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# OPTIMISATION OF MICROWAVE CONDITIONING PROCESS FOR ENHANCED SLUDGE VALORISATION

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## ABSTRACT

Sludge produced from food industry wastewater contains organic matters in a high concentration but additional components such as detergents, disinfectants and other macromolecules and ions originating from wastewater purification processes have unfavorably effect on biodegradability of sludge. Microwave irradiation is considered as intensive method for pasteurization and also provides fast thermal technology to convert organic matters into more soluble form and helps to disrupt the resistant polymeric structure of sludge. Beside the many verified beneficial effect of microwave technique on sludge structure conflicting results can be found in the scientific studies.

In our study was focused on to define appropriate process and control parameters for microwave sludge conditioning methods which are suitable to optimize the microwave operation and also could provide facility to develop continuously-flow microwave conditioning equipments for food industry sludge. During our work novel indicators, the solubilization index and biodegradation index were developed to control the efficacy of microwave process. Our experimental results confirmed that beside the irradiation time and frequency the specific microwave power level (MWPL) has also effect on the biodegradability of food industry wastewater sludge. Results of response surface analysis show that increment of biodegradability was limited. Over the optimum value of irradiated energy or higher intensity of MW treatment BDI was decreased, and the energy balance of the subsequent anaerobic digestion process was also worsened.

## 1. INTRODUCTION

One of the main environmental problems of food industry sector is to ensure efficient wastewater purification technology. Capacity of sludge management technology has become the main influential factor for the overall economy of wastewater treatment systems. Food industry wastewater contains a huge amount of organic matters; therefore in sludge, originating from it, the dosed cations in the precipitation stage of wastewater line altogether with living microorganisms and organic molecules form extracellular polymeric substances (EPS). Different species of microorganisms, biomass produced by the degradation of grease, nitrogen, and phosphorus; heavy metals and synthetic organic compounds agglomerated together with EPS into the polymeric network of sludge. Furthermore, EPS fraction of sludge causes hydrophilic characteristic of sludge, and increases the difficulty achieving effective bioconversion during anaerobic digestion (AD) or aerobic biotransformation processes, e.g. composting.

The key issue of technological development for sludge processing and the main focus area of R&D activity is the organic matter valorization in biological waste handling technologies. Professionals in food industry companies face the high disposal costs of bio-wastes and the rising price of energy sources. In the case of bio-wastes such as wastewater sludge, and other the solid and liquid residues of food processing one of the possible solutions is the biofuel production, such as bioethanol or biogas. Beyond the volume reduction, keeping in mind the cost minimizing, the conditioning and pre-treatments may

also have requirement to modify the structure of processed wastes before further utilization.

Several sludge conditioning methods were developed and investigated to improve the accessibility of organic matter for decomposing bacteria, and to enhance biogas production. Moreover, many alternative methods such as sonication, advanced oxidation processes (AOP's), freezing, electrolysis, and thermal pretreatments have been investigated to improve the dehydrate capability and degree of disintegration of sludge. The other main aim of various pre-treatments method is to enhance the anaerobiosis of sludge and to increase the rate of AD process (Bougrier et al., 2008).

Microwave irradiation is considered as intensive method for pasteurization and also provides fast thermal technology to convert organic matters into more soluble form. Microwave irradiation has also been successfully applied in many fields of organic chemistry, biotechnology and environmental engineering. One of the most important key issue before the scale-up of microwave process is to make more homogenous the electric field in the heterogeneous systems (Géczi and Sembery, 2009).

Thermal and a-thermal effects of the microwave (MW) irradiation play role in the "hot-spot" overheating phenomena, and the different dielectric parameter of cell components led to selective heating manifested in the different thermal stress, which contributes in the intensive degradation of cell wall components. MW pre-treatment alone has verified positive effects on cell wall destruction and releasing of organic matter into the soluble phase (Cheng et al., 2011). Results of diffuse infrared spectrum reflectance methods verified, that MW irradiation increases the molecular motion in the structure of polysaccharides and polarize the macromolecules resulting in the increasing of the decomposition rate. MW irradiation has been successfully adopted as pre-treatment method via the high energy dissipation of polar compounds of sludge. There are several studies concluding MW method advantages over the pre-treatment process operating by conventional heating (Toreci et al., 2009). Increasing effect of MW irradiation on anaerobic digestion of organic solid waste was found, considering the substrate availability a 78% improvement was achieved after MW pre-treatment (Shahriari et al., 2012).

