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Effects of Microwave Pretreatments on the Anaerobic Digestion of Food Industrial Sewage Sludge

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Published online 10 September 2010 in Wiley Online Library (wileyonlinelibrary.com). DOI 10.1002/ep.10487

Microwave irradiation is a novel and very promising technology for sludge conditioning. As pretreatment, it has a verified beneficial effect on the microbial degradation and anaerobic digestion of sewage sludge, but in present work we dealt with the applicability of microwave pretreatments for food industrial sludge. However, studies cannot be found that specialize on the effects of the MW treatments with different intensities on the anaerobic digestion of sludge. In our work we focused on the examination of the effect of MW pretreatment for 0.5, 2.5, and 5 W/g on the carbonaceous biochemical oxygen demand ($CBOD_5$), solubilization of organic matters (sCOD/tCOD), and the mesophilic anaerobic digestion of dairy sewage sludge. It can be concluded that the MW pretreatments were appropriate to enhance the efficiency of anaerobic digestion. With MW pretreatments the specific biogas product could be increased from 220 mL g^{-1} to more than 600 mL g^{-1} because of the increased solubility (from 9.7% to more than 40%), and the enhanced accessibility of organic compounds for decomposing bacteria. © 2010 American Institute of Chemical Engineers Environ Prog, 30: 486–492, 2011

Keywords: sewage sludge, microwave, anaerobic digestion, biogas, biodegradability

INTRODUCTION

Large scale development in the wastewater management technology has occurred in the past few decades. Hereby the cleaning efficiency of the purification processes has been notably improved, but the quantity and environmental risk of the final sludge also has increased. Generally, the solid phase of the wastewater is termed as sewage sludge, which has diverse composition that depends on the source and process of wastewater purification. Now, sludge represents the major solid waste from biological and physico-chemical wastewater treatment processes. In most cases the sludge handling system has been become the bottleneck of the capacity of waste water treatment plants. The anaerobic digestion of biosolids has become a popular stabilization and bio-energy generating process.

Theoretically, sewage sludge has a high potential for biogas production, but the rate and extent of anaerobic digestion was limited by the slow and incomplete disintegration of the complex flock structure of sludge formed by the extracellular polymeric substances (EPS) and the other nonbiodegradable components and toxic materials [1]. The composition of sludge depends on the type and original compounds of the raw wastewater; with the large specific surface it has a high adsorptive capacity for the heavy metals and other organic and inorganic pollutants. Extracellular polymeric substances are present in varying quantities in sewage sludge, occurring as highly hydrated capsules surrounding the cell wall and loose in solution as slimy polymers. It is able to retain a large volume of water within the sludge matrix by electrostatic interactions and hydrogen bounds [2]. The nonbiodegradable polymeric structure does not only originate from cell autolysis and sludge bacterial cell, but also from the compounds of raw wastewater. Therefore, besides the cationic content of the dosed chemicals and the composition of the organic materials, the efficiency and specific removal capacity of the applied waste water treatment technology (mechanical, chemical, biological, or combined) affect the biodegradability of sludge.

There are many possibilities to improve the digestibility and the aerobic biodegradability of sludge. The extent and rate of biological degradation can be enhanced by mechanical-, thermal-, ultrasound-, chemical-, thermo-chemical, and enzymatic pretreatment methods [3–6]. The treatments by oxidants are also suitable to disintegrate the sludge flock [7, 8]. Thermal treatments can alter the characteristics of sludge and transform a part of the suspended organic solids into soluble compounds [9, 10]. The result of the thermal hydrolysis of macromolecules amino acids, volatile acids, and simple sugars are produced, therefore a considerable increase of chemical oxygen demand (COD) can be experienced in the soluble phase [11, 12].

Microwave heating is used as a popular alternative to the conventional heating mainly due to the reduction of process time and the so-called nonthermal microwave effects [13–17]. Microwave digestion methods have been developed for different sample types such as environmental, biological, geological, and metallic materials [18, 19]. Due to the high water content, sewage sludge can absorb microwave energy efficiently. The microwave irradiation has thermal and athermal effects. The thermal effects can be attributed to heat generation in the matter due to the rotation of dipole molecules or ionic conduction; however, in many cases these two mechanisms have been experienced simultaneously. Although the quantum energy of microwave radiation is too low (1.05×10^{-5} eV) to break the chemical bounds, some structures can be altered by the energy carried by the microwaves [20, 21]. The intensive microwave heat generation and different dielectric properties of the cell wall components lead to a rapid disruption of the extracellular polymer network and residue cells of sludge [12, 14, 21]. After microwave pretreatments, the bound water can release into the intercellular liquor with the destruction of cell walls [22]. Moreover the biogas production of microwave pretreated sludge was higher than conventionally heated samples at the same temperature, because of increased volatile solid reduction and the enhanced COD solubilization [23–25].

