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Effect of H_2O_2 dosing strategy on sludge pretreatment by microwave-H_2O_2 advanced oxidation process

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ABSTRACT

Considering characteristics of breaking down H_2O_2 into water and molecular oxygen by catalase in waste activated sludge (WAS), the effect of H_2O_2 dosing strategy on sludge pretreatment by the advanced oxidation process (AOP) of microwave-H_2O_2 was investigated by batch experiments for optimizing H_2O_2 dosage. Results showed that the catalase in sludge was active at the low temperature range between 15 °C and 45 °C, and gradually lost activity from 60 °C to 80 °C. Therefore, the H_2O_2 was dosed at 80 °C, to which the waste activated sludge was first heated by the microwave (MW), and then the sludge dosed with H_2O_2 was continuously heated till 100 °C by the microwave. Results at different H_2O_2 dosages showed that the higher the H_2O_2 dosing ratio was, the more the SCOD and total organic carbon (TOC) were released into the supernatant, and the optimum range of H_2O_2/TCOD ratio should be between 0.1 and 1.0. The percentages of consumed H_2O_2 in the AOP of microwave and H_2O_2 treating the WAS were 25.38%, 22.53%, 14.82%, 13.61% and 19.63% at different H_2O_2/TCOD dosing ratios of 0.1, 0.5, 1, 2, 4, respectively. Along with the increasing H_2O_2/TCOD ratio, the contents of TCOD on particles, soluble substances and mineralization increased and the TCOD distribution on solids decreased.

1. Introduction

As the most widely used biological wastewater treatment for both domestic and industrial plants in the world, one of the drawbacks of activated sludge processes is high sludge production. Treatment and disposal of sewage sludge from wastewater treatment plants (WWTPs) accounts for about half, even up to 60%, of the total cost of wastewater treatment [1]. To manage the excess sludge will be one of the most challenging tasks for the wastewater research field in the years to come [2]. Both sludge reduction and sludge reutilization are good strategies for sewage sludge treatment and disposal. The bottleneck of sludge pretreatment is the barrier of cell wall and the membrane composition of complex organic materials that are not readily biodegradable, therefore sludge pretreatment technologies are of great interest in recent researches to enhance anaerobic digestibility of waste activated sludge (WAS) for biogas production [3,4], to improve dewatering of excess sludge for reducing sludge volume [5,6], to recover nutrients (nitrogen and phosphorus) from sludge [7,8], to reduce sludge production [1,9]. Nowadays, among these sludge pretreatment technologies based on physical, chemical and biological methods [1], microwave (MW) is emerging as a promising method for WAS disintegration. Industrial use of microwave heating as an alternative to conventional heating (CH) in chemical reactions is becoming popular, mainly due to dramatic reductions in reaction time [10], i.e., most of previous researches with regard to microwave irradiation have used the thermal effects of galvanic heating. Recently the application of microwave heating in combination with hydrogen peroxide (H_2O_2) for sludge pretreatment has shown to be an efficient advanced oxidation process (AOP) technology [8,10–14]. H_2O_2 is a strong chemical oxidant that destroys the cell walls of micro-organisms, which leads to the release of cytoplasm, as well as oxidation of many recalcitrant organic compounds. AOPs related to the formation of OH radicals are a new approach to treat waste sludge, which will accelerate an oxidative degradation of numerous organic compounds dissolved in supernatant.

Catalase (H_2O_2:H_2O_2-oxidoreductase, EC 1.11.1.6), a terminal respiratory enzyme, is present in all aerobic living cells, can break down H_2O_2 into water and molecular oxygen with a two-electron transfer mechanism and protect cells from damage caused by reactive oxygen species [13,14]. The measurement of catalase activity has been developed to quantify the microbial content, assess activity of activated sludge and wastewater quality in sewage treatment plant because of its so widespread presence. For example, specific activity of catalase in activated sludge from Jones Island municipal wastewater treatment plant was in the range of 353–550 mmol H_2O_2 decomposed/mg Protein. Catalase is active over a wide range of pH values (3.0–9.0), but catalase activity is temperature sensitive, i.e., active at low temperature (4 °C to 25 °C) and gradually inactive beyond 40 °C [15]. As far as sludge pretreatment by AOPs with H_2O_2

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Table 1
Characteristics of waste activated sludge used in this study.

