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Effect of microwave irradiation on the disintegration and acidogenesis of municipal secondary sludge

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ABSTRACT

This work experimentally determined the effect of microwave treatment on the disintegration and acidogenesis of municipal secondary sludge. Sludge samples (500 g) were heated for 0, 3, 5, 7, 9, 11, and 15 min in a microwave oven (2450 MHz, 700 W). The solubilization degree (soluble chemical oxygen demand (SCOD)/COD) of sludge increased asymptotically with microwave irradiation time from 2% at 0 min to 22% at 15 min. The concentrations of soluble protein, carbohydrate, lipid, and calcium also increased with microwave irradiation time. The biochemical acidogenic potentials (BAP) of sludge increased from 3.58 to 4.77 g COD l^{-1}. The results show that microwave irradiation increases the solubilization degree and BAP of municipal secondary sludge.

1. Introduction

Processing of sewage sludge for disposal is one of the most important and complex problems in the operation of municipal wastewater treatment plants [1]. Anaerobic digestion has long been used to treat sludge generated by the treatment of municipal and industrial wastewater. Sludge consists mainly of bacterial materials that commonly withstand direct anaerobic degradation because bacterial cell walls form physical and chemical barriers to enzymatic degradation and hydrolysis of intracellular organic material from cells in the sludge. As a consequence, long retention times of 20–30 days are required to reach moderate efficiencies of 30–50% in typical anaerobic digestion systems [2,3].

Anaerobic degradation of particulate materials and macromolecules occurs in four steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In sludge digestion, hydrolysis is the rate-limiting step [4]. During this process, bacteria release extracellular enzymes that break down and solubilize organic particulate matter to use particulates as substrates in subsequent reactions. Therefore, to improve digestion efficiency, the most logical approach is to disrupt the microbial cells in the sludge.

Disruption of bacteria in sludge may be performed mechanically, ultrasonically, chemically, or thermally [5]. Mechanical pretreatment is highly effective but is complicated and expensive [1]. Sonication can disrupt 70–100% of sludge cells, but this approach is energy-intensive [1]. Chemical and thermochemical pretreatments are efficient [6], but they require extreme reaction conditions and commonly require the use of specialized materials. Thermal treatment prior to anaerobic digestion has been examined as a possible approach [1]. Conventional low-temperature thermal treatment necessitates a longer contact time than high temperature treatment [7]. The high costs of wet sludge disintegration, in addition to corrosion problems and limited knowledge of the process itself, prevent the approach from achieving its potential as an anaerobic process [1]; consequently, an effective and economical pretreatment method is essential. One possible approach is to use microwave irradiation.

Microwaves are used in many and varied applications, including decomposition of organic materials, sterilization of medical waste, and inactivation of microorganisms [8]. The advantages of microwaves in these applications include rapid heating, pathogen destruction, ease of control, compactness, and low overall cost [9]. Destruction of microorganisms is generally thought to occur due to the thermal effects of microwave exposure, although several researchers have investigated whether such irradiation also has a non-thermal effect [10]. The application of microwave fields can cause polar side-chains of macromolecules to align with the

Abbreviations: BAP, biochemical acidogenic potential; BMP, biochemical methane potential; BOD, biochemical oxygen demand; COD, chemical oxygen demand; HRT, hydraulic retention time; *k*, rate constant; SCOD, soluble chemical oxygen demand; *t*, incubation time; TS, total solid; TVFA, total volatile fatty acid; $TVFA_{max}$, maximum TVFA production; VFA, volatile fatty acid; VS, volatile solid.

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Table 1
Characteristics of secondary sewage sludge analyzed in the present study.