Even though it is reported that the thermal and thermo-chemical pretreatments are suitable for enhancing the biogas yield of secondary municipal sludge, previous research has not been conducted to examine the efficiency of the applied specific microwave power level on the solubility and biodegradability of organic matter or to investigate the anaerobic digestion of food industrial sludge following MW treatments.

MATERIALS AND METHODS

Sludge Sample

The dairy sewage sludge sample originated from a wastewater treatment plant from a local dairy works (Szeged, Hungary). The thickened sludge was collected fresh from the settling tank after precipitation. The total solid content (TS), total chemical oxygen demand (tCOD), solubility of organic matters (given by sCOD/tCOD), volatile solid content (VS), and the carbonaceous biochemical oxygen demand for five days ($CBOD_5$) are given in Table 1.

Table 1. Properties of raw dairy sewage sludge.

TS [w/w%]	25.3 ± 7.21
pH	7.6 ± 0.3
tCOD [kg m^{-3}]	398.52 ± 7.21
$CBOD_5$ [kg m^{-3}]	25.55 ± 5.84
VS [kg m^{-3}]	191.29 ± 6.87
VS/TS [%]	9.32 ± 1.34
sCOD/tCOD [%]	9.66 ± 1.21
$CBOD_5$/tCOD [%]	6.39 ± 1.54
Biogas product [mL g^{-1}]	219.1 ± 4.34

Microwave Pretreatments

The microwave pretreatments were performed in a microwave cavity resonator. The power of maximum loaded magnetron is 700 W in inverter mode at 2450 MHz operating frequency, but the power output is continuously changeable (from 50 W to 700 W) by varying the heating voltage by a toroidal-core transformer. The intensity of microwave radiation was given by the applied specific microwave power level in W/g unit (magnetron power per quantity of dry matter content of sludge). The quantity of the irradiated sludge was 100 g in all tests. Sludge samples were placed in 15 mm layers into a low energy loss polytetrafluoroethylene (PTFE) vessel and a cover was applied to prevent evaporation during irradiation. As a comparison, conventional heating (CH) also was carried out in a temperature-controlled laboratory heating equipment (Medline CM, UK) at 95°C for 60 min.

Analytical Methods

The volatile solid (VS) content was gravimetrically measured via the residue of ignition at 550°C (APHA 2540E standard). The total solid (TS) content was measured by drying to constant weight at 105°C.

The extent of the solubility of the organic matter content of sludge was given by sCOD/tCOD ratio. The total chemical oxygen demand (tCOD) was measured triplicates, according to the dichromate standard method (5250D, APHA 1995). The soluble organic matter content (expressed by sCOD) was determined after centrifugation (6000 rpm, 20 min) from supernatant. For the separation of the water soluble phase of samples a 0.45 μm pore-size disc filter (Millipore) was used.

The carbonaceous biochemical oxygen demand ($CBOD_5$) measurements were carried out in a respirometric BOD system (BOD Oxidirect, Lovibond, Germany) at 20°C for five days. The supernatant phase of centrifuged and prefiltered samples was used for

CBOD$_5$ determination. The prepared samples were diluted with nutrient solution (K_2HPO_4, KH_2PO_4, Na_2HPO_4, $CaCl_2$, $FeCl_3$, NH_4Cl), using dilution factor 10. To avoid the oxygen consumption of the nitrification process, n-allylthiourea was added as a nitrification inhibitor. The BOD bottles were sealed by rubber sleeves containing KOH pellets as CO_2 absorber. To ensure the consistency of the experiments acclimatized standard BOD microbes (Cole Parmer, U.S.) were used as seed (in 1 w/w% concentration) for the measurements and the pH of all samples was adjusted to 7.2.

