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Impact of microwave treatment on dewaterability of sludge during Fenton oxidation

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ABSTRACT

The impact of microwave treatment on the dewaterability of sludge during Fenton oxidation treatment was studied. It was found that MW/Fenton could significantly improve the dewaterability of sludge and reduce reaction time. In this study, the effect of Fenton reagent influence factors, such as initial pH value, reaction time and concentration of H_2O_2 and Fe^{2+}, was investigated to obtain the optimal operating conditions (pH 3, $Fe^{2+} = 0.4$ g L^{-1}, $H_2O_2 = 6.0$ g L^{-1}, $t = 2.0$ min and $p = 648$ W). In addition, the effect of temperature was also studied. Under the optimal operating conditions, the specific resistance to filtration (SRF) and moisture contents was 1.85×10^9 s^2 g^{-1} and 65.93%, respectively. Then the micro-morphological characteristics of sludge under the optimal condition were studied to further explore the responsible mechanisms of this advanced method on sludge dewatering. The results of particle size distribution of raw sludge and conditioned sludge confirmed MW/Fenton could effectively improve the dewaterability of sludge.

Keywords: Dewaterability; Microwave–Fenton pretreatment; Micro-morphological characteristics; Specific resistance to filtration; The moisture contents

1. Introduction

Over the past two decades, activated sludge has become a very popular method to treat municipal wastewater around the world [1,2]. However, the increasing number of wastewater treatment plants produces large amounts of waste, primary and secondary sewage sludge, which contains high moisture contents (95%–99%) [1]. And the nature of high water content usually causes high cost of transportation, and enhanced disposal and technical difficulties in the disposal of sludge [3]. Due to the strong hydrophobicity, sludge is usually regarded as "difficult to dewater" [4]. Besides, to protect the environment, the disposal regulations for sewage sludge become more and more stringent around the world [1,5]. Therefore, an efficient method to improve dewaterability of sludge has

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been one of the major concerns in wastewater treatment processes [6].

In an effort to improve the dewaterability of sludge, a number of methods for reducing the sludge water content have been reported. Highly efficient dewatering technologies include ultrasonication [7], microwave irradiation [6,8–10], alkaline pretreatment [11], ozone oxidation [12], supercritical and subcritical water oxidation and high-temperature hydrolysis [13]. In general, all these sludge reduction technologies are working with the same principle of the disintegration of cell walls and large organic molecules [2]. Amongst these technologies, microwave (MW) irradiation is an efficient and novel sludge pretreatment technology that enhances biodegradability, methane production and digester performance [6,9]. In fact, the mechanism of microwave treatment is a heat treatment. A uniform microwave field generates energy through the realignment of dipoles with oscillating electric fields to generate heat from the inside to the surface of the treated material [6]. So it is a novel pretreatment method for the stabilization of waste activated sludge (WAS) [2]. As we all know, sludge is a multiphase medium containing water, mineral and organic substances, proteins and cells of micro-organisms [6]. During the microwave treatment, sewage sludge absorbs microwave irradiation, and then the kinetic energy of microwave irradiation causes water dipole to reach boiling point value, at last the microbial cells rupture and the bound water are released [6]. Meanwhile, the characteristics of protein that exists on the surface of sludge are also changed by the microwave irradiation, such as its solubility becomes low in water. Hence, compared with digestion of sludge pretreated through conventional heating and untreated sludge, microwave pretreatment of sludge reduces sludge viscosity, improves dewaterability and pathogen decay. Techniques such as microwave drying, pasteurization sterilization, pyrolyzing, gasifying, microwave-hydrothermal synthesis, and organic compound decomposition have been investigated [14–16].

In the past two decades, due to its ease of implementation and negligible environmental impact, Fenton pretreatment, an advanced oxidation processes (AOPs), has also attracted great attention as an alternative method for sludge conditioning [7,17–19]. Fenton pretreatment can use ferrous ions to react with hydrogen peroxide, and produce hydroxyl radicals that have a powerful oxidizing ability to degrade extracellular polymeric substances (EPS) and disrupt sludge flocs [18]. As a consequence, the settleability and dewaterability of sludge are both improved [1,18].