TSS (mg/L)	TCOD (mg/L)	TC (mg/L)	Soluble TOC[a] (mg/L)	Soluble COD[a] (mg/L)	VSS/TSS
5784	5747.51	2034.41	22.76	37.77	0.71

[a] Values of supernatant.

is concerned, it should consider the impact of catalase on sludge disintegration, i.e. which temperature is suitable to begin dosing H_2O_2 during the AOP of microwave and H_2O_2 treating waste activated sludge. However, the information is yet unknown about the impact of catalase on sludge pretreatment by the AOP of microwave and H_2O_2. In addition, high operational pressure in the closed test system using the AOP of microwave and H_2O_2 in these researches [8,10–12] may limit its full-scale application of sludge pretreatment. Therefore a H_2O_2 dosing strategy was proposed in this study, in which H_2O_2 should be dosed at a suitable temperature which the catalase loses its activity during sludge pretreatment by the AOP of microwave and H_2O_2 operated at the ambient pressure. Through investigating the impact of H_2O_2 dosing strategy on sludge disintegration, the aims of this study were to optimize H_2O_2 dosage in the AOP of microwave and H_2O_2 treating the waste activated sludge, and to elucidate the fate and distribution of organic matters in the waste activated sludge.

2. Materials and methods

2.1. Sludge and apparatus

The waste activated sludge was obtained from Gaobeidian municipal wastewater treatment plant (WWTP) with conventional activated sludge process in Beijing, which is the largest full-scale municipal wastewater treatment plant in China. The design capacity of this WWTP is 600,000 t/d, and its sludge age is operated at 20 d. In this study, total suspended solids (TSS) concentration of the waste activated sludge was adjusted to about 15 g/L for batch tests, and characteristics of the WAS are listed in Table 1.

An industrial microwave oven set at 2450 MHz is made by Julong Corp (BaoDing, China) on the basis of our design, which is equipped with a rotating blade homogenizer, and a thermocouple temperature sensor to monitor the real-time temperature. The power of the microwave oven is in the range from 0 W to 1000 W, but the microwave oven was operated at 600 W in this study. Hydrogen peroxide (A.R., 30%, w/w) at density of 1.12 g/ml was used in this study, and its concentration is 336,000 mg/L.

2.2. Batch experiments

2.2.1. Catalase activity test of the WAS

Batch experiments at different temperatures (15 °C, 25 °C, 35 °C, 45 °C, 60 °C, and 80 °C) were firstly carried out to evaluate the catalase activity in waste activated sludge. Except batch experiments at 15 °C in a water bath for 3 min, respectively, the WAS of 290 mL in the beaker of 1 L was moved into the water bath at the same temperatures as soon as it was heated by the microwave oven to the set temperatures (25 °C, 35 °C, 45 °C, 60 °C, 80 °C), and 10 mL of H_2O_2 (30%) was then dosed and mixed with the WAS in the beaker for 3 min at the set temperature of the water bath. Samples were regularly taken to monitor variations of H_2O_2 concentration in the beaker. Batch experiments at each set temperature were carried out in duplicate.

2.2.2. Sludge pretreatment by the AOP of H_2O_2 and microwave

Batch experiments were carried out in 1 L beakers with 300 mL of waste activated sludge in the microwave oven. The WAS in the beaker was firstly heated at the rate of 20 °C/min by the microwave oven to reach the set temperature, and then the microwave oven was stopped in order to dose H_2O_2 (A.R., 30%) in the beaker, and finally the microwave oven was turned on again to heat the WAS mixed with H_2O_2 till 100 °C. The H_2O_2 dosage was determined according to the ratio of H_2O_2 concentration and COD_{total} concentration of the WAS, which was set at 0, 0.1, 0.5, 1.0, 2.0, 4.0 in this study, respectively. Samples were taken immediately after the treatment by the AOP of H_2O_2 and microwave, and then cooled for analysis. All of these batch experiments were carried out in duplicate.

2.3. Analysis

The concentration of residual H_2O_2 in sludge samples was determined by the colorimetric method with Ti [16]. Samples' filtrate of 0.45 μm membrane was used to measure concentrations of soluble total organic carbon (TOC) and soluble chemical oxygen demand (COD). The soluble TOC was determined by a TOC-VCPH analyzer (Shimadzu, Japan). The total TOC of the waste activated sludge was measured by a solid TOC analyzer SSM-5000A (Shimadzu, Japan). Because the residual H_2O_2 in the WAS has strong interference on COD measurement [17], COD were thus determined after the removal of residual H_2O_2 by adding catalase (Sigma C9322). The COD, TSS, VSS (volatile suspended solids) are measured according to the APHA [18], SEM pictures of the WAS before and after the treatment by the AOP of H_2O_2 and microwave were taken by a HITACHI S-3000N (Hitachi, Japan).

