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APPLICATION OF RESPONSE SURFACE METHODOLOGY TO OPTIMIZE MICROWAVE SLUDGE CONDITIONING FOR ENHANCED BIOGAS PRODUCTION

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ABSTRACT: Microwave (MW) conditioning can be considered as intensive process for handling of wastewater sludge. Several studies have dealt with the physicochemical and bio-effects of MW irradiation, but few of them focus on the determination and optimisation of influential parameters for sludge conditioning process. In our work the effects of MW irradiation with different intensities was studied on the solubility of meat processing wastewater sludge (MPWS) indicated by the solubility index (SLI) and biogas production, applying response surface methodology (RSM) with central composite face centred (CCF) experimental design. Our results show that SLI was in good correlation with biogas production, and it could be large scale increased by the MW pre-treatments. To achieve the maximum biogas production corresponding to the highest SLI the optimum value of microwave power level (MWPL) and irradiated microwave energy (IMWE) was determined of 741 kJ and 1.7 Wg^{-1}, respectively.

KEYWORDS: sludge conditioning, microwave, biogas, response surface methodology

INTRODUCTION

Food processing technologies output a great amount of wastewater. Wastewater originated from food operations contains high amount of organic matter, and because of the frequently cleaning and disinfecting procedures, effluents can be characterized by a high content of surfactants. Dosed cations in the precipitation stage of wastewater line altogether with living microorganisms and organic molecules contribute to 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. It causes hydrophilic characteristic of sludge, and increases the difficulty to achieve effective bioconversion during anaerobic condition (AD) or aerobic biotransformation processes, e.g. composting.

Professionals in food industry companies face the high disposal costs of sludge and the rising price of energy sources. In the case of bio-wastes, such as wastewater sludge, one of the possible solutions is the biogas production, according to the waste-to-energy concept. Beyond the volume reduction, keeping in mind the cost minimizing, sludge conditioning operations may also have requirement to modify the sludge structure before further utilization and/or kill pathogens. Several sludge conditioning methods were developed and investigated to improve the accessibility of organic matter for decomposing bacteria, and to enhance biogas production. Moreover, various 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 [1-3].

Generally, thermal treatment is readily used due to its good controllability and the widespread application of the commercial heat exchangers. Among thermal methods heating by microwave (MW) irradiation is considered as a novel and very intensive method. Microwave irradiation is a viable method for operations in which instantaneously and volumetrically heating inside the materials is needed [4]. If the process parameters and the geometry of MW system are sufficiently controlled, heat distribution in the materials is more uniform comparing of them with conventional heating methods. Furthermore, advantages of MW heating alone or combined with other methods can be manifested in less floor space and reduced process time, what provoke energy saving, as well.

Many researches focused on examining the efficiency of MW treatment on sludge characteristic. After MW pre-treatment Eskicioglu et al. [5] and Park et al. [6] found an increment of 79% 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 [7-8]. Synergetic effects of alkaline treatment in combination with MW irradiation resulted in enhanced COD solubilization [9]. After optimized condition of anaerobic process Thungklin et al. [10] reported a considerable higher bio-hydrogen production for MW pre-treated sludge than the hydrogen yield achieved using raw sludge as feed. MW pre-treated sludge contains soluble proteins and volatile fatty acids in a higher concentration, but it has a lower soluble sugar content compared to conventional heated sample [11]. In the work of Yu et al. [12] 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.

Before the scale up of microwave operations for sludge pre-treatment the identifying of the main influential process parameters are needed. Small number of studies is available on the optimization of process parameters for MW sludge conditioning and very few of them deal with the pre-treatment of food industry sludge. Main focuses of our work was therefore defining control parameter for MW sludge condition and optimize the process to achieve maximum biogas yield.

❖ MATERIALS AND METHODS

Meat processing wastewater sludge

Dewatered meat processing wastewater sludge (MPWS) came from the final collecting tank of wastewater treatment plant of a local meat processing factory (Szeged, Hungary). MPWS had an average total solid (TS) content of 17.3±1 w/w%, with total chemical oxygen demand (TCOD) and soluble fraction of COD (SCOD) of 539.8±7.6 w/w% and 101.3±4.3 w/w%, respectively.

