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E-mail: <u>synergy@gek.szie.hu</u>

CONTENTS

I. Section: AGRIFOOD ECONOMY AND LOGISTICS

L. Magó

SHIFT HOURS DEMAND OF LOGISTICAL TASKS IN CUCUMBER PRODUCTION TECHNOLOGY (NO1-2-029)

H. Adanacioglu and N. Adanacioglu <u>USE OF DIRECT MARKETING STRATEGIES BY FARMERS IN IZMIR, TURKEY: A CASE STUDY OF</u> <u>ARTICHOKE GROWERS (NO1-4-059)</u>

I. Husti, M. Daróczi, I. Kovács and K. Béres <u>STRIP TILL: AN ECONOMIC ALTERNATIVE FOR THE HUNGARIAN AGRICULTURE (NO1-5-071)</u>

I. Rudnák and J. Garamvölgyi <u>HUNGARY'S YOUNG, TECHNICALLY EDUCATED WORKFORCE ENGAGED IN WORKING</u> <u>ABROAD (NO1-6-078)</u>

V. Medina, R. Tóth, M. Daróczi and Á. Bak <u>THE IMPACT OF THE SUPPORTS TO THE SEASONALITY OF THE NEW TRACTOR MARKET IN</u> <u>HUNGARY (NO1-7-217)</u>

II. Section: EMERGING ENERGY RESOURCES

R. Kicsiny

SIMPLIFIED MULTIPLE LINEAR REGRESSION BASED MODEL FOR SOLAR COLLECTORS (NO2-2-066)

I. Seres and P. Víg

REALIZATION THE VOLUMETRIC FLOW CONTROL OF A DOMESTIC SIZE SOLAR COLLECTOR SYSTEM (NO2-3-069)

P. Víg and I. Seres EXAMINATION THE EFFECTIVENESS OF FLOW CONTROL IN A SOLAR SYSTEM (NO2-4-070)

Z Bártfai, L Tóth, I Oldal, I Szabó, J. Beke, N.Schrempf, Z.Blahunka <u>MODELLING THE STIRRING PROCESS OF BIOGAS PLANTS USING MIXED MATERIALS (NO2-5-</u> <u>082)</u>

III. Section: ENVIRONMENTAL TECHNOLOGIES

S. Beszédes, P. Veszelovszki, L. Ludányi, G. Keszthelyi-Szabó and C. Hodúr <u>DEVELOPMENT OF BIODEGRADABILITY INDICATORS FOR MICROWAVE SLUDGE</u> <u>CONDITIONING PROCESS (NO3-1-022)</u>

G. Vizi

<u>COMPARING EXAMINATION OF ELECTROMAGNETIC FIELD LEVELS IN DOWNTOWN</u> <u>APARTMENT HOUSES WITH FLATS IN HOUSING ESTATES (NO3-4-075)</u>

IV. Section: FUNDAMENTAL RESEARCH

M.Gharibkhani, R. C.Akdeniz

DETERMINATION OF SOME SHAPE FACTORS OF FISH AND LARVAE FEED USING DIGITAL IMAGE PROCESSING METHOD (NO4-1-007)

A. Bablena

MODELING THE SOIL-WHEEL INTERACTION WITH DISCRETE ELEMENT METHOD (NO4-2-016)

K. Orova, K. Petróczki, J. Beke <u>MECHANIZATION IN GREENHOUSE TOMATO CULTURE AND INDOOR CULTURE FOR RESEARCH</u> <u>PURPOSES (NO4-3-040)</u>

F. Safranyik

DETERMINATION OF MICROMECHANICAL PARAMETERS OF GRANULARS BASED ON STANDARD SHEAR TEST (NO4-4-046)

Gy. K. Nagy BRACING ZONOHEDRA WITH ARTICULATED FACES (ZAF) (NO4-6-076)

V. Section: AGRICULTURAL MECHANISATION AND FOOD ENGINEERING

M. Gündüz, N. Korkmaz and Ş. Aşık <u>EFFECTS OF IRRIGATION WITH DOMESTIC TREATED WASTEWATER ON THE SOME CHEMICAL</u> <u>PROPERTIES OF SOIL EXTRACT (NO5-1-004)</u>