There are numerous studies on the benefits of different pretreatment techniques, including Fenton oxidation and microwave pretreatment, when the methods are applied independently and in combination with other pretreatment options [2,19]. However, to our knowledge, very few works have been reported on the combination between microwave and Fenton oxidation pretreatment (MW/Fenton) of sludge to enhance dewaterability of sludge. Therefore, the aim of this present work was to investigate the effect of two promising pretreatment methods (Fenton oxidation and combined microwave–Fenton oxidation pretreatment) on sludge dewatering.

2. Materials and methods

2.1. Experimental materials

The WAS used in this investigation was taken from the outlet of secondary sedimentation tank of the Sand Lakes Sewage Treatment plant in Wuhan, China. The collected sludge was immediately transferred to the laboratory and stored at 4℃ in the refrigerator before being used. The characteristics of the collected digested sludge are shown in Table 1. $FeSO_4·7H_2O$ used as a catalyst was bought from Bodi Chemical Co. Ltd. of Tianjin. Hydrogen peroxide (30 wt.%) was purchased from Sinopharm Chemical Reagents Co. (Shanghai, China) and directly utilized for Fenton reaction in the study. Dilute sulphuric acid and sodium hydroxide were used for adjusting the pH of the sludge samples during these experiments.

2.2. Experimental procedures

To compare the difference of Fenton treatment, microwave treatment and MW/Fenton treatment, 100 mL sludge samples were placed into 250 mL flasks. The pH of sludge was adjusted with dilute sulphuric acid to 3. Then, we added a certain quantity of $FeSO_4·7H_2O$ into the mixtures to final concentrations of 0.4 g/L, and the mixtures were shaken well. Two minutes later, a known amount of hydrogen peroxide was injected into the flasks to final concentrations of 3 g/L, and the time was started at the same time. The solutions were mixed immediately, and put into the microwave oven to heat for 30 min. Meanwhile, 100 mL sludge samples with pH 3 were added into 250 mL flask, and then immediately placed into the microwave oven to heat for 30 min under the same condition. The moisture contents and SRF of the three differently treated sludges were measured. Microwave oven (Galanz WD900G, China) with a power of 648 W was used in this study.

Table 1
Characteristics of sewage sludge used in this study

Water contents (%)	pH	SV30 (%)	VSS (g L^{-1})	SVJ (mL g^{-1})	TSS (g L^{-1})
98.18	6.5	70	11.87	45.66	16.64

2.3. The influence of the initial pH

The influence of pH on the dewaterability of the sludge system was studied. The sludge pH was adjusted with dilute sulphuric acid and sodium hydroxide to 7.0, 6.5 (raw sludge), 5.0, 4.0, 3.0 and 2.0. Next, a certain quantity of $FeSO_4{\cdot}7H_2O$ was added into flasks to final concentrations of 0.4 g/L, and the mixtures were fast shaken well. Two minutes later, a known amount of hydrogen peroxide was injected into the flasks to final concentrations of 3 g/L. The solutions were mixed immediately and put into the microwave oven to heat, and then the moisture contents and SRF of the sludge were measured after MW /Fenton at room temperature for 2 min.

2.4. The influence of the concentration of H_2O_2 and Fe^{2+}

The impact of Fe^{2+} on the dewatering of sludge was studied. Based on above experiments, 100 mL sludge samples were put into 250 mL flasks with 3.0 H_2O_2 g L^{-1} and different quantity Fe^{2+} (0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 Fe^{2+}g L^{-1}) at pH 3 under the same condition to study the influence of the concentration of Fe^{2+}. Then the influence of the concentration of H_2O_2 was investigated with the optimal quantity of Fe^{2+} and different quantity H_2O_2 (2.0, 3.0, 4.0, 5.0, 6.0, 7.0 and 8.0 H_2O_2 g L^{-1}).

2.5. Influence of temperature on sludge dewatering

Microwave treatment could cause a strong heat effect which results in the increase in temperature. Sludge temperature is a vital parameter that has a significant influence on physical and chemical characteristics of sludge. Hence, to investigate the effects of temperature on sludge dewatering, the sludge temperature at different time (0, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 min) and the corresponding moisture contents and SRF of sludge were measured.

2.6. Analytical methods

The dewatering properties of the digested sludge were studied by determining the specific resistance to filtration (SRF) and moisture contents.

