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Microwave Pretreatment of Mixed Sludge for Anaerobic Digestion Enhancement

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Abstract

The impact of microwave (MW) irradiation of mixed sludge (MS) on biogas production has been investigated. For this purpose, mixed sludge from a nearby activated sludge treatment plant and domestic MW equipment were used in two separate runs. During the first run (TS=2.47% (w/w)), preliminary investigations were carried out to determine the effect of microwave intensity on mixed sludge samples. Second run experiments (TS=1.46% (w/w)) were conducted to further investigate the impact of temperature. Sludge samples microwaved at the same temperature and at different MW intensities, consequently different exposure times, achieved almost equal degree of solubility, as expressed in terms of COD_S/COD_T and VSS. At an ultimate pretreatment temperature of 96±2°C, the level of particulate COD solubility resulted in 3.13±0.001, 2.69±0.33 and 3.48±0.25 fold increase of COD_S/COD_T ratios at MW intensities of 500, 800 and 1000 W, respectively compared to untreated. MS microwaved to 78°C and power intensity of 1000W produced the greatest improvement in cumulative biogas production with 84% increase as compared to the controls after digestion for 33 days at sludge concentration of 1.46% TSS (w/w).

Keywords: *Anaerobic Digestion, Biochemical Methane Potential Test, Microwave Irradiation, Mixed sludge*

1. Introduction

The processing and disposal of sewage sludge is one of the most important and complex problems in the operation of municipal wastewater treatment plants. This is a problem of growing importance, representing up to 50% of the current operating costs of a wastewater treatment plant [1]. Although different alternatives exist for sludge treatment, anaerobic digestion plays an important role as it can produce energy-rich biogas, destroy most of the pathogenic organisms and stabilize the sludge [2].

Anaerobic degradation of particulate materials is a four step sequence: hydrolysis, acidogenesis, acetogenesis, and methanogenesis, in which hydrolysis is considered as the rate-limiting step [3]. Solubility and conversion of slowly biodegradable organic matter to readily biodegradable low-molecular-weight compounds takes place by extracellular enzymes excreted by anaerobic bacteria [1].

Previous studies have indicated that various pretreatment processes, namely; mechanical [4], ultrasonic [5], thermal [6, 7], chemical [8], and enzymatic [9] methods could enhance anaerobic digestion process.

MWs are short waves of electromagnetic energy in a frequency range of 0.3 to 300 GHz with most microwave applications falling between 3 and 30 GHz [10]. In industry, microwave heating is performed at either a frequency close to 900 MHz or at 2450 MHz. MW increases the kinetic energy of water dipoles bringing it to its boiling point very quickly. Although the quantum energy of MW irradiation may not be strong enough to break chemical bonds, some hydrogen bonded structures can be weakened or broken if exposed to MW irradiation [11]. The direct interaction of MW irradiation of biological samples at the molecular or cellular level is still poorly understood [12].

The application of MW- irradiation techniques offers great advantages over conventional methods of sludge treatment and in the production of environmentally clean and value-added products [13]. There is significant potential for microwave technology to be employed as an alternative heating source in the treatment of waste streams. However, several major limitations prevent these technologies from being widely employed. These include the absence of sufficient data to quantify the dielectric properties of the treated waste streams, and technical difficulties encountered when upgrading successful laboratory or pilot-scale processes to the industrial

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scale [14], adding to the fear of unjustifiable additional cost associated with operation and equipment.

The primary organic compounds in sludge are carbohydrates, proteins and lipids. Under microwave irradiation, the hydrolysis pathway of the organics according to Brooks [15] proceeds as follows: hydrolysis of the lipids to stearic acid, and oleic acid; hydrolysis of the protein into a series of saturated and unsaturated acids, ammonia, and some carbon dioxide; and hydrolysis of the carbohydrates into polysaccharides with a smaller molecular weight and, possibly into simple sugars. Subsequently, the simple compounds can be easily decomposed by microorganisms [16].

Pretreatment of sludge by microwave irradiation has been one of the methods reported repeatedly in literature over the last decade. Studies carried out [16, 17, 18, 19] indicated that Microwave (MW) irradiation of municipal sewage sludge improved sludge dewaterability, reduced fecal coliforms count in the sludge and most importantly increased biogas production.