F. Weigler, H. Scaar, G. Franke and J. Mellmann DEVELOPMENT OF A NOVEL MIXED FLOW DRYER DESIGN (NO5-2-006)

Gy. Csima, Zs. Varga, G. Ficzek, I. G. Gyökös and Z. Láng <u>COMPARISON OF GALA MUST APPLE QUALITY GROWN ON DIFFERENT TREE TRELLISES (NO5-</u> <u>3-012)</u>

Z. Gobor, H. Heuberger and R. Rinder <u>PRECISION PLOT SEEDER FOR MEDICINAL AND AROMATIC PLANTS - CONCEPT, DEVELOPMENT</u> <u>AND OPTIMISATION (NO5-4-028)</u>

Cs. Farkas, K. Petróczki, L. Fenyvesi <u>METHOD FOR MEASURING FRUIT FAILURE CAUSED BY DIFFERENT MECHANICAL LOADS NO5-</u> <u>5-057</u>

VI. Section: MULTIDISCIPLINARY MODELING

H. Scaar, F. Weigler, J. Mellmann, K. Gottschalk, Á. Bálint, Cs. Mészáros, I. Farkas <u>NUMERIC SIMULATION OF HEAT AND MASS TRANSPORT IN SOIL SAMPLES (NO6-1-008)</u>

R. Kosztolányi, Á. Bálint, K. Gottschalk, J. Mellmann, I. Kozma, J. Tóth, Cs. Mészáros <u>ANALYSIS OF HEAT TRANSPORT IN SOIL COLUMN MEASUREMENTS SIMULATING SUN CYCLE</u> (NO6-2-034)

A. Csábrági, S. Molnár, P. Tanos, and J. Kovács <u>PREDICTION OF DISSOLVED OXYGEN IN THE RIVER DANUBE USING LINEAR AND NONLINEAR</u> MODELS (NO6-3-055)

S. Molnár, M. Molnár and Zs. Naár-Tóth DATA MINING IN NEWS ANALYTICS: A CASE STUDY (NO6-5-060)

L. Pitlik, Z. Varga <u>THE OPERATIONALISM OF SUSTAINABILITY IS A MATHEMATICAL ISSUE (NO6-6-068)</u>

VII. Section: CONSTRUCTION AND TRIBOLOGY

Á. Horvath, I. Oldal, G. Kalácska <u>OPTIMAL PISTON'S DIAMETER RATIO IN FOUR PISTON CALIPER (NO7-1-047)</u>

J. Sukumaran, B. De Mare, M. Vanoverberghe, P. D. Neis and P. De Baets *IN-SITU* SURFACE MONITORING TECHNIQUE FOR OIL LUBRICATED CONTACTS (NO7-2-014)

L. Földi, E. Sárközi, L. Jánosi POSITIONING ALGORITHMS OF PNEUMATIC CYLINDERS (NO7-3-096)

N. Ungureanu, C. Lung, R. Cotetiu <u>E-MAINTENANCE - A NEW TREND IN INDUSTRIAL MAINTENANCE (NO7-4-087)</u>

T. Baross, L. Jánosi, G. Kalácska and G. Veres <u>DIFFUSION BONDING OF ITER PLASMA FACING COMPONENTS IN FUSION REACTOR</u> <u>ENVIRONMENT (N7-5-077)</u>

J. De Pauw, W. De Waele and P. De Baets <u>THE EFFECT OF RESIDUAL COMPRESSIVE STRESSES ON FRETTING FATIGUE LIFETIME (N07-6-024)</u>

L. Zsidai, Z Szakál <u>THE EFFECT OF THE DIFFERENT LOADS ONTO THE FRICTION AND STICK-SLIP OF POLYAMIDE</u> <u>COMPOSITES (NO7-7-065)</u>

S. Chhith, W. De Waele, and P. De Baets <u>DETECTION OF FRETTING FATIGUE CRACK INITIATION BY THERMOGRAPHY</u> (NO7-8-018)