SRF was performed according to the method of Christensen and Dick (1985) with a vacuum of 0.05 MPa using Whatman No. 3 filter paper. The SRF measuring device includes metre pipe, Buchner funnel, filter flask, pressure gauge and vacuum pump. According to the filtration time, the volume of filter liquid and the moisture of the sludge were used for calculation SRF of the sludge [20].

$$r = \frac{2bpA^2}{\mu C} \tag{1}$$

where r (m/kg): SRF, b: the slope of the line, p (N/m^2): the pressure of filtration, A (m^2): the area of filtration, μ (kg s/m^2): the viscosity of the sludge, and C (kg/m^3): the dry weight of percolate.

The moisture content was performed according to the method of national standard.

$$c = \frac{W_0 - W}{100W_0}\% \tag{2}$$

where c: the moisture content, W_0 (g): the weight of the wet sludge, W (g): the weight after drying in 160˚C oven for 2 h.

The micro-morphological characteristics of sludge samples were observed via an electron microscope (Olympus Microscope, CX31, Japan) and Laser particle size analyzer (MS-2000, British).

Up to four replicates of each experimental condition were performed with the samples analysed in triplicate, and average values and standard deviations were obtained.

3. Results and discussion

3.1. Treatment effect comparison amongst microwave (MW), Fenton and MW/Fenton reaction system

The moisture contents and SRF of sludge treated by MW/Fenton, microwave and Fenton oxidation are shown in Fig. 1. There were significant differences for the moisture contents and SRF of the sludge conditioned by the three methods. It was observed that the moisture contents and SRF of sludge conditioned by Fenton oxidation decreased slightly with the increase

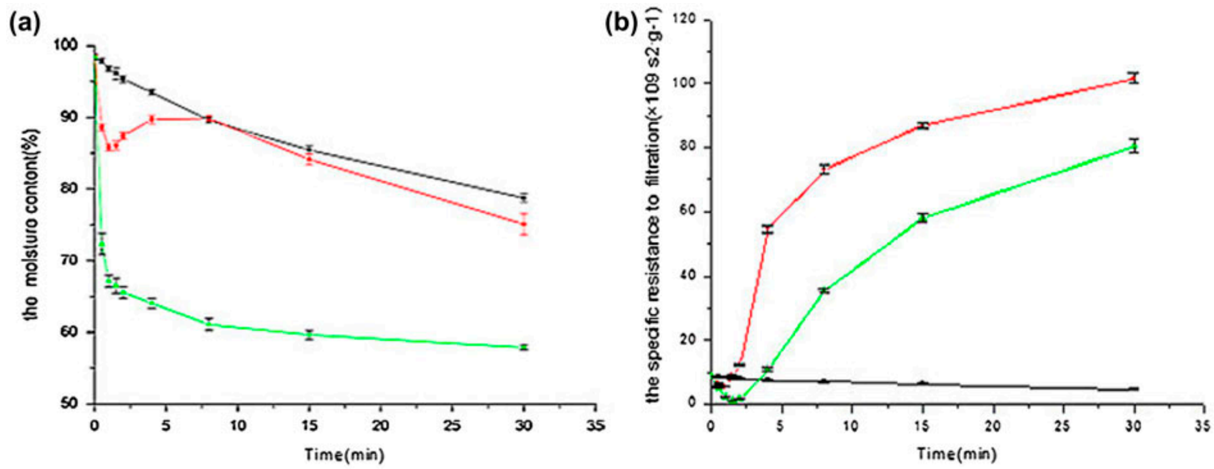

Fig. 1. Effect of various treatment techniques on the SRF and the moisture contents. (a) Effect of various treatment techniques on the SRF and (b) effect of various treatment techniques on the moisture contents.