Experimental set-up

MW pretreatments were performed in a special designed microwave equipment containing continuously irradiating magnetron with a nominal power of 900W, operating at a frequency of 2450 MHz (Yantai Co., China). The magnetron power (P_m) is changeable continuously in a range of 50 to 900W through varying the heating voltage with a toroidal-core transformer. Calculation of penetration depth for sludge is problematic due to the absence of exact value relate to dielectric constant and dielectric loss factor. In order to maintain a quasi-homogenous temperature distribution 100 g of sludge was placed in all tests into the container formed 30 mm thick layer. Sampling for analysis was after homogenization by a laboratory mixer.

To investigate the efficiency of MW pretreatment the process parameters studied were the microwave power level (MWPL), and the irradiated MW energy (IMWE). MWPL (Wg^{-1}) was defined as the ratio of magnetron power to the quantity of treated sludge. IMWE (kJg^{-1}) was calculated as the product of magnetron power (P_m) and the irradiation time (t_{irr}) divided by the quantity of processed sludge (m_{sample}.)

$$IMWE = \frac{P_m \cdot t_{irr}}{m_{sample}} \quad [kJg^{-1}] \tag{1}$$

Analytical methods

Total chemical oxygen demand (TCOD) in the total MPWS was measured triplicated using colorimetric standard method (APHA, 2005). Soluble COD in supernatant (SCOD) was determined after separation by centrifugation (12,000 rpm for 10 minutes) and prefiltration (0.45 ◻m Millipore disc filter). Total solid content (TS) was measured by drying to constant rate at 105°C.

To evaluate the organic matter solubilization and for eliminating the effects of different characteristic of sludge processed the dimensionless solubilization index (SLI) was calculated as follows:

$$SLI = \frac{(SCOD/TCOD)_t - (SCOD/TCOD)_i}{(SCOD/TCOD)_{max}} \tag{2}$$

where $(SCOD/TCOD)_i$ and $(SCOD/TCOD)_t$ the ratio of soluble to total COD initially (untreated MPWS) and at MW irradiation time t, respectively.

According the method of Eskicioglu et al. [7] $(SCOD/TCOD)_{max}$ was defined as the maximum achievable solid solubilization, quantified by COD measurement after NaOH addition into the sample in a concentration of 2 mol L^{-1} and applied contact time of 72 hours at a controlled temperature of 50°C.

Batch mesophilic biogas production tests were carried out triplicate in plasma bottles equipped by Oxitop-C barometrical measuring head (WTW GmbH, Germany). Methane concentration in biogas was measured by Airtox biogas analyzer (Fresenius Umwelttechnik GmbH., Germany), methane yield was determined by dividing the methane specific production by the theoretical maximum methane yield of 350 $mL_{(CH4)}g_{(CODcons)}^{-1}$

Statistical analysis

To study the effects of the two factors and their possible interactions response surface methodology (RSM) with a central composite face centered (CCF) experimental design was performed using MODDE 8.0 statistical experimental design software (Umetrics, Sweden). For the modeling and optimization the studied factors were IMWE and MWPL, the selected responses were the dimensionless SLI.

The total number of experimental run was 22. In order to reduce the systematic error, the runs of the experiments were randomized. Based on our preliminary experiments IMWE and MWPL were varied in the range of 90 – 1050 kJg^{-1} and 0.5-5 Wg^{-1}, respectively. To evaluate the reproducibility of the quadratic model fitted with multiple linear regressions (MLR), three center points were used in the experimental design. The matrix for CCF and the calculated SLI obtained from experiments are summarized in Table 1.

Table 1. Experimental matrix

Exp.	Run Order	IMWE [kJ g^{-1}]	MWPL [W g^{-1}]	SLI
N1	5	90	0.5	0.303
N2	18	1050	0.5	0.734
N3	14	90	5	0.175
N4	17	1050	5	0.907
N5	8	90	2.75	0.253
N6	7	1050	2.75	0.903
N7	16	570	0.5	0.751
N8	20	570	5	0.793
N9	9	570	2.75	0.8
N10	11	570	2.75	0.798
N11	4	570	2.75	0.803
N12	15	90	0.5	0.297
N13	10	1050	0.5	0.745
N14	21	90	5	0.186
N15	6	1050	5	0.899
N16	22	90	2.75	0.267
N17	19	1050	2.75	0.902
N18	3	570	0.5	0.776
N19	13	570	5	0.809
N20	12	570	2.75	0.803
N21	2	570	2.75	0.801
N22	1	570	2.75	0.807

Behavior of SLI was described by second-order polynomial equation (Eq. 3)

$$Y = \beta_0 + \sum_{i=1}^{2} \beta_i x_i + \sum_{i=1}^{2} \beta_{ii} x_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{2} \beta_{ij} x_i x_j \qquad (3)$$

where Y is the predicted response, x_i and x_j are the coded values of the independent variables.