Anaerobic Digestion

The anaerobic digestion (AD) tests were carried out in continuously stirred lab scale reactors with 500 mL total volume, equipped with Oxitop C type barometric measuring heads under temperature controlled mesophilic conditions (35 ± 0.2°C) for 30 days. During our experiments the cumulative biogas production was measured triplicates in batch mode and the pH of samples was adjusted to 7.2. On the basis of our preliminary experiments the inoculums were added in 10 w/w% concentration to achieve the maximum biogas product. The inoculums were collected from an operating mesophilic biogas reactor of the local wastewater treatment plant WWTP (Szeged, Hungary) and they were acclimatized to the pretreated sludge to reduce the initial lag-period of the anaerobic process. The only inoculums containing bottles were used as a blank test. After inoculation, nitrogen gas was flowed through the reactor to prevent exposure to oxygen. For rapid estimation of the methane content in the produced gas mixture, the measurements were performed parallel in two vessels; one with absorber to measure the pressure without CO_2, and in the other digester the total gas pressure was measured. The results of the pressure difference method were validated after 30 days with gas chromatographic and mass spectrometric analysis (Agilent 6890N-5976 GC-MS).

RESULTS AND DISCUSSION

Our work investigated the effects of microwave irradiation with different power levels on the solubility of organic matters, the change of BOD, and the digestibility of dairy sewage sludge. Lab-scale batch fermentation tests were done to determine the mesophilic biogas and methane production. Since our object was to examine the efficiency of the microwave pretreatment, the process parameters of the anaerobic digestion (dry matter content of the fermentation broth, temperature, concentration of inoculums, pH, etc.) were constant in all tests.

In the first series of experiments, the microbial decomposition of the organic matter of sludge was investigated after MW pretreatments. The value of CBOD$_5$ gives information about the amount of the soluble and biodegradable organic components of sludge.

Compared with the untreated sample, the biochemical oxygen demand of the MW pretreated

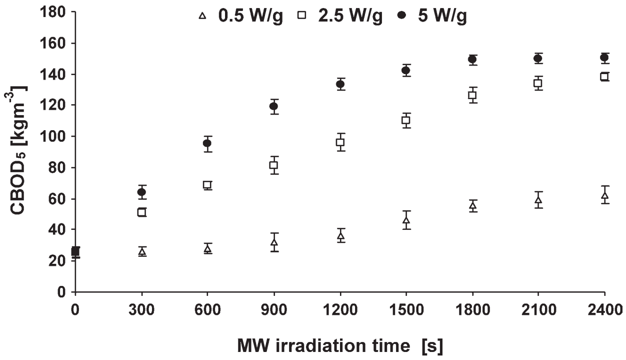

Figure 1. CBOD of MW pretreated sludge after 5 days incubation at 20°C.

sludge shows a considerable increment. Considering the CBOD$_5$ as a function of MW irradiation time, a near linear correlation was observed at the lowest specific power level (0.5 W/g) in the examined irradiation time period (300–2400 s), but at a higher specific MW power level, the connection can be described by saturation graph (see Figure 1). The large scale increment, such as from 25.2 to more than 150 kg m^{-3} after 5 W/g MW treatment, is due to the rapid and strong disintegration and predigestion effect of MW irradiation. It was reported earlier that the thermal and the MW pretreatments can disintegrate the flock structure and EPS of the municipal sludge; therefore the ability of organic matters for biological decomposition can be enhanced with higher solubility [21, 23, 26]. In our case, following a 60-min CH treatment, the CBOD$_5$ was enhanced to 41.8 kg m^{-3}, approximately a 64% increment related to the untreated sludge (Table 2).

To further study the large scale improvements in CBOD$_5$ and to verify the disintegration effect of MW treatments on food industrial sludge, the solubility of organic matters also was measured. Since the rate and extent of aerobic and anaerobic biodegradability are linked to the solubility of substrates, the sCOD/tCOD ratio also can be one of the main influential parameters of biogas production or in composting processes, for instance.

The positive effect of the CH pretreatment resulted in the higher organic matter solubility (sCOD/tCOD) and solid content volatilization (VS/TS), and the increments were approximately 38.8% and 9.2%, respectively (Table 2). The low original solubility of sludge (9.7%) can be explained by the complex flock structure of sludge. The enhancement of sCOD/tCOD after CH treatment was lower than the reported results of Bougrier *et al.* [26, 27]. They obtained an increment of nearly 1.8 times in soluble fraction of COD of municipal waste activated sludge, but in their case the heat treatment was executed in an autoclave, under pressure at 170 and 190°C, respectively. The change in the solubility of total solid content (VS/TS) at 170°C was higher than the increment of sCOD/tCOD, but the temperature increased from 170 to 190°C and the VS solubilization decreased because of the mineralization phenomenon.

Table 2. Results of CH pretreatment at 95°C for 60 min.