Parameters	Concentration ($g\,l^{-1}$)
pH	7.0 ± 0.1[a]
Chemical oxygen demand	24.7 ± 1
Total organic carbon	9.89 ± 0.1
Total solid	25.7 ± 0.3
Volatile solid	19.6 ± 0.1
Suspended solid	23.4 ± 0.1
Volatile suspended solid	17.8 ± 0.2
Carbohydrates	3.63 ± 0.05
Protein	11.6 ± 1
Lipid	1.52 ± 0.01
TKN	1.97 ± 0.16
Sulfate	0.021 ± 0.001
Sodium	0.175 ± 0.001
Potassium	0.07 ± 0.001
Calcium	0.084 ± 0.001
Magnesium	0.049 ± 0.001
Phosphate	0.111 ± 0.003

[a] No unit.

direction of the electric field, possibly leading to the breakage of hydrogen bonds and alteration of the hydration zone. The principal factors of microwave irradiation that affect dielectric materials include temperature, radiation time, and penetration depth [8].

Organic waste is fermented under anaerobic conditions until the volatile fatty acid (VFA) concentration reaches a stable maximum level, the VFA potential, also known as the biochemical acidogenic potential (BAP). The BAP test, which measures the fermentable fraction of an organic waste under anaerobic conditions, is similar to the biochemical oxygen demand (BOD) test for aerobically biodegradable organic matter and the biochemical methane potential (BMP) for anaerobically biodegradable organic matter [11]. The BAP test is a simple procedure employed to determine the amount of VFA that can be formed from organic matter by digestion [12]. The VFA potential of wastewaters has been reported [11–14]; however, little research has focused on VFA production from sludge [15]. To facilitate modeling of anaerobic digestion, and to guide the design and operation of anaerobic digestion systems, the behavior of acidogens should be understood, because they produce the substrate for methanogens [16].

Currently, information is lacking concerning the pretreatment of sludge with microwaves and use of the BAP test for pretreated sludge. Therefore, this study was conducted to investigate the effects of microwave irradiation on hydrolysis and subsequent acidogenesis in the anaerobic degradation of secondary sludge.

2. Materials and methods

2.1. Secondary sludge

Municipal secondary sludge (Table 1) was obtained from a municipal wastewater treatment plant in Pohang, South Korea. The sludge was concentrated by gravity settling of which concentration was 26 $g\,l^{-1}$ total solid (TS). The sludge was stored at 4 °C before use.

2.2. Microwave irradiation

Samples were irradiated in a microwave oven (RE-S50, Samsung, Korea) equipped with a magnetron (2450 MHz, 700 W). The dimensions of the wave guide were 336 mm × 235 mm × 346 mm. For pretreatment, 500 g of the secondary sludge was transferred to a Pyrex vessel (133 mm × 250 mm × 40 mm). The depth of the sludge irradiated was maintained at about 15 mm because the penetration depth of a similar 2450 MHz household microwave oven is 11 mm for secondary sludge [17]. To evaluate the effect of microwave irradiation on the disintegration degree of the sludge, individual samples were irradiated for 0 (control), 3, 5, 7, 9, 11, or 15 min. The initial temperature of the sludge was maintained at 10 °C, and the final temperature was measured as soon as microwaving was terminated. The microwaved sludge was cooled to room temperature, then weighed, and stored at 4 °C before being used in the following experiments.

2.3. Acidogenic inoculum system

Anaerobic seed sludge from a local municipal wastewater treatment plant was cultivated in the system to enrich a mixed population of acidogens by combining biokinetic and chemical controls [18]. A lab-scale continuously stirred tank reactor with a working volume of 1.5 l was used to culture the acidogen inoculum. The system was operated using diluted whey wastewater with a chemical oxygen demand (COD) of 20.0 ± 0.5 $g\,l^{-1}$ at 0.5 ± 0.02 day hydraulic retention time (HRT). Yeast extract was added at 1 $g\,l^{-1}$ to provide trace minerals. Temperature was maintained at 35 °C using an automated heating controller, and pH was maintained at 6 using 6.0N NaOH. The operating conditions remained constant throughout the experiment. The effluent from this system was used as seed culture for BAP experiments to minimize any confounding effects associated with use of inconsistent inocula.

2.4. Biochemical acidogenic potential

The BAP tests were conducted in 500 ml Wheaton bottles which were purged with nitrogen gas to remove oxygen, then sealed with rubber stoppers. The reactors were seeded with centrifuged sludge (26 ml) without supernatant to minimize concentrations of VFAs in the seed sludge, then incubated in a shaking incubator at 140 rpm and 35 °C under anaerobic conditions until total VFA (TVFA) variation was less than 10%. The amount of pretreated sludge used for BAP tests was 500, 493, 482, 466, 452, 431, and 383 g for microwave irradiation times of 0 (control), 3, 5, 7, 9, 11, and 15 min, respectively. Bromoethane sulfonate (1 mM) was added to inhibit methanogenesis [11].

