DETECTION OF SLUDGE DISINTEGRATION PROCESS
BY DIELECTRIC CONSTANT MEASUREMENT

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Abstract
Microwave irradiation has a good potential for sludge pre-treatment. Several studies concluded that microwave pre-treatments increase the disintegration degree and biogas production of waste activated sludge. However, dielectric parameters are not commonly measured for wastewater and sludge, especially for sludge produced in food industry. Moreover, the efficiency of continuously flow microwave sludge conditioning process has not been analyzed in details. Therefore our research aimed to examine the disintegration efficiency of the microwave treatments for sludge originated from food industry carried out in continuously flow reactor. Another aspect of research was to investigate the relationship between the dielectric parameters and the change of disintegration degree.

Introduction
Because of environmental aspects and the need for energy sludge utilization by biological method, such as anaerobic digestion, has been become more and more popular. Wastewater sludge has theoretically good biodegradability. But in practice, because of the flocculants dosed in wastewater treatment technology, or disinfection agents used in food industry together with organic macromolecular components form a complex sludge structure resistant to direct biological degradation. Therefore an appropriate pre-treatments stage is need before further biological utilization of sludge.

There are known several pre-treatments methods. Among them the thermal process are widely used because of their good controllability and flexibility for varying quality and quantity of sludge production. Microwave (MW) irradiation is considered as more effective compare to the conventional thermal processes. MW pretreatments are suitable to increase the organic matter solubility and, therefore, the bioavailability of substrate. This strong disintegration effects of MW irradiation can manifested in higher biogas production, for instance [1] [2]. Considering the promising results related to microwave treatment for municipal sludge further analysis in needed to investigate the applicability of the method for other types of sludge. Beside this, further examination is needed to determine the dielectric parameters of sludge exposed to MW irradiation.

Dielectric polarization and ionic conduction play role in the heat generation mechanisms of microwave irradiation. At microwaves frequencies the polarity of electromagnetic field vary with high frequency, therefore the ionic and dipolar molecules are moving in time. If molecules with dipolar characteristics lag behind the electromagnetic field heat is generated in the materiel, due to the energy dissipation. In high water contented systems the dipole movement is influenced by the strength of hydrogen bonds. For free water, the movement is occurred in the gigahertz frequency range. Dipolar movement is the dominant mechanism for bound water in the megahertz frequency range and for ice at kilohertz frequencies [3].
In order to assess the energetic efficiency of sludge treatment by microwave irradiation investigation of dielectric behavior of processed material is needed. Moreover, deeper analysis of dielectric behavior is necessitated if dielectric parameters are assumed to vary during MW pre-treatments due to thermal effects and/or structural and physicochemical changes. Dielectric constant and dielectric loss factor determined the penetration depth of electromagnetic waves into the materials, as well. In high water contented materials, such as sludge, the moisture content and the physicochemical state of water has a dominant effect of dielectric behavior [4]. During thermal and thermochemical pre-treatments partial hydrolysis of macromolecules is also occurred. Decomposition of high molecular weight components into smaller molecules, especially if it produces compounds with polar characteristics or led to increase of the concentration of ionic components with high migration ability. Increasing of polar characteristic and amount of ionic components can have effect on dielectric behaviors of processed materials. Measurement of apparent dielectric parameters enable to the fast determination of moisture content during dehydration of agricultural and food product [5] and composting process [6]. Main advantages of dielectric measurements over the conventional methods are the fast detection and the possibility for in-situ and non-destructive measurements. By dielectric technique the ripening of apple [7], maturation of meat [8] is controllable.

**Experimental**

For the experiments primary sludge produced in the wastewater purification line of a meat processing work was used. Volatile solid (VS) and total solid (TS) content of sludge was and 6560 ± 281 mgL\(^{-1}\) and 53 ± 3.5 gL\(^{-1}\), respectively. Total chemical oxygen demand (TCOD) and COD of soluble phase (SCOD) was 28300 ± 690 mgL\(^{-1}\) and 3250 ± 212 mgL\(^{-1}\). Microwave treatments were carried out a custom made continuously flow microwave unit equipped magnetron with a frequency of 2450 MHz. Sludge was pumped through the toroidal cavity resonator by a peristaltic pump. Volumetric flow rate was varied in the range of 6-35 Lh\(^{-1}\).

Microwave related process parameters for sludge pre-treatments was the microwave power level (W) and the specific microwave energy intensity (Es, Jg\(^{-1}\)). Es was given from the power of magnetron (P), the volumetric flow rate (Q\(_v\)), the quantity of sludge in microwave reactor (m) and the effective volume of PTFE spiral in the cavity resonator (V).

\[
E_s = \frac{p}{m Q_v V} \quad [Jg^{-1}] 
\]

(1)

For the MW-alkali pre-treatment NaOH dosage was applied. The dosage was given in g\(_{NaOH}/g_{TS}\) unit. Structural and chemical change of sludge was characterized by the disintegration degree (DD). DD was calculated from soluble chemical oxygen demand measured from the raw (SCOD\(_0\)) a pre-treated sludge (SCOD\(_t\)) and the COD of the total sludge matrix (TCOD).

