



Influence of microwave pre-treatment on sludge solubilization and pilot scale semi-continuous anaerobic digestion

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HIGHLIGHTS

- ▶ The effects of a microwave pre-treatment on anaerobic digestion were studied.
- ▶ The microwave treatment resulted in a significant solubilization of organic matter.
- ▶ Volatile fatty acid concentration increased during the treatment.
- ▶ A 50% increase in biogas production was observed for a retention time of 20 days.

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ABSTRACT

Anaerobic digestion is widely applied for the recovery of energy from waste activated sludge. Pre-treatment methods are of high interest to increase the biodegradability of the sludge and to enhance the digestion efficiency. This paper studies the application of a microwave pre-treatment. An experimental set-up of two pilot scale semi-continuous digesters was used. During a long term experiment, one of the reactors was fed with untreated sludge, while microwave pre-treated sludge (336 kJ/kg sludge) was introduced in the second one. A solid retention time of 20 days was kept during the experiments. (Organic) dry solids, carbohydrates, proteins and volatile fatty acids were monitored during digestion. It was seen that the microwave pre-treatment resulted in an effective solubilization of the organic matter in the sludge. The changes to the sludge composition resulted in an increase in biogas production by 50%, while the methane concentration in both reactors remained stable.

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1. Introduction

The huge amounts of waste sludge that are annually generated in biological wastewater treatment plants represent a problem of growing importance. The production of excess sludge is an inevitable drawback inherent to the waste activated sludge system, and the expansion of the wastewater treatment infrastructure will lead to a further increase in sludge production in the future. Anaerobic digestion is generally recognized as a valuable step in the treatment of waste sludge, mainly because of the production of an energy rich biogas (55–70% CH₄), which can be valorized energetically (Appels et al., 2008). Other beneficial properties include the ability to reduce the overall load of sludge to be disposed, the stabilization of the sludge, the improvement of sludge dewaterability and the reduction of pathogens (Appels et al., 2011).

One of the main drawbacks of anaerobic digestion, however, is the rather slow degradation of the particulate organic matter, which leads to long residence times and/or low degradation effi-

ciencies. This is mainly attributed to the hydrolytic stage of the anaerobic digestion, which is considered to be rate limiting (Abelleira et al., 2012). In this sense, a lot of research attention has been paid to pre-treatment methods that cause a disintegration of the sludge (accompanied by the solubilization of organic material) and hence succeed in partially bypassing the hydrolytic stage and leading to a higher biogas production. Different techniques have been studied, including chemical (Dewil et al., 2007), mechanical (Zhang et al., 2012), ultrasonic (Bougrier et al., 2005), enzymatic (Barjenbruch and Kopplow, 2003) and thermal (Val del Río et al., 2011) treatments.

Microwave (MW) pre-treatment is an alternative method to conventional thermal pre-treatment and its potential has been well recognized in sludge treatment (Mudhoo and Sharma, 2011). Microwave irradiation is electromagnetic radiation with a wavelength between 1 mm and 1 m, corresponding to an oscillation frequency of 300–0.3 GHz (Eskicioglu et al., 2007a). Domestic “kitchen” microwave ovens and industrial microwave generators are generally operating at a frequency of 2.45 GHz with a corresponding wavelength of 12.24 cm and energy of 1.02×10^{-5} eV (Jones et al., 2002; Eskicioglu et al., 2007a; Tang et al., 2010).

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The mechanism of the microwave treatment consists of a thermal and an athermal (non-thermal) effect. The thermal effect is caused by the interaction of the oscillating electrical field with dipolar molecules like water, proteins, fats and other organic complexes. The resulting molecular rotation of the permanently and induced dipoles leads to friction which ultimately will result in heating the sludge (Dogan and Sanin, 2009; Eskicioglu et al., 2007a; Yu et al., 2010; Park et al., 2004).

The athermal effect is attributed to the rapidly changing dipole orientation in the polarized side chains of the cell membrane macromolecules, which results in the breakage of hydrogen bonds, subsequently leading to the disintegration of the floc matrix and changes in the secondary and tertiary structures of the proteins of the micro-organisms (Eskicioglu et al., 2007a; Tang et al., 2010; Park et al., 2004, 2010). Strength (frequency), concentration, radiation time and penetration depth are the main factors of microwave radiation that impact the dielectric material (Park et al. 2010). The effect is, however, contested since other studies did not find evidence for the presence of the athermal effect (Sólyom et al. 2011) or contradictory results were obtained.

Studies conducted so far were mainly focused on the enhancement of biogas production, sludge dewaterability and pathogen destruction, and the increase in biogas production was mostly assessed by batch digestion tests (biochemical methane potential, BMP) (Sólyom et al., 2011; Jin et al., 2009; Beszédes et al., 2011; Eskicioglu et al., 2007a).