Parameter	Value	Increment*
$CBOD_5$ [kg m^{-3}]	41.8 ± 3.47	63.92
VS [kg m^{-3}]	206.9 ± 4.24	8.16
VS/TS [%]	10.18 ± 2.51	9.23
sCOD/tCOD [%]	13.4 ± 2.03	38.72
$CBOD_5$/tCOD [%]	10.54 ± 0.85	64.9
Biogas product [mL g^{-1}]	251.8 ± 3.79	14.92
Methane content [v/v%]	43.68 ± 4.16	15.86

*Relate to the untreated raw sludge.

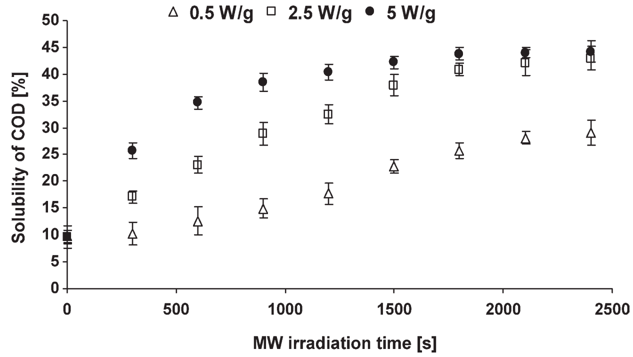

Figure 2. Solubilization effect of MW pretreatments.

Our data show that the MW pretreatments could efficiently increase the solubility of the organic matters of sludge. The solubility of COD could be increased more than 40% following MW irradiation at 2.5 and 5 W/g. Similar to the tendency of $CBOD_5$, the correlation of irradiation time and solubility has a saturation characteristic at the 2.5 and 5 W/g-specific power levels (see Figure 2). The increase of MW power level from 2.5 to 5 W/g was not profitable, because the longer MW "pre-digestion" caused no significant enhancement in the sCOD/tCOD ratio. The MW treatment increased the solubility with the enhanced flock disintegration but the MW irradiation with higher power level does not cause additional disintegration. Applying a higher MW power level is useful for reducing the process time.

Eskicioglu *et al.* [13] reported that the MW pretreatment has an advantage over the CH treatment by the solubilization of sugar content of waste activated sludge at 75°C. But the temperature increase (96°C) reduced the difference and a significant difference was not observed between the CH and MW treated samples. The decrease in the sugar and protein content in the soluble phase at an elevated temperature can be explained by the Maillard reactions occurring between amino acids and reducing sugars. In our case the sludge samples contain proteins with very low sugar compounds; therefore, the longer MW irradiation at higher temperatures could not manifest in a Maillard reaction with a lower sCOD/tCOD ratio. In an earlier study by Yu *et al.* [28], the particle size analysis verified the disintegration effect of MW irradiation when the contact time was longer than 60 s, and a correlation was found between solubilization and filterability.

During the anaerobic digestion of pretreated sludge, differences were observed not only in the total volume of biogas produced but also in the rate and initial lag-phase of decomposition.

Eskicioglu *et al.* [13] verified the advantage of MW irradiation over the CH treatment and reported the MW process as the highest biogas producing pretreatment method in the first 15-day digestion period, which produce approximately 16% more biogas than the untreated sample. During the second period of the AD process the difference in the biogas yield decreased between the different pretreated samples. The longer exponential phase of MW samples are explained by the higher concentrations of readily degradable substances present in the digesters.

In our work, as Figure 3 shows, the microwave irradiations with higher intensities (higher W/g values) caused a shorter initial lag-phase during anaerobic digestion and the initial rate of biogas production increased. The shorter overall time demand of maximum decomposition is due to the disintegration of the sludge flocks and the higher initial sCOD/COD ratio. Following the MW pretreatments the specific surface of sludge particles also increased; therefore, the organic substrate became not only readily but rapidly accessible for the decomposing bacteria and the adaptation of the anaerobic microorganisms in the first period of the AD was shortened and the rate of the anaerobic processes was higher.

Figure 4 represents the total biogas yield after 30 days of digestion for the control sample, CH and the MW pretreated sample (related to the COD consumed during anaerobic decomposition).

The results indicated that after CH pretreatment the volume of biogas produced increased to more than 250 mL g^{-1}COD_{cons}, and the total biogas production could reach 700 mL g^{-1}COD_{con} following the higher intensity and longer MW irradiation. The higher MW intensity and the longer irradiation time increased the biogas yield to a large extent but at the highest power level (5 W/g) following 2400 s irradiation no further significant increment was seen compared with the biogas product of the 1800 s irradiated sample.