Y. Şahin, J. D. Pauw, J. Sukumaran and P.D.Baets <u>SLIDING FRICTION AND WEAR OF POLYOXYMETHLYENE POLYMER</u> (NO7-9-035)

N. Micone, W. De Waele, S. Chhith <u>TOWARDS THE UNDERSTANDING OF VARIABLE AMPLITUDE FATIGUE</u> (NO7-10-079)

DEVELOPMENT OF BIODEGRADABILITY INDICATORS FOR MICROWAVE SLUDGE CONDITIONING

S. Beszédes¹, P. Veszelovszki², L. Ludányi¹, G. Keszthelyi-Szabó¹ and C. Hodúr¹

¹Department of Process Engineering, Faculty of Engineering, University of Szeged Moszkvai krt. 9, Szeged, H-6725, Hungary Tel.:+36 62 546-512, E-mail: beszedes@mk.u-szeged.hu

> ²Technical Institute, Faculty of Engineering, University of Szeged Moszkvai krt. 9, Szeged, H-6725, Hungary Tel.:+36 62 546-512, E-mail: veszelov@mk.u-szeged.hu

Abstract: Microwave irradiation has a good potential to increase the ability of organic matter for biological degradation. Because of the strong disintegration effect of microwave irradiation on extracellular polymeric substances (EPS), which form complex sludge structure, and also on microbial cell walls has led to enhanced solubility of organic matters, given by the ratio of soluble to total chemical oxygen demand (SCOD/TCOD). From the aspect of the further utilization of sludge, the change of biochemical oxygen demand (BOD), which correlates the degradable organic matters of sludge under aerobic condition, can be considered advantageous. For comparison purpose, and for modeling and optimization of process, it is needed to create novel control parameters, by which can be measured the changes in organic matter fraction of sludge independently from the varying characteristic or different origin of raw sludge. Based on our results, it has established that in the range of 90-1050 kJkg⁻¹, and 0.5-5 Wg⁻¹ for IMWE and MWPL, the surface fitted by constructed model can be characterized by a maximum value for BDI. Microwave treatment carried out over a certain value of MWPL and IMWE has resulted in lower biodegradability. Optimum range of IMWE and MWPL was concluded as 600-650 kJ and 2.5-3.0 Wg⁻¹, respectively, and both process parameters have significant effect on the change of BDI and SLI, as well.

Keywords: wastewater sludge, biodegradability, microwave pre-treatment

INTRODUCTION

Nowadays, because of the shortening of water resources, expectations for higher capacity and cleaning efficiency of wastewater purification technologies are growing. Notwithstanding of the technological development of wastewater treatment works the amount of sludge produced in these process is continuously increasing. Taking into consideration the environmental awareness and the more and more rigorous pollution control regulations, the research and development activity focuses on the sludge utilization processes. However the sludge utilization and valorization technologies require applying pre-treatments.

The sludge pre-treatment processes aim mainly the volume reduction to reduce the cost of transportation and storage, the destruction of pathogen microorganisms to decrease the microbial risk of sludge, the removal of the toxic components of sludge to reduce the environmental risk, or the enhanced degree of further utilization efficiency. Respect to the nowadays used industrial scale sludge handling technologies the thermal pre-treatments can be considered as the more commonly used processes. The advantageous effect of thermal pre-treatment on the sludge structure have been verified and utilized for decades (Neyens, Baeyens, 2003). The main aim for applying thermal pre-treatments is to reduce the odour load of sludge processing, to reduce the microbial risk of sludge disposal and to increase the organic matter removal efficiency of anaerobic digestion in industrial scale technologies

Previous investigations have been verified, that microwave irradiation is applicable in various waste and byproducts handling technologies with high efficiency. Beside these advantages, it can be concluded, that the effects and efficiency of microwave processes are not enough deeply investigated and analyzed for many types of materials. It can be also established, that results of studies on microwave sludge conditioning obtained mainly from municipal sludge processing. The main advantages of microwave heating over the conventional heating methods are the following: rapid heat generation, volumetric heating, selecting heating mechanism in multicomponent system resulted from the different dielectric properties of components, and the very short process time demand due to the high energy intensity of microwave irradiation (Clark et al., 2000). Application of MW irradiation combining with the oxidation process, such as ozonation, can also be considered to be promising technology as pre-treatment before AD of high organic matter containing but less degradable sludge (Beszédes et al., 2009).

