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Influence of microwave irradiation on sludge dewaterability

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ABSTRACT

The study focuses on the effect of microwave irradiation on sludge dewaterability. Capillary suction time and specific resistance of filtration were used to evaluate sludge dewaterability. Soluble chemical oxygen demand, extracellular polymeric substance (EPS) content and sludge particle size were determined in an attempt to explain the observed changes in sludge dewaterability. The results indicated that microwave application slightly enhanced sludge dewaterability with short contact time, while it significantly deteriorated sludge dewaterability with long contact time. The results show that 900 W and 60 s is an ideal microwave condition, yielding maximum sludge dewaterability characteristics by generating sludge with optimal disintegration (1.5-2%), EPS concentration (1500-2000 mg/l), and particle size distribution ($120-140 \mu m$).

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1. Introduction

Recent years have seen significant increase in the amount of waste activated sludge production in municipal wastewater treatment plants. Sludge dewatering is one of the fundamental steps in sludge processing because it reduces sludge volume and, consequently, the cost of transporting sludge to its ultimate disposal site. However, the high water content and biological gel structure properties of sludge lead to difficulty in dewatering. Therefore, suitable sludge conditioning processes should be chosen prior to dewatering.

The addition of polyelectrolyte to the sludge, which is presently the most widely used pretreatment in wastewater treatment plants, increases costs and may cause secondary environmental pollution [1]. Thus, various alternative methods have been proposed to improve sludge dewaterability, e.g., the addition of acids and surfactants [2], Fenton's reagent pretreatment [3], fungal treatment [1], ultrasonication [4], and microwave irradiation [5]. Among these methods, microwave irradiation is considered to be an efficient tool for improving sludge dewaterability because of its "thermal" and "athermal" effects [6,7]. Also, some literatures gave examples of the use of microwave radiation for the rapid processing of materials with novel morphologies and properties, which would take much more time via conventional thermal energy transfer methods [8–10].

Sludge is a multiphase medium, containing water, mineral and organic substances, proteins, and microorganism cells [6].

High frequency electromagnetic radiation interacts with the dipolar molecules (such as water, proteins, and some liquids), which initiates a rapid heating from the resultant molecular rotation. Likewise, permanent or induced dipoles in the dispersed phase cause rapid heating of the particles [11,12]. This rapidly raises sludge temperature during microwave irradiation. Another result is that cell structure is destroyed and bound water is released [12]. Wojciechowska [6] observed that microwaving improved sludge dewaterability and the effects depended on the sludge type. He also found that after 180s of microwave heating, specific resistance to filtration (SRF) of mixed sludge (primary and secondary sludge) and anaerobically digested sludge decreased to 73% and 84%, respectively. Seehra et al. [13] found that the overall characteristics of microwave dewatering represented significant advantages over dewatering by thermal heating. And Chan et al. [14] reported that a process combining microwave, hydrogen peroxide, and acid hydrolvsis could recover sludge nutrients. Additionally, using microwave irradiation has a sterilization effect that is beneficial for further utilization [12]. Moreover, microwave irradiation associated with hydrothermal systems has been employed recently for the growth of particles having different structural and morphological characteristics [15-17].

Whether microwave irradiation can in fact improve sludge dewaterability requires further study. In addition, it is necessary to establish optimal dewatering conditions. This study is carried out to examine sludge disintegration, particle size, and extracellular polymeric substance (EPS) content, and to analyze how sludge dewaterability is influenced by microwave irradiation. This work may provide some insights into the changes of sludge properties. The mechanism behind the observed changes in sludge dewaterability is also discussed.

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Table 1

Characteristics of the initial sludge sample (mean \pm S.E.).