2.5. Scanning electron microscopy

The sludge was concentrated by centrifugation (10,000 × *g*, 1 min) and rinsed with 0.1 M sodium phosphate buffer (pH 7.3). The sludge was then fixed at 4 °C for 8 h with 3% glutaraldehyde in 0.1 M sodium phosphate buffer. The sludge was washed twice for 10 min using the buffer, and then post-fixed for 4 h with 1% osmium tetroxide in the buffer. The sludge rinsed briefly with the buffer, then dehydrated using a serial concentration of ethanol. A critical point dryer, employing liquid carbon dioxide as the transition fluid, was used to dry the sludge. The sludge was placed on a stub and then sputter-coated with gold under vacuum and examined using a scanning electron microscope (S-2460N, Hitachi).

2.6. Analytical methods

All analyses were duplicated, and the results are given as mean values with standard deviations. The concentrations of VFAs were determined using a Hewlett-Packard gas chromatograph (Model 6890 plus) equipped with an Innowax capillary column and a flame ionization detector. Helium was the carrier gas, with a flow rate of 2.5 $ml\,min^{-1}$ and a split ratio of 10:1. The COD and solid concentrations were determined according to the procedures in Standard Methods [19]. The protein concentration was determined using the Kjeldahl method [19] and carbohydrate concentration was measured using the phenol–sulfuric acid method [20]. Lipid concentration was measured after extraction of lipid by a solvent

Table 2
Effect of microwave irradiation on sludge disintegration.

Microwave irradiation time (min)	Microwave irradiation energy (kJ g TS^{-1})[a]	Final temperature (°C)	Weight reduction (%)	Ca^{2+} concentration (g l^{-1})	SCOD/COD
0	0	10	0.0	0.084	0.02
3	9.8	59	1.4	0.084	0.10
5	16	77	3.5	0.088	0.17
7	23	91	6.9	0.095	0.19
9	29	Boiling	9.7	0.095	0.21
11	36	Boiling	14	0.100	0.21
15	49	Boiling	23	0.102	0.22

[a] Microwave irradiation energy (kJ g TS^{-1}) = output power (700 W) × microwave irradiation time (min)/amount of sludge (500 g) × density (1000 g l^{-1}) × 0.06/TS (g l^{-1})

(80% *n*-hexane and 20% methyl-*tert*-butyl ether) using an auto fat extraction system (2050 Soxtec, Foss). Total organic carbon concentration was measured using a Shimadzu TOC analyzer (5000-A). The concentrations of cations and anions were measured using two identical Dionex ion chromatographs (DX-120) with IonPac AS14 and IonPac CS12A columns, respectively.

3. Results and discussion

Municipal secondary sludge was pretreated using microwave irradiation to evaluate the disintegration degree of bacterial cell components. After microwave pretreatment, batch experiments were conducted to assess the fermentability of the pretreated sludge, i.e. to determine the amount of organic matter that could be converted into VFA.

3.1. Characterization of the secondary sludge

The high ratio of volatile solid (VS) to TS in the sludge, 76%, indicated that the sludge consisted mainly of organic substances (Table 1). About 90% of the VS was present in suspended form. Cations were predominantly Na^+, Ca^{2+}, Mg^{2+}, and K^+, which occurred at concentrations below those which inhibit the growth of acidogens [21]. The VS in the sludge was 59% protein, 18% carbohydrate, and 8% lipid. The chemical composition of the sludge was similar to that of a prokaryotic cell [22]. The pH of the sludge was 7.0 ± 0.1.