3. Results and discussion

3.1. Catalase activity

As shown in Fig. 1, the concentrations of residual H_2O_2 in the WAS decreased more drastically at the low temperatures of between 15 °C and 45 °C than those at the high temperatures (60 °C and 80 °C) in the first minute. These results clearly revealed that the catalase remained active until 45 °C, and gradually lost its activity beyond 45 °C, confirming their research results [15].

According to the enzyme activity definition, one unit of catalase activity corresponds to the breakdown of 1 μmol of H_2O_2 per

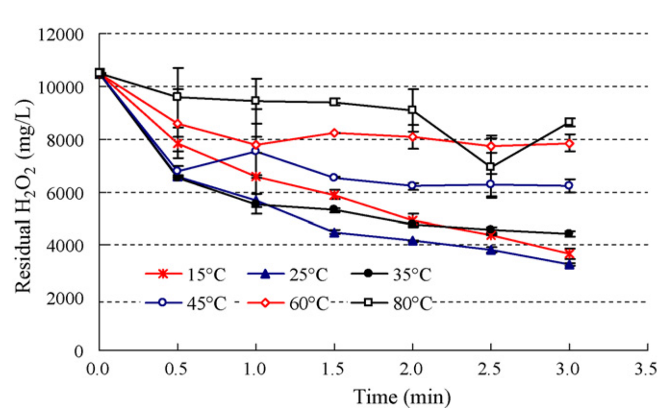


Fig. 1. Changes of H_2O_2 decomposition by catalase activity in waste activated sludge at different temperatures (initial TCOD of the WAS = 5000 mg/L; initial H_2O_2 dosage = 11,000 mg).

minute under specified conditions [14]. In this study, the specific catalase activity in the waste activated sludge was 32.1 units/mgVSS at 25 °C, and decreased to 3.7 units/mgVSS at 80 °C. Evaluation of catalase activity in waste activated sludge is helpful to optimize H_2O_2 dosage in order to save H_2O_2 cost in sludge pretreatment by the AOP using H_2O_2.

On the basis of this study and other study [8] about sludge pretreatment by the AOP of microwave and H_2O_2, the ramp time was set at the rate of 20 °C/min, which means that it takes about 1 min to reach the temperature (40 °C or 45 °C) of the denature point of catalase in activated sludge. In this short time, the H_2O_2 dosed at temperature low than 45 °C will be decomposed by over 60%. Therefore the preheating sludge before H_2O_2 dosing could save the usage of H_2O_2 and make the sludge pretreatment by the AOP of microwave and H_2O_2 more cost effective.

3.2. Temperature of H_2O_2 dosing

Compared to minor soluble COD (SCOD) release into the supernatant in the control experiment of sludge pretreated only by the microwave in the absence of H_2O_2, a considerable increase in sludge solubilization occurred in batch experiments with the AOP of microwave and H_2O_2 (Fig. 2). The higher temperature H_2O_2 dosed at, the quicker SCOD released. For example, the rate of SCOD release in the batch experiment of H_2O_2 dosing at 80 °C was much higher than that at 60 °C, though their behaviors of SCOD release were similar from 90 °C to 100 °C. These results indicated that the sludge disintegration by the AOP of microwave and H_2O_2 was rapid and temperature sensitive since 60 °C. As mentioned above, catalase is active in the range of the low temperature (15–45 °C) and gradually inactive beyond 45 °C. In the test of H_2O_2 dosing at 15 °C, the residual H_2O_2 concentration sharply decreased from 11,000 mg/L to 3190 mg/L in the temperature range of 15 °C to 40 °C due to catalase actively decomposing H_2O_2, and then maintained stable at the range of 3190–2631 mg/L between 40 °C and 80 °C. Different from the behavior of H_2O_2 at 15 °C, the residual H_2O_2 concentrations in both tests of H_2O_2 dosing at 60 °C and 80 °C, respectively, decreased slowly along with temperature increasing, but were higher than that at 15 °C in the end of batch experiments. Due to lost activity of catalase at high temperatures (60 °C and 80 °C), such decrease of H_2O_2 concentration nearly resulted from the AOP of microwave and H_2O_2 treating the WAS rather than catalase degradation. These results showed that the amount of H_2O_2 dosage should be optimized in order to reduce H_2O_2 by catalase degradation and then save H_2O_2 consumption in sludge pretreatment by the AOP of microwave and H_2O_2.

