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Enhancement of microwave effect with addition of chemical agents in solubilization of waste activated sludge

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ABSTRACT

Microwave (MW) is a promising method for solubilization of wasted activated sludge (WAS) due to the accelerated reaction rates, environmental friendliness and low overall cost compared to conventional heating. In this study, to enhance the MW effect on solubilization of WAS, several chemical agents were applied including H_2SO_4 as the acidic material and NaCl and $CaCl_2$ as ionic materials. In the experiment of salt-assisted MW pretreatment, $CaCl_2$ turned out to be ineffective on WAS solubilization, whereas NaCl had a positive effect on WAS solubilization compared to that of MW only assisted pretreatment. Thus, NaCl was considered to be appropriate salt to assist MW pretreatment for WAS solubilization. In the chemically assisted MW pretreatments, H_2SO_4 assisted MW pretreatment was the most effective for WAS solubilization. However, the application of H_2SO_4 to MW pretreatment has the drawbacks of reuse, which may increase the operating costs. Thus, food wastewater was used for enhancement of MW efficiency on WAS solubilization due to its high NaCl and acidity. As a result, the application of food wastewater to WAS afforded the solubilization efficiency higher than that of MW-H_2SO_4 pretreatment.

Introduction

The management of waste activated sludge (WAS) from wastewater treatment plants has attracted increasing concern because of the continuous increase in sludge generation [1]. In Korea, conventional WAS treatment method has been ocean dumping, but other WAS disposal routes should be adopted because ocean disposal was recently banned and the costs of treatment for WAS cause over 50–60% of total operating cost [2]. In fact, WAS, which is mainly composed of microbial cells, consists of 59–88% (w/v) organic materials and can therefore become a source of renewable energy like methane or hydrogen [3].

Anaerobic digestion of WAS has been a widely promising method for WAS treatment because it produces safe and clean energy and also contributes to reducing this abundant waste [4]. However, as the organic compounds of WAS are generally within the cell wall and extracellular polymeric substances (EPS) and as semi rigid structure of the cell envelope inhibits the osmotic lysis of the cells [5], producing biogas from anaerobic digestion requires a long hydraulic retention time. In this aspect, increasing the hydrolysis of WAS, which is the rate-limiting step, is the main concern for anaerobic digestion process. Therefore, many studies to increase biogas recovery have focused on accelerating the hydrolysis of WAS.

Several different pretreatment methods for sludge destruction are being investigated, including acid/alkali, ultrasound and heating [6–8]. Microwave (MW) has emerged recently because it is superior to conventional thermal heating due to its ability to heat rapidly, accelerate reaction rates [9] and provide environmental friendly energy [3]. The main effects of MW irradiation on WAS solubilization reactions are distinguished as thermal effects and non-thermal effects. Thermal effects are based on direct coupling of electromagnetic energy with the molecules of the solvents and reagents, whereby the magnitude of the heating depends on the dielectric properties of the molecules. Non-thermal effects are electrostatic polar effects, leading to dipole-dipole type interactions between the dipolar molecules and charges in the electric field [10,11].

In MW irradiation process, high temperature and pressure treatment conditions increase WAS solubilization, but energy consumption of the process needs to be decreased. In this scenario, chemically assisted MW pretreatment can give a way to save energy and to achieve high sludge solubilization efficiency. Some

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studies showed that combined MW-alkali treatment could produce a synergy effect and become a cost effective alternative for sludge solubilization [12,13]. On the other hand, some studies reported that the presence of salts, which increase the effects of dielectric loss and MW coupling, could produce superheating condition since the addition of salts to solutions could enhance conductivity and have a considerable effect on the rate of heating [14]. Thus, superheating effects can be amplified by the supplement of NaCl. In this sense, the mixture of food wastewater and WAS for the acceleration of solubilization is promising due to the high NaCl concentration of food wastewater.

The objective of the study is to enhance the MW efficiency for the cell destruction of WAS. Many materials were evaluated as the accelerating agents for enhancing the MW efficiency on WAS solubilization. H_2SO_4 was used as the acidic material and $CaCl_2$ and NaCl were used as the ionic materials. We also evaluated the effect of food wastewater for the enhancement of MW effect on WAS solubilization by varying the mixture ratio of WAS and food wastewater.

Experimental

WAS characteristics

WAS samples were sourced from the return line of the secondary settling tank and thickener of Suyoung wastewater treatment plant in Busan, Korea. The sludge used in the study was from conventional aerobic activated process operated at an average hydraulic retention time of 6 h and a sludge retention time of 13 d. Table 1 presents the characteristics of return sludge (WAS 1) in conventional process and thickener sludge (WAS 2). The collected WAS samples were immediately sieved with mesh no. 24 (0.800 mm) and used for every experiment within 5 h.