Parameter	Value
рН	6.68
Soluble chemical oxygen demand (mg/l)	85 ± 1.50
Water content (%)	98.56 ± 0.01
Protein (mg/l)	585.25 ± 25.52
Polysaccharide (mg/l)	97.7 ± 2.80
Capillary suction time (s)	92.50 ± 2.20
Specific resistance of filtration (m/kg)	$5.37 \times 10^9 \pm 2.67 \times 10^8$

2. Materials and methods

2.1. Sludge samples

In this study, secondary sludge was obtained from LiJiao Municipal Wastewater Treatment Plant (Guangzhou, China) and was stored at 4°C prior to use. The wastewater treatment process at the plant is anaerobic–anoxic–oxic. Sludge characteristics are presented in Table 1.

2.2. Experimental equipment and methods

The schematic of the experimental apparatus is given in Fig. 1. A modified household microwave oven (GALANZ Co., frequency 2.45 GHz, max power 1000 W) was used to generate microwave irradiation. A column reactor, a 500 ml-size beaker with an exposed surface area of 59.42 cm², was installed in the middle of the microwave oven. It is constructed from low energy-loss and heatresistant (up to 700 °C) amber boron-silica glass. A sheltered type-K thermocouple was installed on top of the microwave oven. This was used to record the sludge temperature profiles during microwave irradiation. A central control room, which contained the electronic temperature meter, calculagraph, and microwave switch, was installed on the right side of microwave oven. The electronic temperature meter was used to display the real-time temperature value, which was recorded from the thermocouple. The calculagraph displayed the operating time of the microwave, which was designated as the "microwave contact time". The microwave switch was used to adjust the microwave energy and turn the microwave on/off. The sludge samples (400 ml) were put into the reactor and treated with an array of 500 W. 750 W. and 900 W for various durations, e.g., 0 s, 20 s, 40 s, ..., 140 s. They were then cooled to room temperature before analysis.

After the sludge samples were exposed to microwave irradiation and centrifuged at 4000 rpm for 30 min at 25 °C (Universal 32 R centrifuge, Hettich, Germany), soluble chemical oxygen demand (SCOD) of supernatant samples was measured. Additionally, the sludge dewaterability was assessed in terms of capillary suction time (CST) and SRF. The EPS concentrations and particle sizes of the sludge samples were measured to investigate the mechanisms



Fig. 1. Schematic diagram of the experimental apparatus: 1, microwave oven; 2, control room; 3, microwave generator; 4, column reactor; 5, thermocouple probe; 6, electronic temperature meter; 7, calculagraph; 8, microwave switch.

behind the observed changes in sludge dewaterability. Each experiment was performed in triplicate, and average values and standard deviations were obtained. Quadratic regressions of experimental data were calculated using SigmaPlot software (v 10.0).

2.3. Analytical procedures

Water content and chemical oxygen demand (COD) were measured in accordance with standard methods [18]. Sludge particle size distributions were examined with a laser particle size analyzer (LS-pop III, OMEC, China). Particle size results were expressed in terms of its dp90, which is defined as the 90th percentile (by volume) of the particles' diameters. Sludge disintegration degree (DD_{SCOD}) was defined by Li et al. [19], comparing the microwave process and the maximum soluble chemical demand (SCOD_{NaOH}).

$$DD_{SCOD}(\%) = \frac{SCOD_S - SCOD_{S0}}{SCOD_{NaOH} - SCOD_{S0}} \times 100$$
(1)

 $SCOD_S$ and $SCOD_{S0}$ values are for treated and untreated sludge samples, respectively. $SCOD_{NaOH}$ was obtained via an alkaline hydrolysis procedure in which the initial sludge sample was mixed with 0.5 M NaOH at room temperature for 24 h [20].

The CST and SRF were obtained using the method described by Feng et al. [4]. The EPS content of the sludge supernatant was determined spectrophotometrically using a T6 UV/vis spectrophotometer (PGeneral, China). Proteins were determined by the Coomassie Brilliant Blue G-250 method [21], using casein as the standard. Polysaccharides were measured by the anthrone method [22], using glucose as the standard. To elucidate the relationship among sludge disintegration degree, particle size, EPS concentration, and dewaterability, we only analyze the observed changes of above parameters under the 900 W microwave condition.