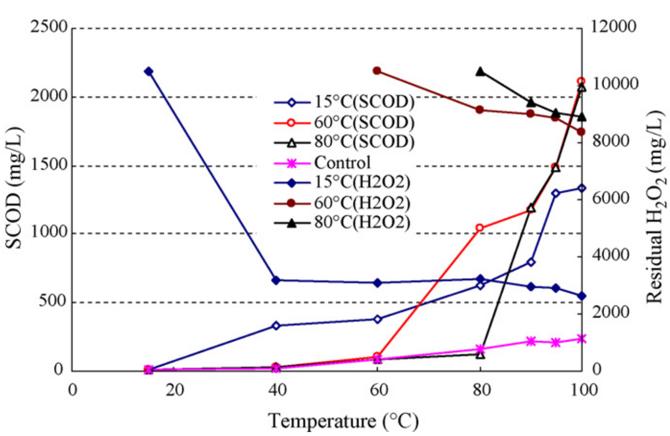


Fig. 2. Changes of SCOD and residual H_2O_2 at different H_2O_2 dosing temperatures (initial TCOD = 5000 mg/L, initial H_2O_2 dosage = 11,000 mg/L).

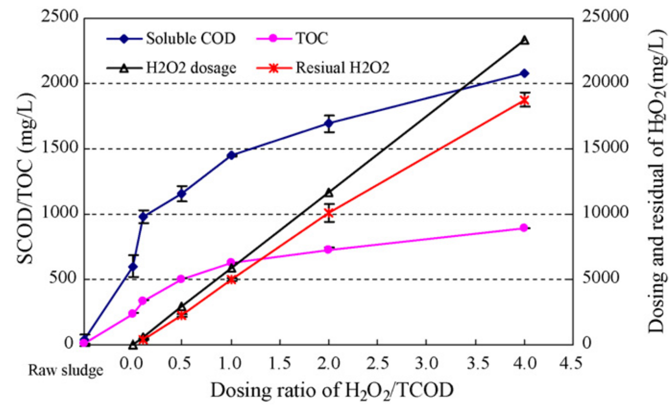
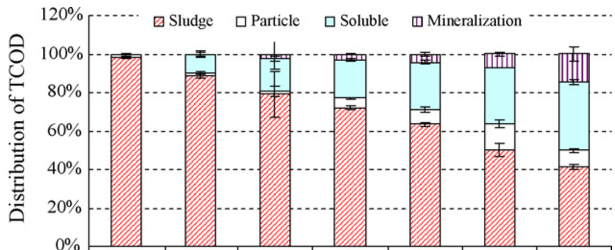


Fig. 3. Profiles of solubilization and residual H_2O_2 in the sludge pretreatment by MV–H_2O_2 at different H_2O_2/TCOD ratio (TSS = 5784 mg/L, TCOD = 5850 mg/L).