in reaction time. The phenomenon might be attributed to the following fact: because of the generation of high concentration hydroxyl radicals, the extracellular polymers EPS were decomposed, the cells were broken and the soluble organic matters such as protein and polysaccharide were released [1,2]. However, the change of the moisture contents and SRF of sludge conditioned by MW/Fenton and microwave were similar. The moisture contents and SRF decreased when the reaction time increased from 0 to 2.0 min. The moisture contents and SRF reached their minimum at around 2.0 min, and then increased sharply as reaction time increased. This phenomenon could be explained as follows: at the beginning, the rapid internal heating destroyed the cell walls of micro-organisms and disintegrated organics, which was helpful for sludge dewatering [21]. But a long time of microwave would result in the excessive damage for cell of micro-organisms, at last great quantities of the cell material were released, and the sludge particles became very tiny. So sludge dewatering was reduced greatly. However, the moisture contents and SRF of sludge conditioned by MW/Fenton were smaller than that of the sludge conditioned by microwave. The findings might be attributed to the excitation of the molecule to high vibrational and rotational energy levels; the hydrogen peroxide in solution irradiated by microwave quickly generated the hydroxyl radicals [22]. Fig. 1 also clearly indicates that compared with Fenton treatment, the time for microwave and MW/Fenton method to achieve the same moisture contents and SRF was greatly reduced. This indicates that during the MW/Fenton, the hydroxyl radicals generated by Fenton reagent and the thermal effect of microwave both played an important role. Meanwhile, the SFR and moisture contents decreased, while the reaction time increased from 0 to 1.5 min, and then increased greatly. The reasons for this phenomenon were the following: at the beginning of the MW/Fenton reaction process, the thermal effect produced by the microwave could promote the generation of hydroxyl radical and the degradation of organic matter. As the reaction continues, the rapid increase in temperature caused by the microwave irradiation resulted in the decomposition of H_2O_2, which decreased the concentration of H_2O_2 [6,8,9]. Therefore, microwave and Fenton had a synergy to improve the dewaterability of sludge.

3.2. *Influence of Fenton oxidation and WM/Fenton on sludge dewaterability*

3.2.1. *Effect of pH on sludge dewatering*

Fig. 2(a) shows the effect of pH on sludge dewatering. The SFR decreased as the pH increasing from 2.0 to 3.0, and reached its minimum at around pH 3.0, and then increased greatly as the pH rose from 3.0 to 7.0. The change of moisture contents was similar with that of the SFR. The only difference was that the moisture contents almost kept constant as the pH rose from 2.0 to 3.0.

Fe^{2+} and Fe^{3+} were immobilized in the form of $Fe(OH)_2$ and $Fe(OH)_3$ when the concentration of ·OH increased, whereas high concentrations of H^+ impeded the formation of $FeOOH^{2+}$ and lowered the concentrations of Fe^{2+} and ·OH, which decreased the efficiency of Fenton oxidation [17]. Comprehensive