Experimental condition

MW irradiation and conventional heating (CH)

MW-based pretreatment was conducted in a MW system (KMIC-2 kW, Korea Microwave Instrument Co., Korea) having a range of 0–2 kW power at a frequency of 2.45 GHz. Conventional heating (CH) experiments were conducted in a water bath (CW-10G, Jeiotech, Korea). In the MW experiment, quartz sample bottles with an effective volume of 50 mL were equipped in turntable for a homogeneous MW distribution. To compare the effect of MW and CH on solubilization of WAS, solubilization experiments using WAS 1 were performed at 50, 75 and 100 °C by each heating method. Table 2 shows the experimental conditions of MW irradiation and CH on WAS solubilization. After reaching the set temperature, reaction time was 10 min for all methods. In the MW system, the temperature for WAS solubilization was automatically controlled by varying the input power and temperature was directly measured using a thermocouple thermometer.

Acid-assisted and salt-assisted MW pretreatment

To enhance the MW effect, several kinds of chemical materials were used. Before the experiments, WAS samples were first irradiated at 200, 400 and 600 W to evaluate the effect of MW power and to select the optimal MW power. After optimal MW power was chosen, the acid-assisted and salt-assisted MW pretreatments for WAS solubilization were carried out with addition of H_2SO_4 as the acidic material and NaCl and $CaCl_2$ as the salt materials to WAS 2 samples, respectively. In the acid-assisted MW pretreatment, WAS was first pretreated with H_2SO_4 for 24 h before MW irradiation. In the salt-assisted MW experiment, however, as soon as the salt was injected into WAS, the sample was irradiated by MW. Table 3 presents the specific experimental conditions. Considering the inhibitory effect of salt on the microbial environment, the maximum concentration of salt was limited with a Cl^- concentration of 7000 mg/L which were 98.5 mM as $CaCl_2$ and 197.2 mM as NaCl.

The high NaCl concentration of food wastewater suggests that it can be used to accelerate WAS solubilization. Two samples of WAS were mixed with food wastewater based on Cl^- concentration, which were 14.1 and 42.3 mM Cl^- concentration after mixture with food wastewater, respectively. The characteristics of food wastewater are shown in Table 4. The suspended solid concentration of food wastewater was higher than that of WAS. Thus, food wastewater was also sieved with mesh no. 24 (0.800 mm) and then filtered with membrane filter before mixture with WAS to exclude any particles.

Table 1
Characteristics of the raw WAS used in the experiments.

Item	WAS 1	WAS 2
pH	6.72 ± 0.04	6.60 ± 0.05
VSS (g/L)	8.20 ± 0.38	47.79 ± 5.00
VSS/TSS	0.859	0.763
SCOD (g/L)	0.030 ± 0.003	0.193 ± 0.074
Water content (%)	99.045 ± 0.046	95.221 ± 0.500

Table 2
The experimental conditions of MW irradiation and CH.

Trial	Methods	Temperature(°C)	MW power (W)	Reaction time (min)
1	MW irradiation	50, 75, 100	900	10
2	CH	50, 75, 100	–	10

Analytical methods

All experiments were performed in triplicate to minimize random errors and the results are presented in mean values. Total suspended solid (TSS), volatile suspended solid (VSS), chemical oxygen demand (COD), total nitrogen (TN) and total phosphorus (TP) were analyzed by the procedure of Standard Methods [15]. In the study, soluble COD (SCOD) was defined as any organic material that passed through a 0.45 μm membrane filter. The Cl^- concentration was also analyzed after filtration by ion chromatography (ICS-1000, DIONEX, USA) equipped with column (Dionex IonPac^{TM} AG14, 4 mm × 250 mm, USA) and suppressed detector using solvent consisting of 0.084 g/L $NaHCO_3$ and 0.371 g/L Na_2CO_3. Column and detector temperature were 30 °C. pH was measured by the pH meter (Orion, Model 520A, USA).

Results and discussion

Comparison of MW irradiation and CH

The main organic compounds in sludge are carbohydrates, proteins and lipids. Under MW irradiation, floc structure of

Table 3
The experimental conditions of WAS solubilization by chemically assisted MW pretreatment.

Trial	Chemical	Dosage	MW power (W)	MW reaction time (min)
1	H_2SO_4	0, 0.1, 0.2, 0.4, 0.8, 1.0 [M H_2SO_4]	600	3, 6, 10
2	$CaCl_2$	0, 0.8, 2.0, 14.1, 42.3, 70.4, 126.8, 197.2 [mM Cl^-]		
3	NaCl	0, 14.1, 42.3, 70.4, 126.8, 197.2 [mM Cl^-]		
4	Food wastewater	0, 14.1, 42.3 [mM Cl^-]		