3.3. Optimization of H_2O_2 dosage

On the basis of the above mentioned results of H_2O_2 dosing at different temperatures, the waste activated sludge was firstly heated to the temperature of 80 °C, at which H_2O_2 was thus dosed in the sludge in order to avoid its degradation by the catalase, and the WAS with the addition of H_2O_2 was then continuously heated till 100 °C by the microwave oven. For optimizing the H_2O_2 dosage, batch experiments were carried out to investigate the impact of H_2O_2 dosages on sludge pretreatment by the AOP of microwave and H_2O_2. As shown in Fig. 3, the degree of sludge solubilization was strongly affected by the dosage of H_2O_2. The higher the H_2O_2 dosing ratio was, the more the SCOD and TOC released into the supernatant. Compared to the SCOD and TOC released in the test of sludge only pretreated by the microwave without H_2O_2, the SCOD release rates at 0.1, 0.5, 1, 2, 4 of dosing ratios of H_2O_2/TCOD, respectively, in the WAS pretreated by the AOP of microwave and H_2O_2 were 63.67%, 91.87%, 140.59%, 181.61% and 246.36%, respectively, and the TOC release rates at these different dosing ratios of H_2O_2/TCOD were 42.20%, 115.50%, 167.15%, 211.98% and 282.52%, respectively. Although the AOP of microwave and H_2O_2 was effective for sludge pretreatment in this study, there were still high concentrations of residual H_2O_2 in the WAS, ranging 436–18773 mgH_2O_2/L. The higher the H_2O_2/TCOD dosing ratio was, the less the consumed H_2O_2 was. The percentages of consumed H_2O_2 in the AOP of microwave and H_2O_2 treating the WAS were 25.38%, 22.53%, 14.82%, 13.61% and 19.63% at different H_2O_2/TCOD ratios of 0.1, 0.5, 1, 2, 4, respectively, neglecting the degradation of H_2O_2 by catalase in the WAS because of H_2O_2 dosing at 80 °C. Therefore, the optimum range of H_2O_2/TCOD ratio should be between 0.1 and 1.0 based on the results of SCOD and TOC releases and the residual H_2O_2 concentration in the batch tests.

In a study of comparing mechanical, thermal and oxidative disintegration techniques, thermal treatment with or without the H_2O_2 addition was considered the least interesting sludge pretreatment technology among the four based on results of specific energy and COD release [19]. Sludge thermal pretreatment [20] and sludge ozonation [21] have been successfully applied in practice. However, compared with the threshold value of released COD at 150 °C in sludge thermal treatment processes [19], the sludge disintegration in the AOP of microwave and H_2O_2 mainly occurred in the range of 80 °C and 100 °C (Fig. 2), which implied that it would save energy consumption if the recovered heat were used to preheat the excess sludge before the AOP of microwave and H_2O_2. The sludge pretreatment by the AOP of microwave and H_2O_2 is less cost effective than that by ozonation, because the prices of ozone and H_2O_2 were €1.0–1.5/kgO_3 [21] and €1.0/kgH_2O_2 [22], and the recommend ozone dosage (0.2 kgO_3/kgTSS) [23] in sludge pretreatment



	Raw sludge	0.00	0.10	0.50	1.00	2.00	4.00
Mineralization	0.01	0.00	149.88	188.42	246.22	415.79	847.41
Soluble	37.77	601.19	983.93	1153.50	1446.38	1693.02	2082.25
Particle	65.51	61.66	80.93	296.74	447.04	817.00	508.70
Sludge	5747.51	5252.66	4636.06	4212.15	3711.16	2925.00	2412.45

Dosing ratio of H_2O_2/TCOD

Fig. 4. The fate of organic matters in the WAS pretreated by the AOP of microwave and H_2O_2 at different ratios of H_2O_2/TCOD (TCOD = 5850 mg/L) (concentration unit in the table, mg/L).

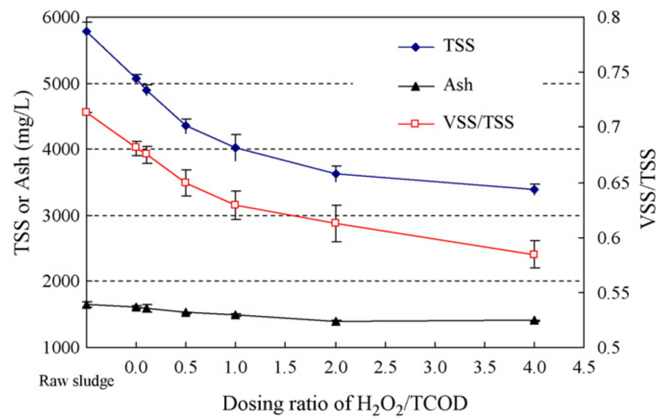


Fig. 5. Changes of TSS, ash and VSS/TSS in the sludge pretreatment by MV–H_2O_2 process at different H_2O_2 dosages (TSS = 5784 mg/L, TCOD = 5850 mg/L).

by ozonation was less than that (1 kg H_2O_2/kgTSS) in the AOP of microwave and H_2O_2 of this study. Notably, it is found in this study that the residual H_2O_2 in the sludge bulk solution was as high as over 75%, which means that there is great potential to reduce the dosage of H_2O_2. Therefore the H_2O_2 dosage in the AOP of microwave and H_2O_2 for sludge pretreatment still needs further optimization to increase the amounts of H_2O_2 consumption in order to make it cost effective technology through reducing both H_2O_2 costs and residual H_2O_2 in sludge.