Compared with the biogas yields observed in other studies [13, 21, 25], we measured notably higher increments but their data was related to the anaerobic digestion of municipal sludge with lower initial COD. There are some possible explanations in the work reported earlier on the higher biogas production of the MW-treated sample compared to the CH treated sludge. One explanation is that the MW damaged or inactivated cells undergoing anaerobic digestion would be more susceptible to further disintegration and lysis, which could result in a higher extent of biodegradation [29]. Because of the stronger flock disintegration and cell wall destruction the MW treatment could decrease the viscosity to a larger extent than the CH treatment. It is also reported that the viscosity determines the heat distribution and heat transfer properties and plays a

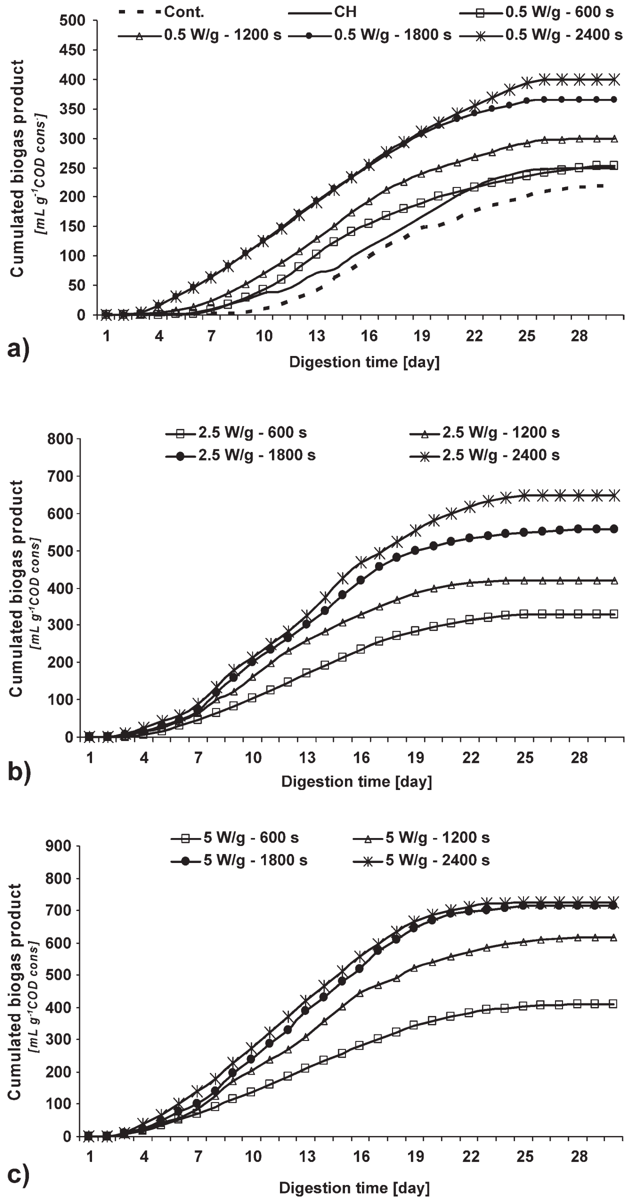

Figure 3. Cumulative biogas product of sludge samples. (a, 0.5 W/g; b, 2.5 W/g; c, 5 W/g pretreatments).

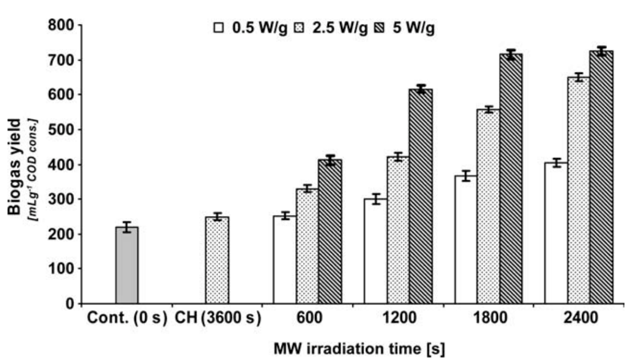

Figure 4. Biogas yield of pretreated dairy sludge. (CH, convective-heated sample).