3.2. Effect of microwave irradiation on sludge disintegration

We hypothesized that microwave irradiation would disrupt the complex activated sludge floc structure and release extracellular and intracellular biopolymers such as proteins, carbohydrates, and lipids from the floc structure into the soluble phase, and would also enhance the solubilization of particulate COD [23,24]. The disintegration degree of the substrate can be estimated from a variety of measurements, including soluble COD (SCOD) and the concentrations of proteins, carbohydrates, and lipids. All of these measurements were greater in the pretreated sludge than in the raw sludge, and this difference increased with microwave irradiation time (Table 2; Fig. 1). These results demonstrate that microwave irradiation increased sludge solubilization [25].

During ultrasonic pretreatment in a previous study, SCOD/COD increased to about 20% after 120 min sonication at 0.33 W ml^{-1}, equivalent to 288 kJ g TS^{-1} [26]. In the present study, a similar degree of sludge disintegration was achieved after 9 min of microwave irradiation, although with lower energy consumption, 29 kJ g TS^{-1} (Table 2). The sludge boiled when subjected to 9 min of microwave irradiation, and its weight was reduced by up to 23% over 15 min (Table 2). From a microscopic perspective, an increase in the temperature of micro-bubbles is expected to generate many free radicals that would make a large contribute to organic dissolution and microbial inactivation [26,27].

As microwave irradiation time was increased from 0 to 15 min, the SCOD concentration increased from 0.38 to 5.52 g l^{-1} and SCOD/COD increased from 0.02 to 0.22 (Table 2), comparable with the results of other studies [17,28–30]. The soluble protein, carbohydrate, and lipid concentrations also increased due to sludge disintegration by microwave irradiation (Fig. 1). The exact mechanisms of protein, carbohydrate, and lipid disruption are poorly understood, but microwaves may affect chemical bonds under certain circumstances [25].

Concentrations of soluble protein show a relatively linear increase with pretreatment time up to the boiling point (Fig. 1) [29,31], but show no further increases at higher temperatures. As temperature increases, the kinetic energy of molecules increases, causing them to vibrate so violently that the bonds are disrupted; consequently, some of the hydrogen bonds and non-polar hydrophobic interactions that stabilize the protein's structure may begin to break, destabilizing the protein.

The concentration of soluble carbohydrate increased by a factor of about 11 within 15 min of microwave irradiation (Fig. 1) [29,31]. This increase may be due to disruption of cell wall polysaccharides, which are composed of many (sometimes hundreds or even thousands) sugar-ring monomers units connected by covalent glycosidic bonds [22]. These glycosidic linkages within the polysaccharides may have been broken during irradiation [32]. The concentration of soluble lipids increased with microwave irradiation time (Fig. 1), in part because phospholipids are a major structure in the cytoplasmic membrane [22]. All of these results suggest the protein, carbohydrate, and lipid concentrations increased with microwave irradiation time because the cell wall, cell membrane, or both were disrupted by irradiation.

The overall structure of the cytoplasmic membrane is stabilized by hydrogen bonds and hydrophobic interactions. Divalent cations such as Ca^{2+} also help to stabilize the membrane by combining ionically with negatively charged phospholipids [22,26]. Some divalent

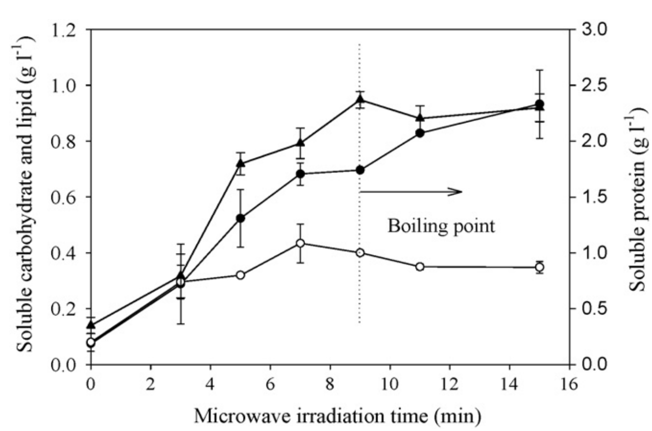

Fig. 1. Temporal trends in protein (▲), carbohydrate (●), and lipid (○) concentrations with microwave irradiation time.