3.4. Fate of organic matters in the WAS

Due to disintegration, solubilization and mineralization in the WAS pretreatment by the AOP of microwave and H_2O_2, the waste activated sludge was transferred into four parts as residual solids, particles in suspension (after 30 min settling), soluble substances, and CO_2. In this study, the changes of these four parts were investigated using their distribution in TCOD. Fig. 4 shows changes of the TCOD distribution in the WAS pretreated by the AOP of microwave and H_2O_2. It is clear that nearly all TCOD contents in the raw sludge are distributed on solids, which are similar to the sludge pretreatment by the ozonation [23]. Along with the increasing H_2O_2/TCOD ratio in the WAS pretreated by the AOP of microwave and H_2O_2, the contents of TCOD on particles, soluble substances and mineralization increased, and the TCOD distribution on solids decreased, i.e., contents of TCOD on particles, soluble organic matters and mineralization were increased from 1.04%, 10.16% and 0% in the WAS pretreated only by microwave without H_2O_2, respectively, to 8.69%, 35.59% and 14.48% at 4 of H_2O_2/TCOD ratio in the sludge pretreated by the AOP of microwave and H_2O_2; the content of TCOD on residual solids were correspondingly decreased from 88.79% to 41.23%. The fractions of soluble organic matters and particles significantly

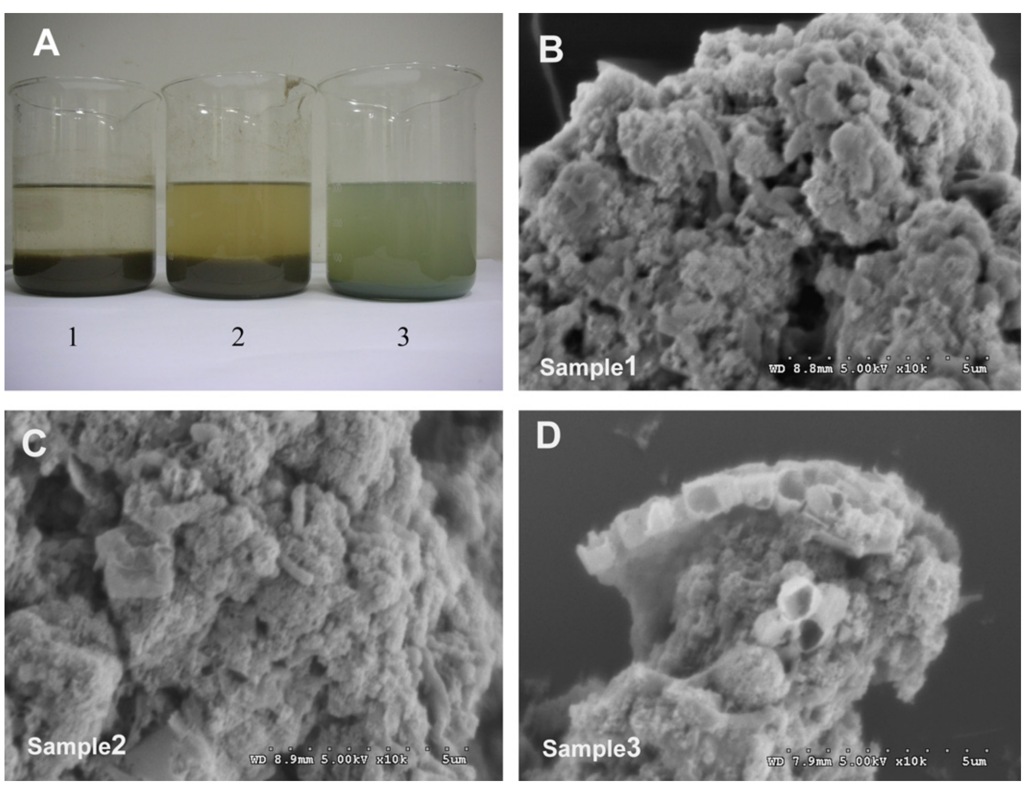


Fig. 6. Morphological changes of sludge. (A) Sludge suspension after pretreatment (1, raw sludge; 2, sludge treated by microwave; 3, sludge treated by microwave-H_2O_2 at 4 gH_2O_2/gTCOD); (B)–(D) are SEM photos of these three samples (×10 K).