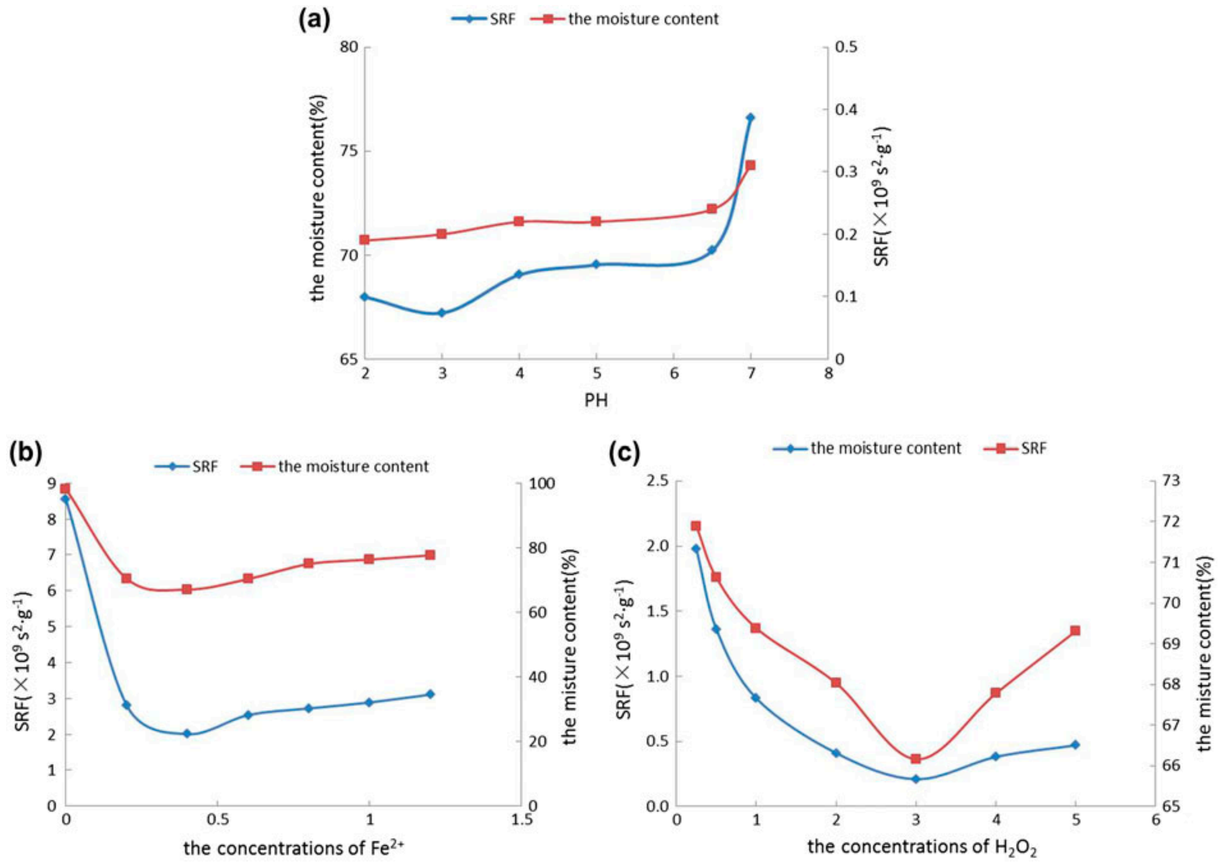

Fig. 2. The effects of Fenton reagent influence factors during WM/Fenton. (a) The influence of the initial pH on the moisture contents and SRF; (b) the influence of the concentration of Fe^{2+} on the moisture contents and SRF and (c) the influence of the concentration of H_2O_2 and SRF.

consideration, the optimum pH value in the present study was determined to be 3.0, where SRF and moisture content almost at the same time reached their minimum.

3.2.2. Impact of Fe^{2+} on sludge dewatering

As shown in Fig. 2(b), when the concentration of Fe^{2+} was below 0.4 Fe^{2+}g L^{-1}, sharp fallings in moisture content and SRF for MW/Fenton treatment was both observed. And then the moisture content remained stable even if the amount of Fe^{2+} was increased. But SRF rose markedly when the quantity of Fe^{2+} increased from 0.4 to 1.2 Fe^{2+}g L^{-1} for MW/Fenton treatment. The moisture content and SRF reached their minimum when the concentration of Fe^{2+} was around 0.4 Fe^{2+}g L^{-1}. Fe^{2+} is the catalyst of the Fenton reaction that promotes the production of ·OH. The generation of ·OH radicals was insufficient under the low concentration of Fe^{2+}, so the dewatering efficiency was not good. Besides, the organic substances, microbes and inorganic materials in the sludge could inhibit the recycling process in Fenton oxidation. Thus, sufficient Fe^{2+} was required for the reaction [15]. When the concentration of Fe^{2+} was higher than the optimal concentration, the excessive Fe^{2+} had a scavenging effect on hydroxyl radicals (Eq. (3)) [9], thus the concentration of ·OH radicals was smaller.

$$Fe^{2+} + {}^{\cdot}OH \rightarrow OH^{-} + Fe^{3+} \quad (3)$$

3.2.3. Effect of H_2O_2 on sludge dewatering

Fig. 2(c) shows that the moisture content for MW/Fenton oxidation decreased when the concentration of H_2O_2 increased from 0 to 6.0 H_2O_2 g L^{-1}. And then the moisture content increased slightly with the increase in the concentration of H_2O_2. The moisture content reached its minimum when the concentration of H_2O_2 was around 6.0 H_2O_2 g L^{-1}, which was far smaller than that of the raw sludge. The change of

SRF was similar with that of moisture content, and its minimum was reached at around 6.0 H_2O_2 g L^{-1} for MW/Fenton treatment. It was evident that the overloading of H_2O_2 did not mean a further enhancement in dewatering efficiency.