3. Results and discussions

3.1. Dewatering characteristics of sludge with microwave irradiation

CST indicates how quickly sludge releases its water, and is measured to evaluate sludge dewatering behavior. The sludge CST was initially 92.50 s, and substantially decreased with increasing contact time. The lowest CST values, which were on average 53.00 s, 59.22 s and 62.44 s, were obtained for the contact time of 60 s, 100 s, and 120 s at the microwave energy of 900 W, 750 W, and 500 W, respectively. However, when the contact time was extended further, the CST values rapidly increased and even exceeded that of unconditioned sludge (Fig. 2). The SRF values decreased from 5.37×10^9 m/kg for the untreated sludge to 1.93×10^9 m/kg, 1.84×10^9 m/kg, and 1.59×10^9 m/kg at 500 W, 750 W, and 900 W, respectively. When the contact time was increased further, the SRF significantly increased (Fig. 3).

The contact time varies with the microwave energies at the lowest CST and SRF values because the sludge temperature changes due to the thermal effect of microwave irradiation [23]. Another possible reason for this is the athermal effect [12]. In other words, at larger microwave energies, shorter contact time is needed for the irradiation to generate maximum dewaterability.

According to the CST and SRF data, a suitable microwave energy and contact time could improve sludge dewaterability. This may be because sludge flocs, which are repositories for water, are broken into smaller fragments at short contact time. These smaller fragments can be re-flocculated to tighter particles with the help of flocculation agents, improving the sludge dewaterability. The slight increases in the EPS concentration may also explain the observed enhancement of sludge dewaterability [24]. These results are verified elsewhere [6]. Similar effects of ultrasound treatment have



Fig. 2. Effect of microwave energy and contact time on capillary suction time.

also been observed [2,4,25]. When the contact time is longer than a certain value (which varies with microwave energy), the sludge dewaterability worsens. This may result from the complete disruption of the floc structure by excessive microwave irradiation and the release of intracellular and extracellular materials. The process also produces many fine particles, which is not beneficial for sludge conditioning.

Based on the changes observed in the parameters that characterize sludge dewaterability, we conclude that the optimal energy level is at 900 W with an exposure time of 60 s. Increasing the contact time not only consumes more energy but also decreases sludge dewaterability.

3.2. Effect of sludge disintegration on sludge dewaterability

3.2.1. Changes in sludge disintegration following microwave conditioning

The sludge disintegration degree increased with the contact time and microwave energy (Fig. 4). It indicated that SCOD increased gradually and these rates increased with microwave irradiation. This was due to the extent of sludge floc structure disruption by microwave irradiation. At short contact time, the floc structure becomes relatively loose and some filamentous bacteria are exposed during this transition, but the structure change was minimal enough so as not to destroy the microorganisms. When microwave contact time increased above a specific value, the floc



Fig. 3. Effect of microwave energy and contact time on sludge specific resistance of filtration.



Fig. 4. Effect of contact time on sludge disintegration and particle size.

structure was disrupted completely, and extracellular and intracellular biopolymers, such as proteins and sugars, were released from activated sludge flocs into the soluble phase [20,26]. Similar effects of ultrasound treatment and ozonation have been observed [27,28]. Different microwave contact times generated different sludge temperatures. Thus, it could be concluded that temperature was the most influential parameter on sludge disintegration. Li et al. [19] similarly concluded that temperature had a significant influence on sludge disintegration by ultrasonic sludge treatment.

3.2.2. Effect of disintegration degree on CST and SRF

Microwave irradiation can disrupt flocs and destroy microbial cells [12,26]. Increasing irradiation contact time increased both the SCOD of the supernatant as well as the DD_{SCOD} that describes the degree of sludge disintegration. Fig. 5 shows the relationship between the sludge disintegration degree and CST/SRF. The CST and SRF values decreased when the sludge disintegration degree was increased by less than 2%. When the disintegration degree was increased by more than 2%, the CST and SRF values increased sharply. When the CST and SRF values were at a minimum, the sludge disintegration degree was optimum for maximum sludge dewaterability. According to the data in Fig. 5, the optimum value was calculated to be approximately 1.5–2%. As a result, a proper sludge disintegration degree is vital for the sludge dewaterability.