Energy transfer carried by microwave irradiation affect the biodegradability of materials in two ways. Thermal effect is expressed in the increase of internal pressure of intracellular liquor caused by internal heating and rapid evaporation, which altogether can lead to cell wall disruption (Géczi et al., 2013). The non-thermal effect of high frequency electromagnetic field contributes to alter the structure of macromolecules with polarization of side chains and breaking of hydrogen bounds (Park et al., 2004; Lakatos et al., 2005). High efficiency of MW treatments in the biomaterial processing and also on the rate of chemical reactions is often explained by the non-thermal effects of microwaves due to the direct interaction of electromagnetic field with molecules.

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 such as cellulose and pectin (Banik et al., 2003). MW pre-treatment has verified positive effects on cell wall destruction and releasing of organic matter into the soluble phase, but combining of it with addition of chemicals such as alkali, acid and oxidizer agents cause synergetic mechanism to accelerate the decomposition under aerobic and anaerobic condition., as well (Beszédes et al., 2011).

Despite the good potential of microwave irradiation for material processing and the promising results of the field of microwave research the adaptation of them in industrial scale processes has been not realized yet. Available scientific results and experiences from the microwave sludge conditioning are not enough to scale up the microwave process. Furthermore, beside these advantages, it can be concluded, that the effects and efficiency of microwave processes are not deeply enough investigated and analyzed for many types of materials.

Published scientific work and research reports in the field of microwave sludge processing find microwave irradiation as an efficient method to dehydrate the sludge, and to recover some valuable component, such as some metallic compound, and microwave irradiation is suitable as pre-treatments method before anaerobic digestion to increase the biogas yield. This advantageous and beneficial feature of microwave irradiation is caused by the effect of high frequency electromagnetic field on the secondary and tertiary structure of proteins, which can manifested in the decreased stability and the enhanced disintegration degree of sludge particles (Tang et al., 2010). It has been also concluded, that the increment in the solubility of carbohydrate compounds of sludge was approximately 10-15% higher, than the improvement in protein solubilisation after microwave treatment (Appels et al., 2010).

Comparing of the results obtained from different studies has difficulties because of the different condition of experimental set-up, and the undefined or not exactly defined control parameters used for evaluation of microwave process. Therefore these observations are often led to incorrect conclusion, determined optimum parameters cannot be used for another material or equipment, and make complicated the scale up of processes. Additionally, the comparison of the efficiency of microwave operation and process parameters is difficult because the control parameters are undefined or the calculation methods are different, and the analytical methods are not standardized.

There are some studies concluded the increasing of microwave power has an increasing effect on the organic matter solubility (Climent et al., 2007; Appels et al., 2013), nevertheless other studies summarized decreasing effect of the enhanced microwave power (Park et al., 2010; Toreci et al., 2009). Applying microwave treatment with high power intensity in open vessel systems, the agglomeration of partly dehydrated sludge particles are observable, because of the intensive evaporation from the surface of the sludge exposed to microwave irradiation. In this case, the change in sludge structure is considered irreversible, because the water addition after the microwave treatment is not able to resolve the organic matters (Sólyom et al., 2011).

It can be also noticed, that results of studies on microwave sludge conditioning obtained mainly from municipal sludge processing, which has lower organic and dry matter contant than the food industry sludge. It has been established, that microwave pre-treatments of municipal sludge enhance the organic removal efficiency of anaerobic digestion process (Appels et al., 2013) and increase the biogas production (Toreci et al., 2009).