$^{\cdot}$OH radicals generated by H_2O_2 with strong oxidation ability could consume activated sludge and degrade the extracellular polymers EPS which almost occupies 80% of the amount of activated sludge. The generation of $^{\cdot}$OH radicals was insufficient under the low concentration of H_2O_2, which inhibited the dewatering efficiency. Besides, thermal effect generated by microwave during the MW/Fenton reaction would accelerate the volatilization of $^{\cdot}$OH radicals. Thus, sufficient H_2O_2 was required for the reaction [15]. If the concentration of H_2O_2 was higher than the optimal concentration, the excessive H_2O_2 had a scavenging effect on hydroxyl radicals (Eq. (4)), so the concentration of $^{\cdot}$OH radicals was smaller.

$$H_2O_2 + {}^{\cdot}OH \rightarrow {}^{\cdot}HO_2 + H_2O \quad (4)$$

3.2.4. Influence of temperature on sludge dewatering

As shown in Fig. 3(a), the sludge temperature quickly increased until the temperature was close to the boiling point. When the contact time was further elongated, the water in the sludge evaporated quickly, and the sludge temperature increased as the water was removed. The reason for this phenomenon was that the interaction between high frequency electromagnetic radiation and the dipolar molecules (such as water, proteins and some liquids) caused rapid rise on the sludge temperature by the resultant molecular rotation, and leaded to the sludge temperature to reach the boiling point during irradiation. Due to the carbonization of humic substances in organic matter-rich sludge by a high temperature, it was insignificant for the further application of sludge with high temperature. In other words, higher temperature meant more energy or longer contact time used, which was uneconomical for sludge dewatering. So the moisture content and SRF of sludge were measured in the first 3 mins to study the effect of temperature on sludge dewatering. As shown in Fig. 3(b) and (c), the moisture content and SRF of sludge sharply decreased with the increase in contact time during the initial 2 min. This could be attributed to the rapid increase in temperature at short contact time, which resulted in sludge flocs breaking into smaller fragments [23]. However, when the contact time was increased further, the SRF significantly increased, while the moisture content almost kept constant. This might result from the complete disruption of the floc structure by excessive microwave irradiation and the release of intracellular and extracellular materials. The process also produced many fine particles, which was not beneficial for sludge conditioning [9].

3.3. Microstructure of sludge

3.3.1. The change and distribution of particle size

To further explore the responsible mechanisms of this advancement in sludge dewatering, the measurements of the change and distribution of sludge floc sizes of raw sludge and the sludge conditioned by MW/Fenton were shown in Table 1 and Fig. 4, respectively.

Different size distributions were obtained for raw sludge and sludge conditioned by MW/Fenton. According to the distributions shown in Table 2, compared with that of the raw sludge, the distribution of particles of sludge conditioned by MW/Fenton appeared to have a big difference. After MW/Fenton pretreatment, surface-weighted mean and volume-weighted mean were both reduced by 40.7 and 39.2%, respectively, while the specific surface area increased by 69.1%. Besides, the d (0.1), d (0.5) and d (0.9) values of sludge treated by MW/Fenton indicated that 10, 50 and 90% of the particles measured were almost a time smaller than that of raw sludge. As shown in Fig. 4, although the both sludge samples had similar particle size distribution patterns, it was evident that the particle size of sludge treated by MW/Fenton decreased. For the raw sludge, the proportion of the volume fraction of particles between 104.713 and 120.226 μm was the maximum (7.0%). But for sludge treated by MW/Fenton, the maximum proportion of the volume fraction of particles was between 69.183 and 79.433 μm. This was expected to be linked to the destruction of the sludge and oxidation of extracellular which caused floc particles break into small particles, and the average particle size and particle size both decreased [3].

3.4. Microstructure of sludge

Microscopic examination in this study suggested that MW/Fenton treatment could effectively damage the EPS biopolymers that surrounded the sludge micro-floc aggregates, which eventually weakened the attachment between cells and disrupted the structure of the sludge flocs (Fig. 5). As shown in Fig. 5(a), the microscopic structure of raw sludge was intact, well formed, and connected. The sludge became loose and thin after being conditioned by MW/Fenton treatment (Fig. 5(b)). It was clearly shown that the flocs surface