After MW pre-treatment alone an increment of 79% was found in biogas production. MW irradiation of the sludge containing  $H_2O_2$  as oxidizer agent could accelerate the decomposition of it into hydroxyl radicals which is manifested in the enhanced organic matter solubilization and increased disintegration rate of sludge flock, as well (Eskicioglu et al., 2009). Synergetic effects of alkaline treatment in combination with MW irradiation resulted in enhanced organic matter solubilization. In the work of Yu et al. (2010) reported MW treatment to enhance the settling velocity of secondary municipal sludge. Increasing of irradiation time and MW energy to a certain the sludge particles are destabilized therefore particle size was become smaller.

However, effects of MW process parameters on the physico-chemical properties and structure of sludge are controversial. Some studies suggested the high power MW irradiation for enhanced organic matter solubilization and disintegration of sludge particles (Climent et al., 2007), other authors concluded lower solubilization after high intensity MW irradiation (Park et al., 2010). Energy transfer carried by microwaves affect the biodegradability in two ways. Thermal effect is expressed in the increase of internal pressure of intracellular liquor caused by internal heating and rapid evaporation, that altogether can lead to cell wall disruption. The non-thermal effect of high frequency electromagnetic field is contributed to alter the structure of macromolecules with polarization of side chains and breaking of hydrogen bounds (Park et al., 2010).

Over the last decade many reports can be found investigating the effects of MW irradiation on the sludge disintegration and anaerobiosis, but in very few of them were examined food industry wastewater sludge. There can be found studies reporting conflicting results regard to the optimal parameters of MW pre-treatments. Furthermore, the very few experience regarding to continuous-flow operating equipment make difficulty the scale-up of MW processes. In our earlier work was verified that beside the applied microwave power and the irradiation time the specific intensity of irradiation has also effect on the cumulative biogas product and the kinetic of anaerobic decomposition (Beszédes et al., 2011). But increasing of irradiated energy and also the specific intensity has limited positive effect on the degree of biodegradation, therefore the optimization of process parameters are needed. Taking into consideration of above mentioned aspects, in our present work we focused on the investigation of microwave conditioning method for food industry sludge with defining appropriate control parameters to quantify the change in biogas production influenced by the degree of anaerobic digestion. Our second objective was to optimize the MW process parameters such as irradiated MW energy (IMWE) and intensity of MW pre-treatment characterized by the specific MW power level (MWPL).

## 2. MATERIALS AND METHODS

### 2.1. Sludge sample

Thickened meat processing wastewater sludge (MPWS) came from the sedimentation tank of wastewater purification line of a local meat processing factory. Wastewater was mainly originated from flushing and cleaning operations of slaughterhouse processes, meat cutting and sausage making, and it was purified by mechanical and chemical precipitation processes. Samples were taken from equalized sludge of one week period, collected samples was stored in closed polyethylene bags at 4°C to avoid the change in sludge structure during the freezing and thawing procedure. The basic characteristics of the raw MPWS are summarized in Table 1.

*Table 1. Main characteristic of raw MPWS*

TS [ww <sup>-1</sup> %]	TCOD [kgm <sup>-3</sup> ]	SCOD [kgm <sup>-3</sup> ]	pCOD [kgm <sup>-3</sup> ]	BOD <sub>5</sub> [kgm <sup>-3</sup> ]	SCOD/TCOD [%]	BOD/TCOD [%]	BOD/SCOD [%]
17.3±1.9	539.8±7.6	101.3±7.3	438.6±7.1	31.39±2.1	18.76±1.1	5.82±0.6	30.9±1.3

### 2.2. Microwave treatments, AD tests and analytical procedures

MW pretreatments were performed in a tailor-made microwave system containing continuously irradiating magnetron with changeable power in the range of 50 W to 700 W operating at a frequency of 2450 MHz. To minimize the energy loss of sample holder polytetrafluoroethylene (PTFE) container was applied for MW treatments. To investigate the efficiency of MW pretreatment the process parameters studied were the microwave power level (MWPL), and the irradiated microwave energy (IMWE). MWPL (Wg<sup>-1</sup>) was defined as the ratio of magnetron power to the quantity of treated sludge. IMWE (kJ) was calculated by the magnetron power and the irradiation time.

The sludge samples were analyzed for total solid content (TS), total and soluble chemical oxygen demand (TCOD and SCOD) and five days biochemical oxygen demand (BOD<sub>5</sub>). Total solid content (TS) was measured by drying to constant rate at 105°C. TCOD

from the total MPWS matrix was measured triplicate using colorimetric standard method (APHA, 2005). SCOD was determined after separation of soluble from solid fraction by centrifugation (12,000 rpm for 10 minutes) and prefiltration (0.45  $\mu$ m Millipore disc filter). Biochemical oxygen demand (BOD) tests were carried out in a respirometric BOD system (BOD Oxidirect, Lovibond, Germany) at 20°C for five days with dosing of n-allylthiourea in a concentration of 12  $\mu$ mol L<sup>-1</sup> to avoid the nitrification process.