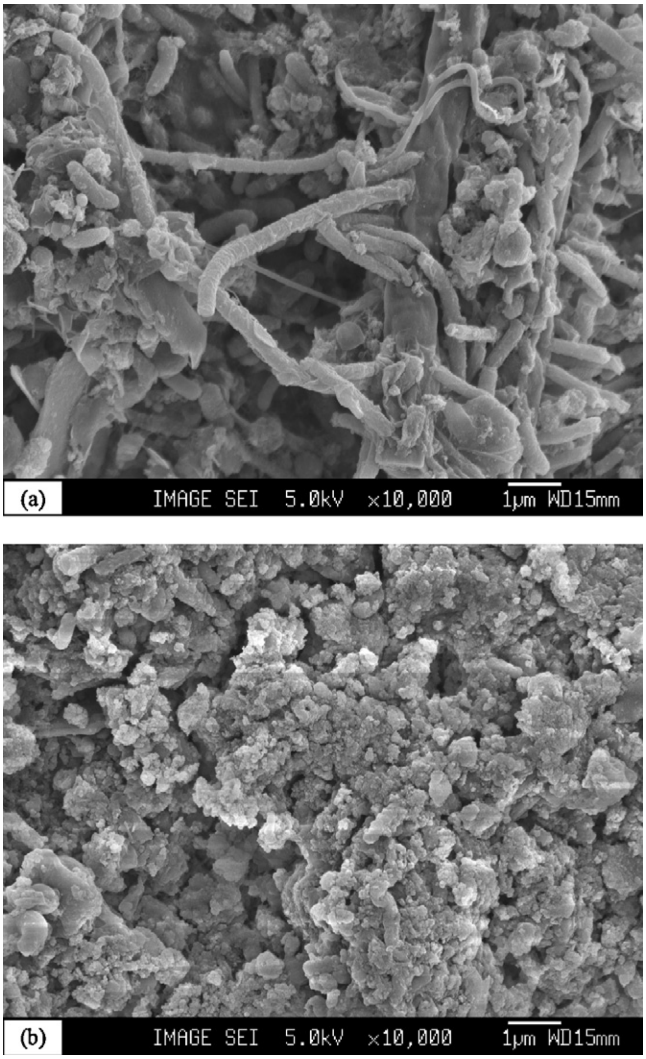

Fig. 2. Scanning electron microscope images of (a) control and (b) 7-min pretreated sludge (×10,000).

cations are released when cells are disrupted [26]; in the present study, calcium concentrations in the sludge increased from 0.084 to 0.102 g l^{-1} (Table 2); this observation is consistent with the hypothesis that microwaves disintegrate cell membranes of bacteria in the sludge.

The control and pretreated sludges were examined using a scanning electron microscope (Fig. 2). The untreated flocs were highly porous and contained filamentous bacteria (Fig. 2a), whereas after 7 min of irradiation, the structural integrity of flocs was broken down (Fig. 2b). This degradation demonstrates indirectly that intracellular substances were released from within the microorganisms.

3.3. Biological acidification of pretreated sludge

After completing the microwave irradiation experiments, BAP tests were conducted to assess the effect of sludge pretreatment on subsequent anaerobic acidogenesis (Fig. 3). The TVFA concentration of control sludge reached a maximum level at 15 days, whereas that for microwave-treated sludges reached a maximum at 11 days, thereby indicating that microwave irradiation enhances the acidogenesis rate in the sludge. The BAPs of sludges pretreated for 0, 3, 5, 7, 9, 11, and 15 min were 3.58 ± 0.10, 3.94 ± 0.13, 4.61 ± 0.08, 4.77 ± 0.11, 4.68 ± 0.09, 4.74 ± 0.11, and 4.69 ± 0.14 g COD l^{-1}, respectively (Fig. 3). High VFA concentrations in anaerobic digestion can inhibit methanogenesis. It was previously reported that the inhibitory concentration of TVFA (18% acetic acid, 50% propionic acid, 5% *n*-butyric acid, 12% iso-butyric acid, 5% *n*-valeric acid, 5% iso-valeric acid, 2% caproic acid, and 3% heptanoic acid) on the production of biogas was about 9.4 g COD l^{-1} when substrate was cellulose and 12.6 g COD l^{-1} when it was glucose [33]. In this study, maximum TVFA concentration was less than 5 g COD l^{-1}, which was lower than the inhibitory concentration.