MATERIALS AND METHODS

Investigated wastewater sludge was originated from meat industry with a total solid (TS) content of 9.7 %, initial total COD of 105.9 kgm⁻³, and soluble COD of 20.1 kgm⁻³. Microwave experiments were carried out in a tailor made microwave sludge processing and measuring system developed at the Process Engineering Department of the University of Szeged Faculty of Engineering. Microwave equipment has a continuously irradiating magnetron with changeable power in the range of 50 W to 700 W operating at a frequency of 2450 MHz. Power of the continuously irradiating microwave magnetron is adjustable by varying of anode voltage through a transformer with variable voltage. To investigate the efficiency of the MW pre-treatment process the parameters studied were the microwave power level (MWPL), and the irradiated microwave energy (IMWE). MWPL (Wg⁻¹) was defined as the ratio of magnetron power to the quantity of treated sludge. MWPL and IMWE were calculated from the weight of sludge samples (m_{s1} [g]), the power of magnetron (P_m [W]), the irradiation time (τ [s]) and the percentage irradiation time period of magnetron (I [%]).

$$IMWE = \frac{P_m \times \tau \times I}{100 \times m_s} \quad [Jg^{-1}]$$

Carbonaceous biochemical oxygen demand (BOD) tests were carried out in a respirometric BOD system (BOD Oxidirect, Lovibond, Germany) thermostated at a temperature of $20\pm0.2^{\circ}$ C (TS606, WTW). BOD analysis was according to the APHA5210D method. To ensure the consistency of the experiments, BOD seed microbe capsules (Cole Parmer, USA) were used for the measurements. The chemical oxygen demand (COD) was measured by the standardized colorimetric dichromate method using HACH test cuvettes, according to USEPA 5520D method. For the thermal digestion ET108 (Lovibond) thermoblock was used for 2 hours at 150°C. The COD was measured colorimetrically using PC Checkit photometer with inner calibration for COD measurement. The total chemical oxygen demand (TCOD) was measured from the total sludge matrix. The soluble organic matter content was given as the soluble chemical oxygen demand unit (SCOD). SCOD was determined after separation of soluble from solid fraction by centrifugation (RCF of 32000 g for 15 minutes, MPW-350 centrifuge) and pre-filtration (0.45 μ m Millipore cellulose-acetate disc filter).

The solubility index (SLI) and the biodegradation index (BDI) are calculated from the total organic matter content (given by the TCOD), the soluble organic matters content (SCOD) and the biochemical oxygen demand (BOD), measuring them initially (0), at a given time (t) and from the parameters of maximum biodegradable sample (max).

$$SLI = \frac{\left(\frac{SCOD}{TCOD}\right)_{t} - \left(\frac{SCOD}{TCOD}\right)_{0}}{\left(\frac{SCOD}{TCOD}\right)_{max} - \left(\frac{SCOD}{TCOD}\right)_{0}} \quad [-] \qquad BDI = \frac{\left(\frac{BOD}{SCOD}\right)_{t} - \left(\frac{BOD}{SCOD}\right)_{0}}{\left(\frac{BOD}{SCOD}\right)_{max} - \left(\frac{BOD}{SCOD}\right)_{0}} \quad [-]$$

RESULTS AND DISCUSSION

Our earlier results verified that MW pre-treatments have numerous advantages over the conventional heating, such as higher biogas yield and accelerated anaerobic decomposition. Increment of solubility and biodegradability depend on the MWPL applied during microwave pretreatment and also on the irradiation time.

To analyze the effect of the two process parameters, i.e. IMWE (x_1) and MWPL (x_2) , on the change of SLI and BDI response surface methodology (RSM) with CCF design was applied. Equations with significant terms remained showing that both of the factors have a linear and quadratic effect on the value of control parameters studied. Interaction among variables of IMWE and MWPL was not significant for BDI at the significance level of 0.05 (α =5%), but it was significant for SLI. Multiple linear regression analysis of the experimental data yielded the following second-order polynomial equation:

$$SLI = 0.8085 + 0.301x_1 + 0.014x_2 + 0.0706x_1x_2 - 0.237x_1^2 + 0.036x_2^2 \quad [-]$$
$$BDI = 0.8921 + 0.071x_1 - 0.002x_2 - 0.298x_1^2 + 0.043x_2^2 \quad [-]$$

Results of the ANOVA for the quadratic model for BDI show a high degree of correlation between the predicted and observed values (R^2 value was over 0.9), the lack of fit values indicated that the lack of fits were not significant relative to the pure error at a level of 0.05. Results of RSM show that the degradation effects of MW pre-treatment were limited due to the demineralization effects. Results of response surface analysis show that MW pre-treatments enhance the SLI from 0.18 to above 0.9 at high IMWE and MWPL levels, but the increment of SLI was limited. Maximal SLI was obtained if irradiated MW energy reach IMWE value of 650 kJ and MW intensity was over MWPL of 2 Wg⁻¹, higher value of irradiated energy or higher intensity of MW treatment caused any further increment in the SLI (Fig. 1.)