Eskicioglu et al. [18] compared MW pretreatment at temperatures below and above boiling point and using various sludge types. Park et al. [20] applied response surface analysis to determine the combination of temperature-increase rate and ultimate temperature on solubility degree of sludge. Park et al. [21] investigated the effect of MW power, temperature, and TS concentration on the solubility degree of waste activated sludge (WAS). They concluded that within the design boundaries, the conditions predicted to maximize the solubility degree of 17.9% were determined to be 400 W, 102^{o}C, and 2.3% TS. Elagroudy and El-Gohary [7] studied impact of changing MW operational parameters such as temperature, intensity, contact time, and sludge concentration on the physic-chemical and microbiological properties of WAS. Tiehm et al. [3] and Muller et al. [4] indicated that power intensity (I) and MW contact time (t) affect the efficiency of the pretreatment step.

None of the previous studies indicated the limiting factor for enhancing anaerobic digestion using microwave irradiation (microwave intensity, temperature and contact time). In addition, most of the studies focused on waste activated sludge; however very few studied mixed sludge [18, 20]. Since, in most WWTPs, primary sludge and excess sludge are both mixed before being anaerobically digested, it was therefore, the main objective of this study to find out the limiting factor for MW pretreatment of mixed sludge (MS).

2. Materials and Methods

2.1. Mixed Sludge

Mixed sludge was collected from El-Gabal El-Asfar WWTP, which is the main WWTP on the East bank of the Nile river, Egypt, and is considered the largest WWTP in the Middle East. The plant receives an average flow of 2 million m^3/day. The plant is of a conventional activated sludge type and includes screening and grit removal, primary sedimentation, surface aeration, final clarification and chlorination facilities. The mezophillic anaerobic sludge digestion facilities treat a mixture of thickened primary sludge and waste activated sludge subjected to dissolved air floatation. For this study, two samples of MS were collected from the influent to the anaerobic digester at two times.

Measurements indicated that the volatile solid (VS) to total solid (TS) ratio in the sludge were 50.3 to 60.9%. Most of the chemical oxygen demand (COD) was associated with the solid phase rather than soluble phase, as evidenced by the rather low soluble COD (COD_S) to total COD (COD_T) ratio of 3.9%.

2.2. Microwave Irradiation

The microwave used for this study is a household type microwave oven (MW886S, Kenwood, Japan, 1000 W, 230V, 2450 MHz, and cavity size of 520mm x 444mm x 335mm). Sludge samples were placed in microwave containers covered with a plastic lid. In the present study, initial microwave temperatures were kept less than 100□ to avoid the carbonization of humic substances in organic matter-rich sludge [22].

2.3. Experimental Runs

The experimental work plan in this study is divided into two runs using mixed sludge samples taken from the influent to the same anaerobic digester at El-Gabal El-Asfar WWTP.

2.3.1. First Run

The characteristics of MS used for this run are presented in Table 1. During this run, preliminary investigations were carried out to determine the effect of microwave intensity on mixed sludge samples. Different MW intensities (I = 500, 800 and 1000W) were used. At each of the three MW intensities, sludge was heated to reach to two temperature values of 55±3□ and 96±2□; respectively.

Table 1. Characteristics of sludge for first run

Parameter	COD_S (mg/l)	TSS (%) (w/w)	VSS (%) (w/w)	VSS/TSS *100 (%)
Value	1750	2.47	1.31	50.3

2.3.2. Second Run

The characteristics of MS used for this run are presented in Table 2. Second run experiments were conducted to further investigate the impact of temperature. For this purpose, sludge was microwaved at three different power intensities (I = 500, 800 and 1000W). At the three MW power intensities, sludge samples were exposed to MW irradiation for various contact times (t) and MW temperature (T) as well sludge sample characterization were recorded at all times.