The anaerobic digestion (AD) tests were carried out in continuously stirred anaerobic reactors with 500 mL total volume, equipped with Oxitop C type barometric measuring heads under temperature controlled mesophilic conditions (35  $\pm$  0.2°C) for 30 days. The inoculums were collected from an operating mesophilic biogas reactor of the local wastewater treatment plant acclimatized to the pre-treated sludge. Methane concentration in biogas was measured by Airtox biogas analyzer (Fresenius Umwelttechnik GmbH., Germany). In order to examine the energy efficiency of MW process the net energy products of pre-treated MPWS samples were also calculated by the following equation.

$$\Delta E = (q_{comb} \times V_{methane}) - (P_{magnetron} \times \tau_{irradiation})$$

In the calculations, 40 MJ·(Nm)<sup>-3</sup> average volumetric energy content for methane was used; the magnetron power varied by the intensity of irradiation.

### 3. RESULTS AD DISCUSSION

Our experimental data show, that MW pre-treatment was suitable to enhance the solubility of organic matters and the biodegradability, as well. The higher the MWPL or IMWE the higher the solubility, but the more intensive MW pre-treatment caused a decreasing in the biodegradability overt a certain value. Response surface modeling (RSM) and optimization of MW sludge conditioning process was carried out to find optimal process parameters for maximum biodegradability. In the case of BDI constructed contour plot indicate the optimum region for maximum biodegradability in IMWE range of 550-700 kJ, and MWPL range of 1.75-3.5 Wg<sup>-1</sup>, respectively (Fig.1.).

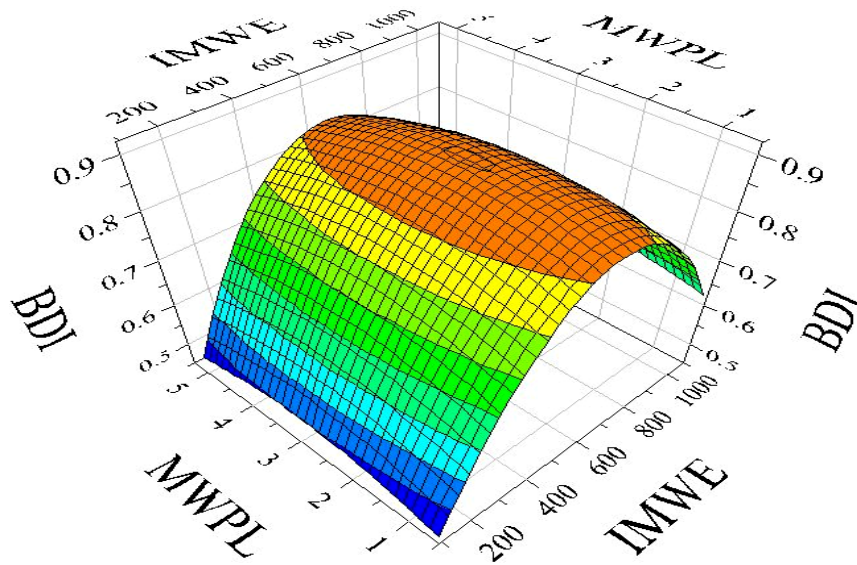


Figure 1. Response surface for BDI

In the study of Toreci et al. (2009) was concluded that increasing of MW power led to higher biogas production from secondary municipal sludge. In our case, MW pre-treatments increased also the cumulative biogas product from MPWS, but the worsening effect of high MWPL over a certain value of IMWE was also experienced, similar to the tendency of the change in BDI.

Compare to the untreated control sample the MW irradiated MPWS produced higher volume of biogas. Our results show, that both IMWE and MWPL affect the efficiency of the AD process, as well.