increased with the H_2O_2 dosage increasing at the low range of H_2O_2/TCOD ratio between 0.1 and 1.0, meanwhile the fraction of mineralization was relatively small, less than 5%. However, the rate of mineralization increased faster than those of solubilization at high H_2O_2 dosages (ratios of 2 H_2O_2/TCOD and 4 H_2O_2/TCOD), e.g., the concentration of soluble substances at 4.0 of H_2O_2/TCOD ratio was 2.12 times of that at 0.1 of H_2O_2/TCOD ratio, but the mineralization at 4.0 of H_2O_2/TCOD ratio was as high as 5.65 times of that at 0.1 of H_2O_2/TCOD ratio. For sludge pretreatment, disintegration and solubilization are preferred to mineralization because it is advantageous to recycle the released organic matters in the biological wastewater treatment processes, i.e., cryptic growth for reducing sludge production and providing carbon source for denitrification, as well as mineralization would result in more CO_2 emission and more H_2O_2 consumption.

The effects of sludge disintegration, solubilization and mineralization by the AOP of microwave and H_2O_2 were also determined in terms of VSS/TSS ratio, TSS and ash contents in the WAS. As shown in Fig. 5, the ratio of VSS/TSS in the range of 0.1–1.0 of H_2O_2/TCOD ratios decreased more sharply than that in the range of 2.0 to 4.0 of H_2O_2/TCOD ratios, but it is interestingly found that the contents of ash in the WAS pretreated by the AOP of microwave and H_2O_2 at different H_2O_2 dosages maintained stable and varied in the range of 1617–1406 mg/L. These results, as well as the above mentioned fate of organic matters, clearly showed that both the solublized and the mineralized fractions increased along with the WAS reacted more H_2O_2.

3.5. Morphological changes of sludge

From direct observation as shown in Fig. 6(A), the color of sludge was changed from the original dark-brown to the pale after treatment by the AOP of microwave and H_2O_2. And the bulk solution was changed to green after 5 min treatment gradually with the increase dosage of H_2O_2. Compared to the agglomerated floc in raw sludge shown in Fig. 6(B), little morphological changes of sludge flocs occurred in the sludge pretreated by microwave in the absence of H_2O_2 shown in Fig. 6(C), but the cells in sludge were completely destroyed by the AOP of microwave and H_2O_2, with the breakage of the cell membrane and no whole cell observed in the sight field (Fig. 6(D)). Although the cell in the sludge pretreatment by the AOP of microwave and H_2O_2 was totally broken, the percentage of organic matter released into the supernatant was not over than 40% (Fig. 4). In addition, it is regarded that the microwave heating process could break down particles with or without hydrogen peroxide [11]. However, it is noted in this study that the sludge flocs in the sludge pretreatment only by microwave without H_2O_2 were not completely disintegrated because of short time heating below 100 °C. Obviously, the AOP of microwave and H_2O_2 is effective to disintegrate sludge and release cytoplasm into bulk solution through breaking the cell membrane.

4. Conclusions

The effects of sludge pretreatment by the AOP of microwave and H_2O_2 at the ambient pressure were investigated by the batch experiments, and conclusions are made as follows:

(1) The sludge pretreatment by the AOP of microwave and H_2O_2 should consider the impact of catalase in the activated sludge on degradation of H_2O_2. In this study, the catalase was active at low temperatures of between 15 °C and 45 °C, and gradually lost its activity at the high temperatures (60 °C and 80 °C).

(2) The H_2O_2 dosing strategy in the AOP of microwave and H_2O_2 treating the waste activated sludge was determined on the basis of the results of catalase activity. The waste activated sludge was firstly heated by the microwave oven to 80 °C, at which H_2O_2 was dosed in the sludge, and the mixed liquor was then continuously heated till 100 °C. Meanwhile, the H_2O_2 dosage in the AOP of microwave and H_2O_2 treating waste activated sludge should be in the range of 0.1–1.0 of H_2O_2/TCOD ratios considering its sludge disintegration performance and costs of H_2O_2.

(3) Along with the increasing H_2O_2/TCOD ratio in the WAS pretreated by the AOP of microwave and H_2O_2, the contents of TCOD on particles, soluble substances and mineralization increased, and the TCOD distribution on solids decreased.

Acknowledgements

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