Results from the BAP test were used to evaluate the TVFA production rate of pretreated sludge for microwave irradiation times. The cumulative TVFA productions increased exponentially to a maximum value (Fig. 3). Therefore, an exponential rise-to-maximum equation (Eq. (1)) was fitted to the data using Sigmaplot 2002 for Windows Version 8.0.

$$TVFA = TVFA_{max}(1 - e^{-kt}) \quad (1)$$

$TVFA_{max}$ is the maximum TVFA production (g COD l^{-1}), k is a rate constant (d^{-1}), and t is incubation time (d). The coefficient of determination (r^2) of the regression was greater than 0.90 in all cases. The k value was 0.18, 0.34, 0.53, 0.61, 0.68, 0.60, and 0.69 d^{-1} for microwave irradiation times of 0, 3, 5, 7, 9, 11, and 15 min, respectively. The k values for pretreated sludge were between 1.9 and 3.8 times higher than that for the control. This result implies that microwave pretreatment hydrolyzed a portion of the complex organic matter in the sludge to a readily degradable substrate before acidogenesis. The biodegradability of the sludge is enhanced by microwave irradiation; however, there was little difference in the BAPs of sludges pretreated for microwave irradiation times ranging from 5 to 15 min (Fig. 3). Therefore, in this study the optimum microwave irradiation time for BAP of secondary sludge was 5 min.

The magnitude of VS reduction in pretreated sludge increased with microwave irradiation time (Fig. 4); however, TVFA yield—the ratio of TVFA production to VS reduction—decreased with irradiation time, demonstrating that VS was transformed not only into VFA but also soluble microbial production [34]. The TVFA yield decreased from 0.77 to 0.43 g COD g^{-1} $VS_{removed}$ (0.18–0.22 g acetate g^{-1} VS_{fed}) with increased microwave irradiation time (Fig. 4). Previous studies have reported highly variable TVFA yields (0.05–0.20 g volatile acids g^{-1} VS_{fed}) [15], comparable with the results of the present study. Despite the production of VFAs, pH was maintained at 6.4 ± 0.1 in all non-pH-regulated reactors, regardless of microwave irradiation time; this finding demonstrates that the buffering capacity of the sludge naturally controlled the degree of pH reduction by VFA production.

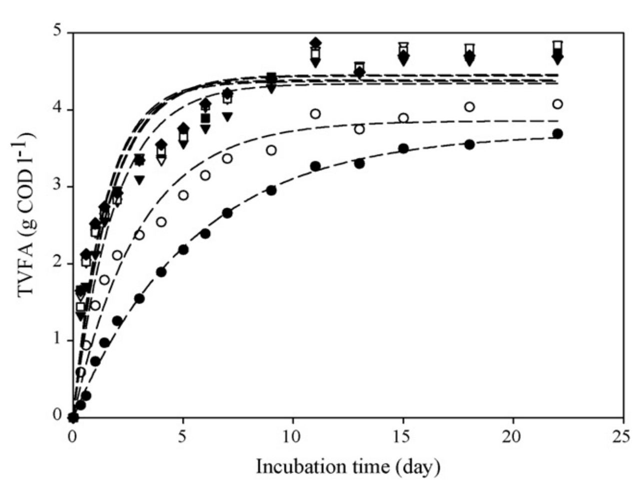

Fig. 3. Observed and predicted total volatile fatty acids productions of pretreated sludge for various microwave irradiation times (●, 0 min; ○, 3 min; ▼, 5 min; ▽, 7 min; ■, 9 min; □, 11 min; □, 15 min; - - - -, fitted equations).