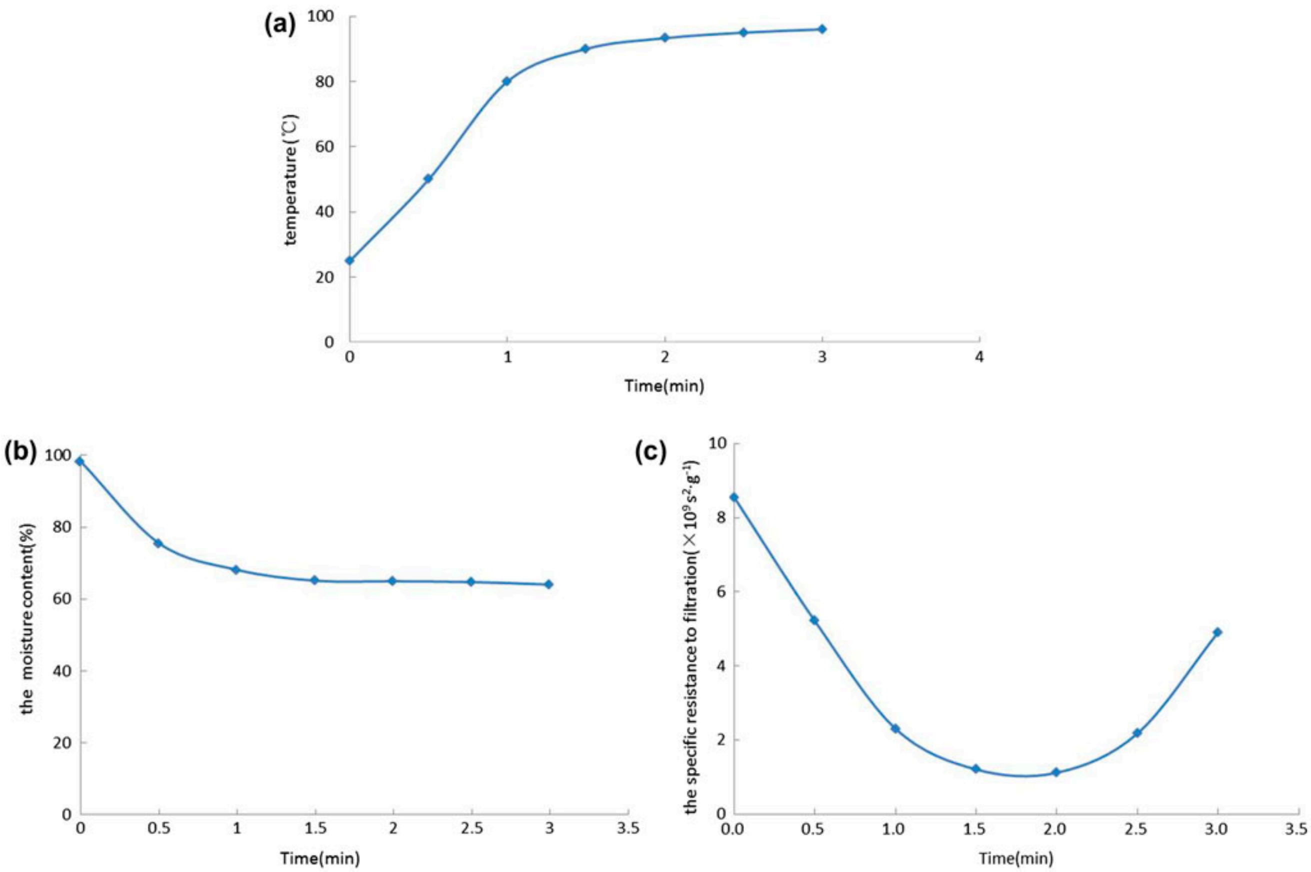

Fig. 3. The effects of temperature on sludge dewatering. (a) Effect of MV irradiation to temperature rise; (b) the effects of temperature on the moisture contents and (c) the effects of temperature on t the SRF.