Table 2. Characteristics of sludge for second run

Parameter	Value
COD_S (mg/l)	23708
COD_T (mg/l)	929
COD_S/COD_T*100 (%)	3.9
TSS (%) (w/w)	1.46
VSS (%) (w/w)	0.89
VSS/TSS*100	60.9
Soluble Protein (mg/l)	22.5
Soluble TKN (mg/l)	88

2.4. Samples Characterization and Analytical Methods

All sludge samples were analyzed before and after the microwave pretreatments. Temperature measurements were carried out outside the MW oven as soon as microwaving was terminated as suggested by (Louppy 2002) and after vigorous stirring of the sample for 10 s. Standard Methods [24] was used for TS/VS analysis. Colorimetric COD measurements were performed based on Standard Methods procedure 5250D [24]. The soluble phase was obtained by centrifugation; the supernatant of which was filtered through membrane filter paper of 0.45 µm. Total suspended solids (TSS), volatile suspended solids (VSS), soluble TKN and soluble protein were measured according to Standard methods (APHA 2005)

2.5. Biochemical Methane Potential Tests

Batch mesophilic anaerobic digestion tests were carried out for both runs using biochemical methane potential (BMP) test to assess sludge biodegradability. For the first run, MS samples pretreated using MW irradiation at the three MW intensities (500, 800 and 1000W) that reached MW temperature of 96±2 □ were tested using BMP test.

For second run, MS samples pretreated using MW irradiation for MW contact time of 150 s and at the three MW intensities (500, 800 and 1000W) were tested using BMP test. At a MW contact time of 150 s, the temperature reached 66, 72 and 780C for 500, 800 and 1000W MW power intensities, respectively. For both experiments, the inoculum was taken from the effluent line of the anaerobic sludge digesters at El-Gabal El-Asfar WWTP (TSS = 2.84%, VSS = 1.72%). The final concentration of MS with the inoculum was brought to 5 gVSS/l. Untreated MS samples were used in BMP tests of both runs for the sake of comparison.

BMP assays serve to measure the effect of different parameters on biodegradability of sludge. This method has been used widely to determine the ultimate methane production from a variety of feed stocks [25]. The BMP test was performed using 500 mL flask Erlenmeyer with butyl rubber stoppers as batch reactors. Nitrogen is bubbled through the mixture to displace oxygen. After addition of a buffer solution, the reactors were sealed. Batch reactors were kept in a temperature controlled incubator shaker at a temperature of 33±1ºC and mixed at 90 rpm to keep the mixture in suspension. Gas production was monitored every day during the experimental period. CODS, soluble TKN, TS, and TVS were measured for raw, pretreated and digested MS according to Standards Methods [24].

3. Results and Discussions

3.1. Results of MW Pretreatment of First Run

MS samples exposed to microwave irradiation of 500, 800 and 1000W reached 55±3 □ after 120, 90 and 60 s; respectively. It is hypothesized that pretreatment using microwave irradiation disrupt the complex sludge floc structure and release extracellular and intracellular biopolymers such as proteins, carbohydrates, and lipids from the floc structure into the soluble phase, as well as enhance the solubility of particulate organic matter [26]. The degree of solubility of the substrate can be estimated from the increase in COD_S and VSS/TSS ratio.

Figure (1a) illustrates the effect of MW treatment on COD solubility. CODS values were found to be higher at all MW intensities compared to the control sample. MS samples subjected to microwave temperature of 96±2^{o}C experienced higher solubility compared to those heated to 55±3 □ . At a MW temperature of 96±2^{0}C, MW samples experienced additional 54, 63 and 73% increase in their COD_S concentrations, respectively, compared to untreated sample.

The ratio of VSS/TSS of MS experienced a similar trend compared to CODS as presented in Figure (1b). VSS/TSS ratios for all pretreated MS samples were higher than the control ones. Maximum VSS/TSS ratio (77.3%) was reported for sludge sample treated at 1000W MW intensity and temperature of 96±2 □ .

From the available data presented in Figure (1), it could be concluded that the temperature is the limiting factor for MW treatment prior to anaerobic digestion. It is important to emphasize that power intensity and time required to reach the same temperature is different at different MW power intensities. However, it is worth mentioning that previous studies by Elagroudy and El-Gohary [17] indicated that to reach temperature above 90°C, it is more economical to use high MW intensity. This was attributed to the fact that the higher the MW power, the shorter the process time needed to reach high temperature values.

