from drinking water purification. However, the addition of high concentration of H_2O_2 may increase the volume of sludge, which limits practical application of Fenton technology.

Ultrasound assisted Fenton (US/Fenton) treatment is a novel treatment option. Ultrasound can enhance the Fenton's oxidation rate due to the generation of more $\cdot\text{OH}$ caused by the cavitation within ultrasonic irradiation. Moreover, ultrasonic irradiation produces the intense micromixing resulted from various mechanisms such as microstreaming through ultrasound propagation and microturbulence through cavitation bubbles for facilitating the utilization of $\cdot\text{OH}$ generated from either transient cavitation or Fenton's reagent [24]. US/Fenton has received significant attention on wastewater treatment [25,26]. However, as far as is known, little work has been done to address the effect of US/Fenton technology on the physicochemical properties of textile dyeing sludge. Furthermore, less information is available on the relationship between the changes of sludge dewaterability and sludge disintegration.

This paper aims to examine sludge disintegration and dewaterability synchronously during US/Fenton treatment process, and to analyze their relationship. The mechanism behind the changes observed in sludge dewaterability is also discussed.

2. Materials and methods

2.1. Sludge and apparatus

The sludge samples for this experiment were obtained from a textile dyeing wastewater treatment plant (Dongguan City, China), where the treating capacity is 9000 m³/d using A/O activated sludge process. The sludge samples were precipitated gravitationally for 24 h and the supernatant was decanted to obtain thickened sludge samples. The thickened samples were stored in a refrigerator at 4 °C for use. The sludge characteristics were presented in Table 1.

The ultrasonic irradiation of sludge was performed in an ultrasonic cleaner (KQ2200DA, China) that emitted a 28 kHz ultrasound through the bottom of the reactor. The actual power density in the liquid phase could be changed from 0 W/ml to 0.16 W/ml by manually adjusting the electrical power of the reactor.

Table 1
Characteristics of raw sludge sample.

Parameters	Average value
pH	6.62
Moisture content (%)	98.35
Total chemical oxygen demand (mg/l)	9206
Soluble chemical oxygen demand (mg/l)	96
Total suspended solids (mg/l)	17429
Volatile suspended solids (mg/l)	7072

2.2. Experimental procedure

The sludge samples were treated with varying pH values and ultrasonic power densities in order to investigate the suitable conditions for sludge disintegration. H_2O_2 dosage of 428 mg/g and Fe^{2+} dosage of 42.8 mg/g were set as the desired values for this experiment according to the previous studies [27]. Additionally, experiment with Fenton treatment alone was accessed in term of particle size in order to observe the influence of Fenton reaction on the disintegration degree of sludge.

For US/Fenton treatment, sludge samples of 500 ml were initially adjusted to the required pH value by sulfuric acid (H_2SO_4) and were transferred to the reactor of the ultrasonic cleaner. Ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was added into the sludge samples and Fenton reaction was then initiated after adding H_2O_2 (30 wt.%). Then ultrasonic irradiation exposed to the sludge samples immediately. Samples were collected at desired time intervals. For Fenton reaction or ultrasonic treatment, the sludge samples were treated with only Fenton's reagent or ultrasonic irradiation at the above-mentioned experimental conditions, respectively. After US/Fenton treatment, sludge dewaterability was represented by CST and SRF. The viscosity of sludge was also examined to help evaluate the dewatering results. The EPS concentration and sludge disintegration degree (DD_{SCOD}) of the sludge samples were measured to investigate the mechanism behind the observed changes in sludge dewaterability.

2.3. Analytical methods

Sludge dewaterability was determined by CST, which was measured using a standard apparatus (304 M, Triton, UK). The standard Buchner funnel test was also selected to determine sludge dewaterability and was expressed in terms of SRF. The SRF was obtained using the method described by Lo et al. [28]. The viscosity of sludge was measured by a viscosity analyzer (NDJ-5S, Changji, China).

Chemical oxygen demand (COD) was measured in accordance with standard methods [29]. Due to the strong interference of H_2O_2 on COD measurement [30], manganese dioxide (MnO_2) was used to quench the residual H_2O_2 in the sludge samples. The total organic carbon (TOC) of samples' filtrate was determined using a TOC-V_{CPH} analyzer (Shimadzu, Japan). Sludge particle size distributions within sludge samples were examined by a laser particle size analyzer (Eye Tech, Ankersmid, Netherlands). The DD_{SCOD} was defined as the ratio of soluble chemical oxygen demand (SCOD) increment by US/Fenton treatment to the maximum possible SCOD increment [31], which could be calculated as follows:

$$\text{DD}_{\text{SCOD}} (\%) = \frac{\text{SCOD} - \text{SCOD}_0}{\text{TCOD} - \text{SCOD}_0} \times 100 \quad (1)$$

where TCOD is the total COD of the untreated sludge, SCOD and SCOD_0 values are the soluble COD of the treated and untreated sludge samples, respectively. COD of samples' filtrate after 0.45 μm membrane was referred as SCOD. The EPS concentration of the sludge supernatant was analyzed for proteins and polysaccharides, which were determined spectrophotometrically using a

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