Significance and reliability of the model terms was examined by regression analysis, lack of fit tests and analysis of variance (ANOVA).

RESULTS AND DISCUSSION

As preliminary experiments MPWS pretreatments were carried out applying different MW intensity in the range of 0.1 – 10 Wg^{-1} with duration time of 0 to 3000 sec. Results shown, that MW irradiation increased the solubility of organic matters indicated by the enhanced SLI. Amount of SLI increment depend on the MWPL applied during microwave pretreatment and the irradiation time.

Our experimental data are in good agreement with the results of Ahn et al. [13] and Eskicioglu et al. [7], respectively; but in our work was focused the effects of different MWPL on sludge characteristic. The effects of MW irradiation at higher MWPL was manifested in the enhanced SLI, but increase the intensity above a certain value limited value of SLI was obtained (Figure 1.). Limited increment in organic matter solubility after MW pretreatment was also experienced earlier by dairy wastewater sludge [14].

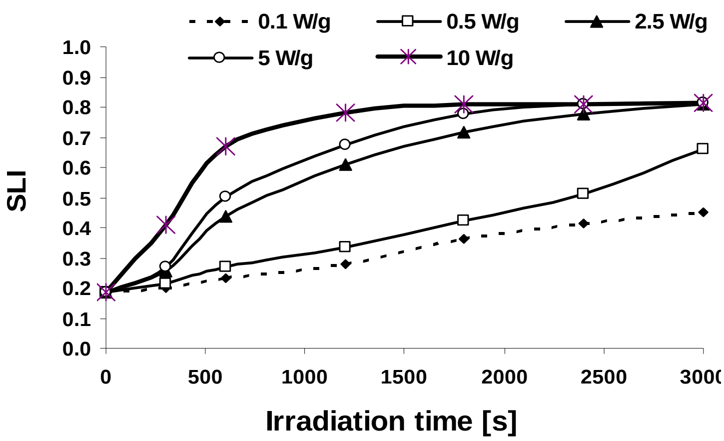


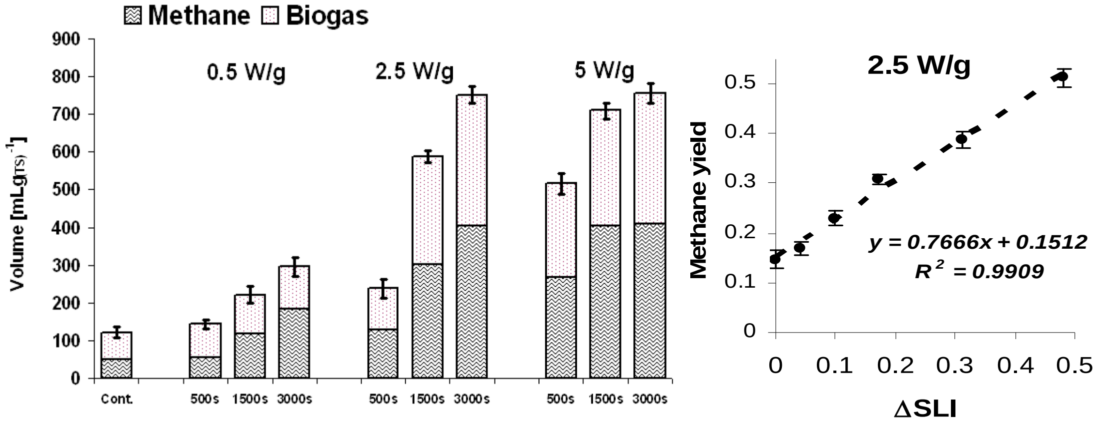
Figure 1. Change in SLI after MW pretreatments

Based on the results of preliminary experiments MWPL and IMWE was supposed to be influential parameters on solubility, therefore both of them can be considered suitable for modeling and optimization of MW sludge conditioning process.

As anaerobic decomposition is a microbial biotransformation process, efficiency of AD process is linked to the amount of organic matter in the soluble fraction of sludge. Hence MW pretreated sample was digested under anaerobic condition at 35°C, the results of AD tests are summarized in Figure 2.