\[
DD = \frac{SCOD_t - SCOD_0}{TCOD - SCOD_0} \times 100 \quad [%] 
\]

(2)

COD was measured photometrical method using special COD test cuvettes containing potassium dichromate. Soluble phase was separated from particulate organic matters by centrifugation (10000 rpm for 10 minutes) and pre-filtration (0.45 μm).
Dielectric constant ($\varepsilon'$) was measured by a special designed, tailor made dielectrometer. For dielectric measurements the same frequency -2450 MHz- was used that of applied for microwave pre-treatment of sludge. Diode power sensors were positioned in a distance of $\lambda/8$ to detect the perturbation of waveform and form of standing waves. Power was measured by a dual channel power meter (NRVZ, Rohde&Schwarz). From the voltage standing wave ratio the reflection coefficient and phase shift was calculated. From the geometry of waveguide and positioning of sample holder with the phase shift and reflection coefficient the dielectric constant was derived.

Results and discussion

Effects of microwave-alkaline treatment on sludge disintegration was characterized by DD. Alkaline dosage and irradiated microwave energy - as control parameters - have significant effects on DD. Results of ANOVA show that linear and quadratic terms and interaction of variables can be considered significant at the level of 95%. Increasing of $E_s$ and alkali addition increased the DD, but over certain values of energy intensity and NaOH dosage disintegration start to decrease (Fig. 1.). Initial DD of meat processing sludge was 11%, maximum achievable DD was 27.2% if alkaline dosage and irradiated microwave energy was in the range of 0.35-0.55 g$_{NaOH}$/g$_{TS}$, and 6000-8000 Jg$^{-1}$, respectively.

![Fig.1. Effect of alkaline dosage and $E_s$ on DD](image)

Our results verified, that microwave irradiation combined with alkaline dosage was suitable to increase the disintegration degree of meat processing wastewater sludge. During the microwave heating under alkaline condition the sludge particles are break up, and the macromolecular components are partially hydrolyzed [1]. These effects are manifested in higher solubility of organic matters, which was detected by SCOD measurements.

Physicochemical change of sludge structure can provoke the change of dielectric parameters. Our results verified that dielectric constant of microwave-alkaline pre-treated sludge has been changed and the tendency of the change of dielectric behaviors was similar that obtained for DD (Fig. 2.). In order to investigate the relationship between the dielectric behavior and disintegration process dielectric constant ($\varepsilon'$) was depicted as a function of DD. It was found that in the DD range of 13-28% a strong linear correlation was found between the DD and dielectric constant (Fig. 3.a.).
To test the validity of dielectric measurements for detection of the change of DD, in the next stage of experiments measurements were carried out for freeze stored sludge sample, as well. Independently from the freezing-thawing process the linear connection of dielectric constant with DD has been also revealed (Fig.3.b). Disintegration of sludge particles increase the ratio of free to bounded water content. Increasing of free water with higher smaller molecular weighted components produced during thermochemical treatment of sludge enhances the orientation ability of compounds in high frequency electromagnetic field [4]. Enhanced polarization ability was detected by the change of dielectric constant.

**Conclusion**

Efficiency of microwave pre-treatment for municipal waste activated sludge was verified in numerous studies. But suitability of microwave treatments for sludge produced in food industry wastewater purification is not investigated in details. Generalization of experimental results and scale-up of microwave sludge pre-treatment for pilot and industrial system has difficulties, mainly the experiences from continuously flow microwave sludge treatment
process is missing. Further investigations are needed to analyze the energetic efficiency of microwave irradiation. Energy need and payback period of microwave sludge treatment is depended by several factors. From the aspects of material characteristic, the composition and dielectric parameters determined mainly the energetic efficiency. Sludge has high water content and, therefore, good microwave power dissipation. But dielectric parameters of sludge have not been investigated deeply, moreover, during sludge treatments the dielectric behavior can be changed due to the physicochemical changes of sludge.

Therefore, in our work the applicability of the continuously flow microwave treatment combined with alkaline addition was investigated for meat processing wastewater sludge. Our results verified, that microwave irradiation was suitable to increase the disintegration degree of sludge.

Enhanced disintegration can manifest in higher concentration of soluble organic matter content. Higher bioavailability of soluble organic substrate provoke that efficiency of a subsequent biological degradation process can be increased, as well. Higher biodegradability is suitable to intensify the controlled biological utilization of sludge, in composting or anaerobic digestion process, respectively. Improvement of biogas yield provoked by the microwave pre-treatment could led to enhanced energetic efficiency and shortened overall payback period of sludge pretreatment-digestion technology.

Another aspect of our research was to determine the dielectric constant for meat processing sludge. Our results verified that dielectric behavior was influenced by the disintegration efficiency of microwave-alkaline treatment. Dielectric constant has a good linear correlation with disintegration degree in the investigated range. Therefore dielectric measurements can be considered suitable to the fast, reliable and non-destructive detection of the change of organic matter solubility in laboratory scale batch and continuously flow process and, however, for in-line and real time characterization of the disintegration efficiency of sludge pre-treatments.

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References