During sludge microwave irradiation, the diffuse sludge flocs were broken into smaller fragments, which improved the sludge dewaterability when these particles flocculated. If the contact time of microwave irradiation was increased to a point where



Fig. 5. Effect of disintegration degree on sludge dewaterability.



Fig. 6. Effect of microwave contact time on EPS concentration in the sludge supernatant.

the sludge disintegration degree was too high, many fine particles were produced and the sludge dewaterability gradually deteriorated. The effect of disintegration degree on sludge dewaterability was also reflected by the correlation between DD_{SCOD} and CST/SRF (R = 0.9136 and R = 0.8934, respectively). These results are in agreement with those of Li et al. [19], who observed that the dewaterability was dependent on the sludge disintegration degree during ultrasonic treatment.

3.3. Effect of EPS on sludge dewaterability

3.3.1. Changes in EPS concentration of the sludge supernatant

Proteins and polysaccharides in the sludge samples were initially estimated at concentrations of 585.21 mg/l and 97.70 mg/l, respectively. After sludge disintegration by microwave irradiation, the EPS and cellular substances were released into the aqueous phase, leading to an increase in protein and polysaccharide levels (Fig. 6). The slope (21.39) of the regression line for proteins versus time of microwave exposure at 900W was higher than that for polysaccharides (11.68), indicating that proteins were released to the aqueous phase more quickly than polysaccharides. For microwave irradiation of 900 W for 140 s, the increases in the levels of proteins and polysaccharides were 297% and 654%, respectively, and the yields of the two substances continued to increase with the contact time. The interactions of the very weak forces binding EPS components together, which are very important to the colloidal stability of flocs [29], are also disrupted during microwave irradiation. Therefore, the higher microwave contact time could not only break the flocs and release extracellular materials but also completely destroy cells and release intercellular materials from cells into the aqueous phase. The effect of microwave contact time on the EPS concentration was also reflected by the correlation between contact time and proteins/polysaccharides concentration (R = 0.9938and R = 0.9729, respectively).

3.3.2. Effect of EPS on CST and SRF

EPS, presumed to be the predominant protein and polysaccharide, is regarded as one of the most important factors that influence the dewaterability characteristics of wastewater sludge [30,31]. When the sludge is exposed to microwave irradiation, large quantities of EPS are released into the aqueous phase (Fig. 6). The high EPS concentration not only increases the sludge viscosity but also decreases its dewaterability [2,32]. The relationship between EPS concentration and sludge dewaterability, however, is not always



Fig. 7. Effect of supernatant EPS concentration on sludge dewaterability.

directly proportional. This means that increasing the EPS concentration does not always decreased the sludge dewaterability.

Fig. 7 shows that the CST and SRF values decreased with an increasing EPS concentration less than 2000 mg/l. When the EPS concentration is further increased, the CST and SRF values increased significantly. The lowest CST and SRF values correspond to the optimum value of the EPS concentration for maximum sludge dewaterability. Fig. 7 shows that the optimal value of EPS concentration ranged from 1500 mg/l to 2000 mg/l, which is much higher than that of untreated sludge. The above results are in agreement with other work [33], and similar effects have been observed for ultrasound treatment [4]. Increasing concentrations of EPS in the sludge are initially likely to result in a high extent of flocculation [34], which would improve dewaterability characteristics. When the optimal flocculation and deflocculation balance is reached, further increases in EPS concentration would worsen sludge dewaterability.