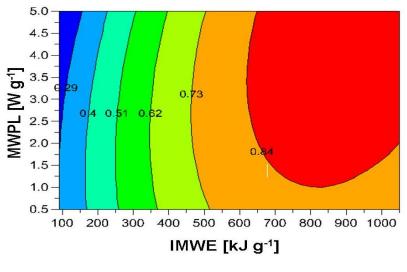


Fig. 1. Contour plot for SLI

Trends of our experimental results are in good agreement with those reported by Ahn et al. (2009) and Eskicioglu et al. (2006), respectively; but our work was focused on the effects of different MWPL on sludge characteristic and the absolute value of differences in SLI was due to the different origin of processed sludge. Because of the strong disintegration effect of microwave irradiation on extracellular polymeric substances (EPS), which form complex sludge structure, and also on microbial cell walls has led to enhanced solubility of organic matters of food industry sludge. Degree of solubility was influenced by the preliminary treatment, increase in the absolute value of solubility was lower when secondary municipal sludge was used.

Contour plot for BDI indicate the optimum region for maximum biodegradability in IMWE range of 600-650 kJ/g, and MWPL range of 2.5-3.0 Wg⁻¹, respectively (Fig. 2). Applying IMWE and MWPL at optimum level initial BDI for untreated sludge (0.21) enhanced to above 0.8 due to the microwave treatment.

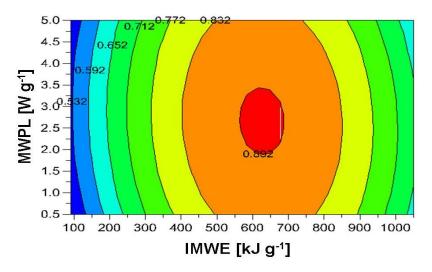


Fig. 2. Contour plot for BDI

On the contrary to the results related to SLI, if the MW pre-treatment was carried out with higher intensity than the optimum region, or the irradiated energy was higher than that of it, a decreasing of BDI occurred. Results of RSM show that the degradation effects of MW pre-treatment were limited due to the demineralization effects. These effects play a significant role in the worsened biodegradability after high intensity MW irradiation of high water contented sludge. Additionally, in the materials containing carbohydrates and proteins, such as the investigated sludge, Maillard reactions can be take place under high temperature treatments which can manifested in an inhibited decomposition.

CONCLUSIONS

In present work the effects of MW pre-treatments on the biodegradability of organic matters, and biogas product from meat processing wastewater sludge (MPWS) were examined. To quantify the change in the solubility of organic matter of sludge and in the degree of biodegradability the solubilization index (SLI) and the biodegradation index (BDI) were developed. Defined SLI and BDI as control parameters, effects and significance of irradiated microwave energy (IMWE) and microwave power level (MWPL) were investigated with experimental design and response surface methodology (RSM).

Our results show, that MW pre-treatments could increase the biodegradability of organic matters of MPWS. However, over a certain value of MWPL and IMWE the degree of aerobic degradation and also the biogas production was worsened. Experimental results verified, that defined solubility index (SLI) and biodegradability index (BDI) is suitable to quantify the change of physicochemical characteristics and bioavailability of organic matter fraction of sludge. Based on the results of response surface modeling (RSM) optimum region for maximum biodegradability was determined in IMWE range of of 600-650 kJg⁻¹, and MWPL range of2.5-3.0 Wg⁻¹, respectively. It can be summarized, that the MW pre-treatments could be advantageous in numerous process based on biological transformation, such as activated sludge recycling processes, composting and anaerobic digestion.

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