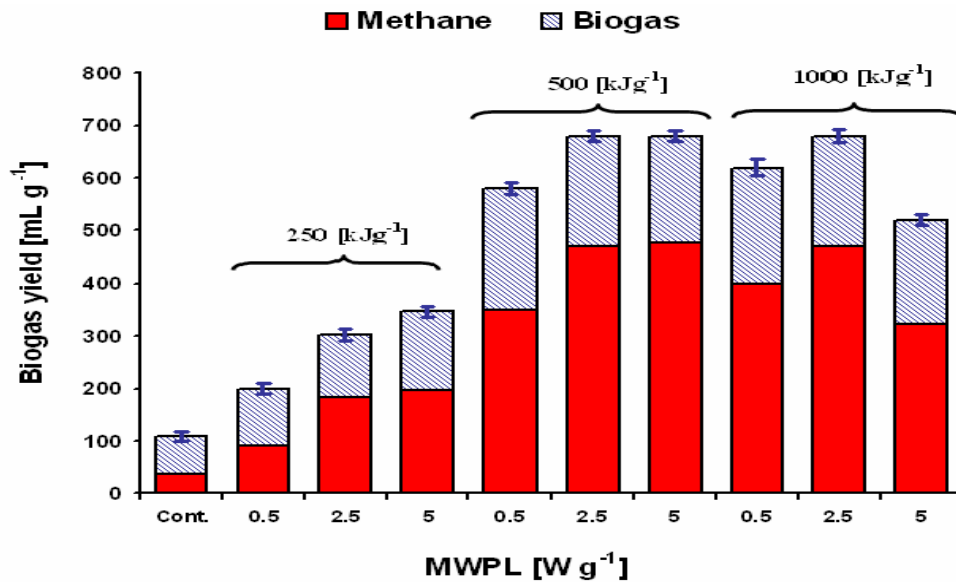
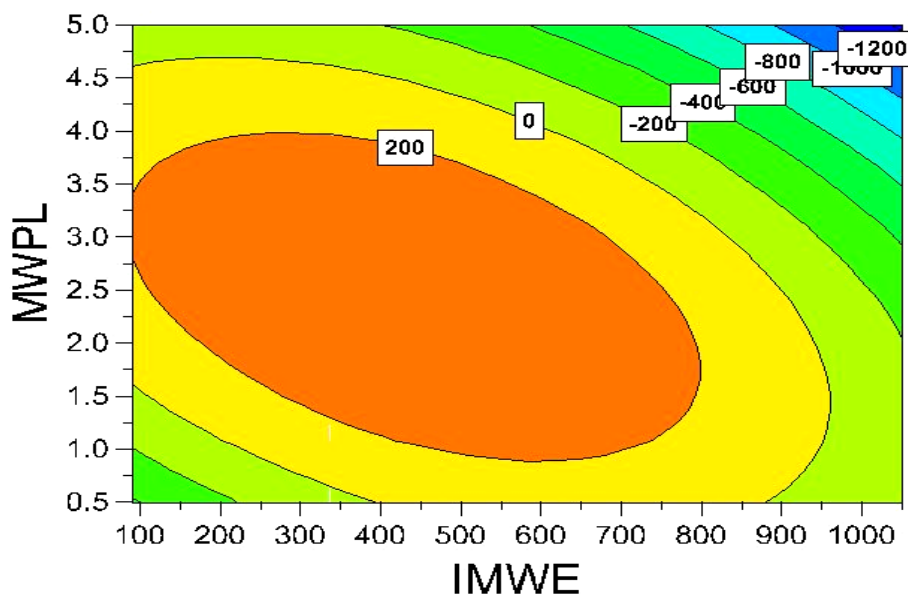


Figure 2. Biogas and methane yield of MPWS after MW pre-treatments

Similar to the investigation of SLI and BDI, RSM was used for modeling and energy optimization of MW process. Fitted mathematical model to predict  $\Delta E$  was given by the following equation:

$$\Delta E = 326.12 - 27.29 X_1 - 237.74 X_2 - 4.017 X_1^2 - 470.79 X_2^2 - 385.9 X_1 X_2$$

Results of our calculations show, that despite of higher biogas yield the MW pre-treatment at higher MWPL ( $X_2$ ) and using increased IMWE ( $X_1$ ) were not favorable from energetically aspects (Fig. 3.).



*Figure 3. Contour plot for  $\Delta E$*

Over a certain value of MWPL and IMWE the surplus of biogas production could not compensate the higher energy demand of the more intensive MW process. Considering the option to vary the operating parameter of MW pre-treatment optimum range of IMWE and MWPL was concluded as 600-650 kJ and 2.5-3.0 Wg<sup>-1</sup>, respectively.

#### 4. CONCLUSION

In our present work the effects of MW irradiation on the biodegradability of MPWS, furthermore the energy efficiency of MW pre-treatment followed by AD were examined. Investigated operating parameters were the irradiated microwave energy (IMWE) and microwave power level (MWPL). The net energy product ( $\Delta E$ ) of biogas production from microwave irradiated sludge was defined as control parameter.

Our results show that MW irradiation is suitable method for conditioning of food industry sludge. Effect of MW pre-treatments on the solubilization and biodegradation was influenced by both the irradiated MW energy (IMWE) and the specific MW power level (MWPL). Increasing of IMWE and MWPL has a positive effect on biodegradability, but over a certain value of them the value of BDI was worsened.

Results of optimization based on the constructed mathematical models obtained from response surface analysis show that the optimal condition of MW process of MPSS for highest biogas product with minimum irradiated energy and lowest MWPL were determined as IMWE range of 600-650 kJ at MWPL of 2.5-3.0 Wg<sup>-1</sup>.

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