In this study, the correlations between the EPS concentration and the CST/SRF were 0.8594 and 0.9360, respectively. These results are in consensus with those of Feng et al. [4], who observed the correlations between EPS and the CST/SRF (R = 0.9576 and R = 0.8314, respectively), and also with those of Wang et al. [32], who observed correlation between EPS and CST (R = 0.9223). The variation in the correlation coefficient values may be attributed to the difference of sludge types. This finding agrees with those of Poxon and Darby [30], which showed that the relationship between EPS and sludge dewaterability was dependent on digester feed composition, or that original sludge composition caused differences in this relationship.

3.4. Effect of particle size on sludge dewaterability

3.4.1. Changes in sludge particle following microwave conditioning

The particle size distribution was measured in terms of cut-off diameters dp90 in sludge that was subject to different energies and contact times (Fig. 4). The dp90 of untreated sludge was 83.67 μ m, and its mean diameter was 42.58 μ m. The maximum dp90 was 143.41 μ m at the microwave energy of 900 W and exposure time of 60 s. When the contact time increased, the dp90 decreased. This is because fresh sludge consists of hydrophilic colloidal particles that are in equilibrium with the electrostatic repulsive-force interaction in nature. When sludge is exposed to a certain microwave energy level for a suitable contact time, the high frequency electric fields make the sludge and water inside it move and rotate [35]. During this movement, the Zeta-potential and the structure of surface electric double layers are destroyed. These movements coarsen the sludge particles by destabilization and flocculation. However, when



Fig. 8. Effect of particle size on sludge dewaterability.

microwave irradiation is excessive, the sludge particle size become smaller due to the disruption of the sludge's floc structure and the complete breaking of the sludge cell.

The number of ultra colloidal particles (defined as having a diameter between 1 μ m and 100 μ m) is an important factor that affects the sludge filtration characteristics [36]. A smaller proportion of ultra colloid improves the sludge dewatering. Suitable microwave irradiation reduced the amount of ultra colloid in sludge and enhanced sludge dewaterability. In this study, the ultra colloid proportion fell from 100% to 66.56% at the inflection points.

3.4.2. Effect of sludge particle size on CST and SRF

The effect of the particle size distribution on CST and SRF is presented in Fig. 8. The values of CST and SRF decreased with increasing dp90. Initially, the decrease of CST and SRF was moderate, but at larger particle sizes, it dropped quickly. This indicates that larger particle sizes reduce the CST and SRF values and improve dewaterability. Taking into consideration the microwave factor and the values of CST and SRF, the optimal dp90 was ranged from 120 μ m to 140 μ m. Particles of this size were bigger than the holes in the filter paper and did not clog the pores. Therefore, it was easy for the free water to pass through the filter paper when dewaterability index was measured. This is how a large particle size helps to improve the sludge dewaterability. Nevertheless, the optimal value may change if the sludge sources and moisture contents are different.

From Fig. 8, we also find that there is a strong correlation between the CST and the SRF values and dp90 (R=0.8596 and R=0.9096, respectively). This indicates that the particle size strongly affects the dewaterability. This finding is in agreement with other authors' [4,36]. In addition, Jin et al. [37] and Higgins and Novak [38] have reported that the "supracolloidal" particles in the range of 1–100 μ m had the greatest negative effect on the sludge dewaterability, and that as the concentration of the particles in this size range increased, the dewaterability decreased.

4. Conclusions

This research experimentally determined the effects of microwave irradiation on sludge dewaterability. Sludge was subjected to irradiation at various microwave power levels and exposure times, and the effect of these parameters on sludge dewaterability was observed and investigated. A short contact time slightly enhanced sludge dewaterability, while a long contact time significantly worsened sludge dewaterability. An appropriate microwave energy and contact time was proved to improve sludge dewaterability. The optimal condition based on the CST and SRF results was determined to be 900 W with a contact time of

60 s. Sludge disintegration, EPS concentration and particle size were found to play a vital role in the observed changes in sludge dewaterability. The optimal condition generated sludge disintegration, and the resultant EPS concentration and particle size distribution facilitated sludge dewaterability. It can be concluded that microwave irradiation is a useful tool for enhancing dewaterability. However, the conditioning efficiency of adding polyelectrolytes should be examined in further studies.

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