Studies on semi-continuous digesters focused on low solid retention time (SRT) or the possible reduction of the retention time. There is an agreement that MW pre-treatment increases the biogas production, but varying levels of improvement have been found. Park et al. (2004), for instance, noticed that the improvement in biogas production was more pronounced at shorter SRT (increases from 24% to 36% when the SRT decreased from 15 to 10 days for a MW pre-treatment at 91 °C). This enhancement would enable to reduce the SRT from 15 to 10 days with no decrease in conversion efficiency. Eskicioglu et al. (2007b) on the other hand only noticed a 5% increase in biogas production for a SRT of 10 days for sludge MW pre-treated at 96 °C. At very low SRT, i.e. 5 days, a more significant increase of 28% was found.

Most research has been conducted on MW pre-treatments at temperatures lower than boiling point. The effects of a higher treatment temperature on sludge solubilization were studied by Toreci et al. (2008, 2009). Their work considers temperatures between 110 and 175 °C and they noticed considerably higher degrees of solubilization compared to other, low temperature studies. Also, a strong relationship between temperature and solubilization was noticed in the studied temperature domain, leading to the conclusion that treatment temperature is an important factor. In a study of Toreci et al. (2007), a mild inhibition was noticed in the early stage of biochemical methane production (BMP) tests, although the inoculum had been acclimatized with pre-treated sludge for 4 months. An increase of biogas production by 31% was observed for sludge pre-treated to 175 °C. Higher treatment temperatures are, however, more energy consuming, and an elevated pressure is necessary.

For more background information on the application of MW in sludge treatment, including the occurring mechanisms, the reader is referred to a recent review paper by Mudhoo and Sharma (2011).

The available literature results imply that there is a potential to increase biodegradability in full scale continuous digesters at shorter retention times. However, most sludge digesters work at longer retention times, and from a point of view to optimize the organic matter degradation during digestion, rather than to increase the solids throughput of a digester, it is interesting to see what effects a MW pre-treatment has on continuous digesters at longer retention times. The objective of this work is, therefore, to study

the effects of a low temperature MW pre-treatment on a semi-continuous pilot scale digestion for a hydraulic retention time of 20 days. (Organic) dry solids, carbohydrates, proteins and volatile fatty acids were followed during digestion. Also the effects of the MW pre-treatment on the solubilization of these classes of organics were assessed.

2. Methods

2.1. Microwave pre-treatment

Sludge samples were irradiated in a microwave oven (Sharp R-212) with a maximum power output of 800 W. For each treatment, 500 g of sludge were introduced in the MW oven, in a closed glass recipient (MW irradiated at 800 W for 3.5 min). The microwave energy applied to the samples for the semi-continuous tests was 336 kJ/kg sludge, leading to a final temperature in the sludge of 80 °C. After the treatment, the sludge was immediately cooled in an ice bath to room temperature prior to analysis and digestion. This was done for scientific reasons to ensure a controlled time during which the sludge was subjected to the elevated temperature. In practical applications, obviously, this should not be done since the longer the sludge is subjected to an increased temperature, the higher the increase in biogas production will be (Appels et al., 2010).

2.2. Semi-continuous anaerobic digestion

Two pilot scale (semi-)continuous digesters (50 L working volume each) were run in parallel for 42 days at mesophilic conditions (37 °C). Twice a week, sludge samples were taken from the buffer tank (thickened sludge) of the municipal wastewater treatment plant of Mechelen-Noord (Belgium) and used to feed the digesters. One digester was fed with blank (untreated) sludge, while the other received microwave pre-treated sludge. Unavoidably, the composition of the feed sludge varied during the 42-day digestion experiment. However, since working with two parallel reactors, all changes in the sludge fed to the blank reactor, were also present for the MW pre-treated digester, so a comparison between both digesters could be made. Two small (16 L) buffer tanks were available on the pilot scale installation, from which 1.25 L sludge was fed to the digesters every 12 h, leading to a hydraulic retention time (HRT) of 20 days. The associated organic loading rate (OLR) equaled 1.425 kg/m³ day, with a standard deviation of 0.27 kg/m³ day. The digesters were continuously mixed with a blade stirrer running at 20 rpm.

For the start-up of the digesters, the reactors were filled with digested sludge from the industrial scale digester of the WWTP of Antwerpen-Zuid (Belgium) and were fed during 40 days with untreated sludge in order to let them stabilize. After this period the experiment was started. During the digestion experiment, the produced biogas was measured using drum-type gas meters (Ritter). The methane content was relatively constant at 61 vol.%. Twice a week, both the digester feed and the effluent were sampled. Since perfect mixing is considered, the composition of the effluent equals the composition of the digestion mixture inside the digesters.

2.3. Measurements and analysis

The dry solid content is the ratio of the mass of sludge after drying at 105 °C and the original mass of the sludge sample. The dry solids consist of an inorganic and an organic part. The inorganic part is the part that is left after heat treatment at 605 °C. An empty porcelain crucible is weighed. This crucible is subsequently filled with sludge and again the crucible is weighed. It is placed in the

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