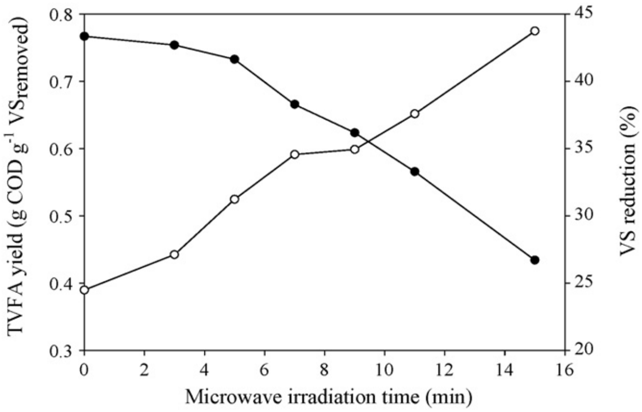

Fig. 4. Total volatile fatty acid yield (●) and volatile solid reduction (○) attained in the biochemical acidogenic potential test using pretreated sludge.

3.4. Economic aspect of sludge pretreatment using microwave irradiation

Technical and economical aspects of pretreatment methods must be considered before microwave pretreatment equipment can be integrated into a conventional digestion process [35]. Economic evaluation of adding a pretreatment to the digestion system must consider the cost of pretreatment and the benefits gained from the pretreatment process [36]. In general, heating sludge consumes a great deal of energy. Microwave heating is more effective than conventional heating. Microwaves can be focused to heat materials directly internally so heat loss through convection and conduction can be minimized, whereas during conventional heating, materials are heated from outside to inside, so heat loss of these paths cannot be avoided [9]. Microwave pretreatment has also the added benefits of athermal effects [10]. Unless the sewage treatment plant has a free source of steam, microwave heating of sludge is more efficient than conventional heating. Introducing microwave pretreatment to a digestion system can increase the rate of stabilization because HRT can be decreased from 20 days to 10 and even 5 days [36]. By decreasing HRT, sludge can be stabilized in smaller reactors, which would decrease the heating cost and the space required to house digestion reactors. Another advantage of microwave pretreatment is better dewaterability properties of digested sludge [36], which results in a smaller volume of sludge for disposal if it is not used for other purposes.

The economic viability of microwave pretreatment cannot be assessed using the results of this study because it was a bench scale experiment conducted with unfocused microwave generators. Industrial scale microwave generators are generally more efficient than lab-scale units [36]. Additional factors such as plant capacity, sludge characteristics, energy requirement of the plant, disposal costs, and regulatory requirements are major factors that would affect the economic analyses [1,37]. To make microwave pretreatment incorporation to the system feasible, it is also important to use the waste heat that is produced after microwave heating [36].

Pretreatment costs vary considerably among different treatment processes. The microwave irradiation energy required per unit mass of sludge is lower than the energy required for sonication to attain the same degree of solubilization (Table 3). This fact alone demonstrates that microwave disintegration is a highly promising approach to enhancing the energy efficiency of sludge digestion.

Table 3
Comparison of sludge disintegration by microwave and ultrasonic pretreatments.

	Microwave[a]	Ultrasonic[b]
Sludge volume (l)	0.50	0.25
Total solid ($g\,l^{-1}$)	25.7	8.24
Irradiation time (min)	0–15	0–120
Instrumentation	2450 MHz, 700 W (magnetron)	20 kHz, 110 W (vibrator)
Specific energy ($kJ\,g\,TS^{-1}$)	0–49	0–288
Cell lysis (SCOD/COD)	0.02–0.22	0.005–0.21

[a] This study.
[b] [26].

4. Conclusions

SCOD and the concentration of protein, carbohydrate, lipid, and calcium in municipal secondary sludge pretreated by microwave irradiation were significantly higher than those in the raw sludge. This result indicates that microwave irradiation significantly increases sludge solubilization. Specifically, SCOD/COD increased from 0.02 to 0.22 as the microwave irradiation time was increased from 0 to 15 min. The soluble protein, carbohydrate, and lipid concentrations were also increased by microwave irradiation. Microwave irradiation increased the BAP of the sludge. However, the BAPs were similar in sludges pretreated for microwave irradiation times ranging from 5 to 15 min so that the optimum microwave irradiation time for BAP of sludge was 5 min in this study. Improved accessibility to organic substances also resulted in higher rates of VFA production with increasing irradiation times. Therefore, we suggest that microwave irradiation enhances both solubilization and the biodegradability of secondary sludge.

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