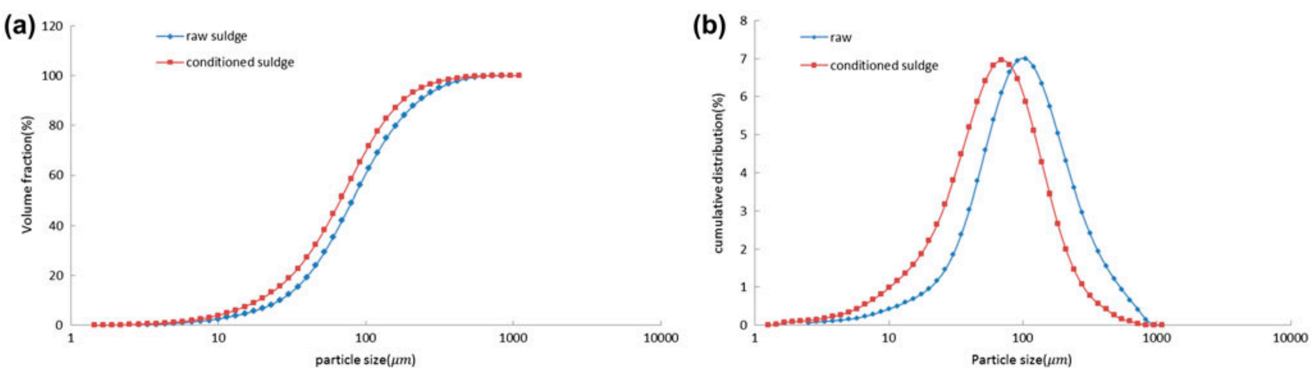

Fig. 4. The particles size distribution of the raw sludge and the conditioned sludge. (a) The volume distribution of the raw sludge and the conditioned sludge and (b) the cumulative distribution of the raw sludge and the conditioned sludge.

was wrapped by a thick EPS layer, which led to steric stabilization [24] and poor performance in sludge dewatering. After being conditioned by MW/Fenton treatment, EPS were degraded and damaged rapidly, which caused the destruction of the sludge and the exposure of partial cells. Therefore, the floc size became small. The destruction of the sludge could provide more passages for the release of water, so it was beneficial to improve the dewaterability of sludge. Furthermore, due to the thermal effect produced by

Table 2
Particle size distribution of different digested sludge samples

Sludge samples	d (0.1)/μm	d (0.5)/μm	d (0.9)/μm	Specific surface area ($m^2 g^{-1}$)	Surface-weighted mean (μm)	Volume-weighted mean (μm)
Raw sludge	34.382	106.822	304.639	0.0952	63.005	145.501
Treated sludge	18.687	67.092	178.039	0.161	37.381	88.442

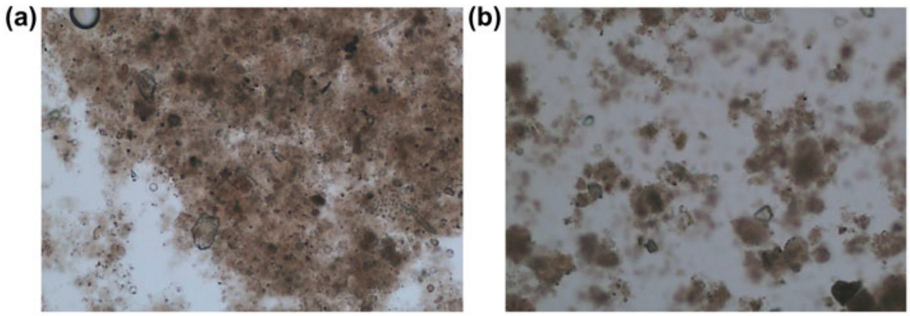

Fig. 5. The microstructure of sludge flocs. (a) The microstructure of the raw sludge flocs and (b) the microstructure of the conditioned by WM/Fenton sludge flocs.

the microwave and the generation of hydroxyl radical, cell membranes were cracked, which further resulted in the release of bound water and intracellular large molecular organics.

4. Conclusion

The effect of MW/Fenton treatment on sludge dewatering was studied. The moisture contents and SRF of the sludge conditioned by MW/Fenton were much lower than that conditioned by Fenton oxidation or microwave at the same conditions. It clearly indicated that microwave promoted the production of more ·OH, thus the dewaterability of sludge was improved. The optimum conditions for MW/Fenton treatment were found to be 2 min of microwave treatment with H_2O_2 6.0 g L^{-1}, Fe^{2+} 0.4 g L^{-1} and initial pH 3.0 at room temperature, at which the SRF and the moisture contents was 1.85×10^9 $s^2 g^{-1}$ and 65.93%, respectively. Besides, particle size distribution of raw sludge and conditioned sludge further confirmed MW/Fenton could effectively degrade and damage EPS, caused the break-up of sludge matrix and micro-organism cells lysis, which enhanced the dewaterability of WAS. Therefore, it was reasonable to draw a conclusion that MW/Fenton treatment was a novel and useful method to improve the dewaterability of WAS. However, more work should be undertaken to further determine the mechanisms underpinning the enhanced sludge dewatering using MW/Fenton.

Acknowledgements

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