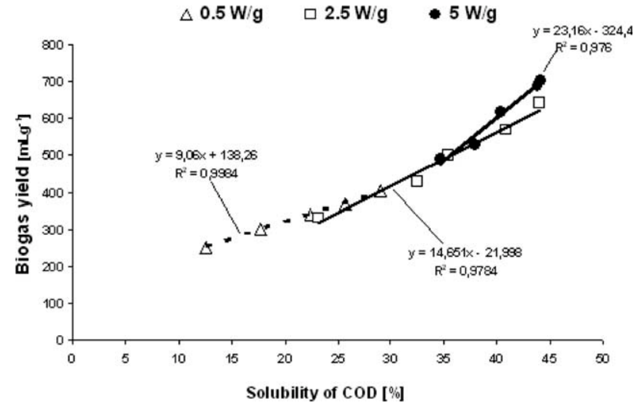

Figure 5. Correlation of solubility of organic matters and biogas yield.

significant role in the efficiency of the decomposition process of microbes [30, 31].

As the degree of anaerobic digestion is linked to the solubility of organic matters the connection between the solubility of COD and the biogas production also was examined.

Our data represent a linear correlation between biogas production and the solubility of the organic matters at all MW power levels. However, because of the stronger disintegration and degradation effect of higher MW power levels, the rates of fitted linear graph were different. In the case of MW pretreatments at 5 W/g the biogas production exceeds the specific biogas volume produced for 2.5 W/g, despite of the same solubility value (see Figure 5).

To further study the efficiency of MW pretreatment we calculated the total irradiated microwave energy (magnetron power $\times$ irradiation time) and presented the ratio of methane component in biogas. Since the same applied total MW energy can be reached with a different combination of the specific power level (W/g) and irradiation time, further information can be obtained about the effect of MW power levels on the efficiency of anaerobic digestion.

In general, the increased MW energy enhanced the methane ratio from ~37% to more than 60%. However, the applied specific MW power level also was an influential process parameter. It was found that the MW power level affects the methane ratio specifically by the lower range of total irradiated MW energy (see Figure 6).

In terms of the total biogas production, the higher MW power is beneficial. In spite of a higher biogas product, the methane ratio is limited; therefore, if MW pretreatment with high energy is used, the effect of lower MW power with the combination of a longer irradiation time will be similar to the effect of a higher power level with a shorter process time. However, from a practical approach the applied power level (given by the ratio of magnetron power and the mass of treated sample) can be useful to characterize the intensity of MW treatment. Further investigation is needed to determine the extent of the energy absorbed during microwave sludge treatments.

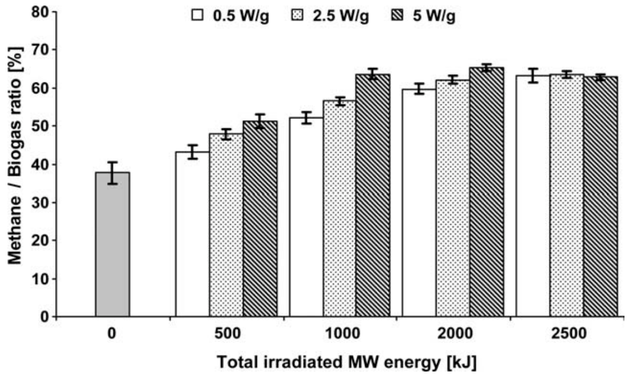

Figure 6. Methane content of biogas after pretreatments.

CONCLUSION

In this paper our aim was to examine the effect of MW pretreatment with different power levels on the COD solubilization, change of $CBOD_5$, and the anaerobic digestion of dairy sewage sludge. Our measurements show that the microwave pretreatment can be an effective method in sludge handling technologies. As a consequence of higher biological degradability and the increased solubility of organic matters of MW pretreated sludge, the biogas product was also enhanced. The main advantage of MW irradiation was the rapid pre-degradation effect, because it enhances the access to organic substrates for anaerobic microorganisms. Our experiments found that the solubility of organic compounds of dairy sludge could be increased from 9.2% to more than 40%, and the biogas production of MW pretreated sludge also was raised on a large scale (from 220 mL g^{-1} to more than 600 mL g^{-1}). Considering the change of sCOD/tCOD ratio, there was no notable difference between the MW irradiation with 2.5 and 5 W/g following 2000 s pretreatments. Among the tested conditions from the point of view of biogas production, the optimal specific power level and irradiation time for MW treatment of dairy sewage sludge was 5 W/g and 1800 s, respectively. Also, it was observed that besides the total MW irradiated energy, the specific microwave power level of pretreatments also affected the anaerobic digestion process.

ACKNOWLEDGMENTS

This work has been carried out with the financial support from the Hungarian Office for Research and Technology (NKTH) under contract the University of Szeged Regional University Knowledge Center for Environmental and Nanotechnology (RET-07/2005 Project).

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