With the MW irradiation the specific biogas product of MPWS increased considerably, at higher MWPL the increment was above 650%, related to control. The large scale enhancement in biogas volume was due to the intensive pre-digestion effect of microwave irradiation. Thermal treatment and MW irradiation had verified effect on the solubilization and volatilization [14, 15].

Furthermore, sludge particles are disintegrated during MW irradiation and the compact sludge matrix formed by EPS has been also degraded [13]. The linear connection between the change in SLI and the change in methane yield (relate to theoretical maximum methane production) was also confirmed by our experimental data, indicated in Figure 2.b. Taking into consideration the strong correlation between the organic matter solubilization and methane yield, SLI was concluded as a suitable parameter to predict the biogas production.



a) b)

Figure 2. Biogas production of MW pre-treated MPWS (a) and correlation between the change of SLI and methane yield related to theoretical maximum biogas product (b)

To study the effect of the two process parameters, i.e. IMWE and MWPL, on the change of organic matter solubility indicated by the dimensionless SLI response surface methodology (RSM) with CCF design was applied. Levels of factors and the value of response parameter (SLI) were shown in Table 1. Multiple linear regression analysis of the experimental data yielded the following second-order polynomial stepwise equation:

$$SLI = 0.8085 + 0.301\, x_1 + 0.14\, x_2 + 0.0706\, x_1 x_2 - 0.237\, x_1^2 - 0.036\, x_2^2 \tag{4}$$

where IMWE and MWPL are encoded as x_1 and x_2 , respectively.

Table 2. Variance analysis of regression model for SLI

Source	Degree of freedom	Sum of squares	Mean of squares	F	P
Model	21	147.31	70.02	12.4	0.004
Residual	16	46.34	2.896		
Lack of fit	9	38.76	1.29	10.3	0.093
Pure error	7	7.68	0.583		
R^2 = 0.917	R^2(adj) = 0.894	R^2(pred) = 0.904			

Results of the analysis of variance for the response surface quadratic model for SLI are shown in Table 2.

The calculated R^2 value of 0.917 for Eq. (4) indicates a high degree of correlation between the predicted and observed values. Adjusted value of R^2 suggests that only 10.6% of the total variations were not explained by the constructed model. ANOVA of the regression demonstrated that the model is significant, the lack of fit value indicated that the lack of fit is not significant relative to the pure error at level of 0.05. Model constructed for SLI was found to be adequate for prediction within the range of variables applied.

Two-dimensional contour plot of fitted response surface is shown in Figure 3.

Contour plot for SLI are good agreement with our experience, i.e. limited SLI was obtained, if irradiated MW energy reach IMWE value of 650 kJ and MW intensity applied was over MWPL of 1 Wg^{-1}. Determination coefficient for IMWE and MWPL was about 0.3 and 0.14, respectively, which show that the effect of the two factors can be considered as significant influential process parameters. Based on the optimal region determined by RSM and aiming minimized energy demand of MW pretreatments the optimum value for IMWE and MWPL was 741 kJ and 1.7 Wg^{-1}, respectively.

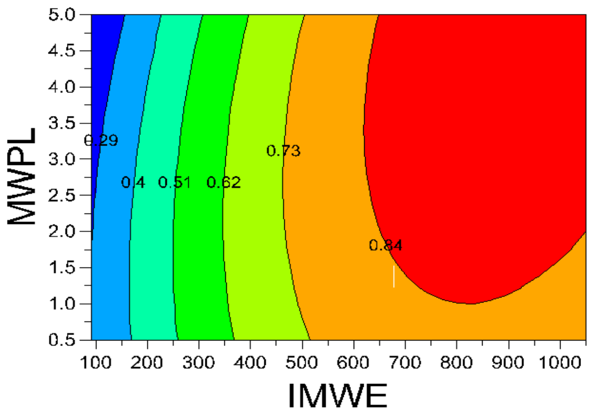


Figure 3. Contour plot for IMWE and MWPL on SLI

❖ Conclusion

MW conditioning of MPWS was considered as appropriate process to enhance the solubility of organic matter content in food processing wastewater sludge and it is suitable to increase the biogas production. RSM was adequate method for estimating the change of SLI during MW pre-treatment. Both the factors studied, such as IMWE and MWPL, had significant effect on the response parameter of SLI. Maximum value of SLI and biogas production can be achieved by applying IMWE and MWPL of 741 kJ and

1.7 Wg^{-1}, respectively. With determination of influential parameters and optimization of them make MW process suitable for scaling-up and allow to design continuous flow MW reactor for sludge conditioning.

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