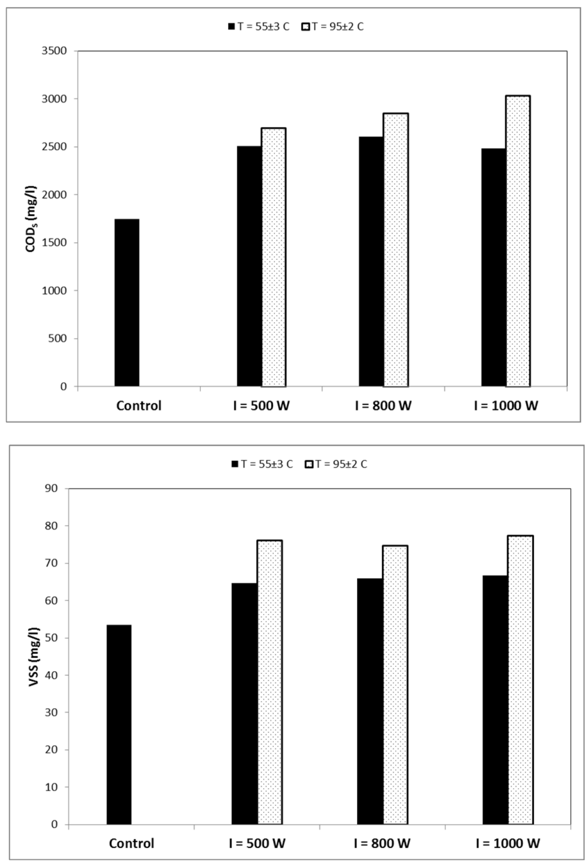


Fig. 1. Solubility effect of microwave irradiation on mixed sludge, control Vs. MW pretreated, for phase one experiments (I=500, I=800 and I=1000W; T: MW pretreatment temperature, I = MW Intensity)

3.2. Results of BMP Test of First Run

Enhancement of MS biodegradability has been evaluated by comparing biogas volumes produced by untreated and treated samples using different conditions. MS samples microwaved at a temperature of 96±2 ▫ and at the three MW intensities (500, 800 and 1000W) were tested using BMP test along with untreated samples for 70 days.

For all sludge samples, MW pretreatment led to higher biogas volume production compared to control samples (Figure 2). MS samples exposed to MW intensity of 1000W produced 1.89 times cumulative methane volume compared to untreated samples. This could be attributed to increase of sludge biodegradability as a result of MW pretreatment.

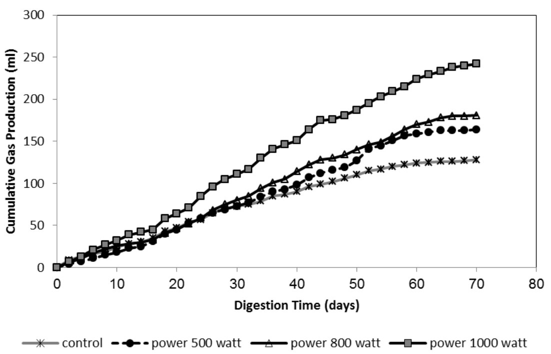


Fig. 2. Cumulative biogas productions from control and MS samples of Run 1 experiments TSS=2.47% (w/w), MW power intensity = 500, 800 and 1000W

3.3. Results of MW Pretreatment of Second Run

To investigate the impact of temperature on sludge solubility in terms of CODS and VSS as a result of increasing MW irradiation contact time (t) and MW intensities (500, 800 and 1000W), another set of experiments were carried out. Variations of sludge temperature versus time for the MS samples microwaved at the three different MW intensities (500, 800 and 1000W) are presented in Figure 3. Available data indicated that at lower power intensity (500W) longer exposure time is required to reach the same temperature.

Table 2 presents the effect of MW intensity on MS characteristics in terms of TSS, VSS, CODT, CODS, soluble proteins as well as soluble TKN concentrations. From the results obtained, it can be seen that solubility increases by increasing power intensity. Solubility was always the highest at MW intensity of 1000 W. At a MW temperature of 96±20C and MW power of 1000W, MW samples experienced additional 1.2, 3.61, 1.9 and 2.56 fold increase in their VSS, COD_S, soluble proteins and TKN concentrations, respectively, compared to untreated sample.

MW irradiation treatment resulted in significant (1.63–2.56 fold) TKN release into solution over the control. These results are in agreement with previous studies which concluded MW treatment enhances the release of TKN depending on the MW temperature [18, 27]. Among the pretreatment temperatures tested, the MS sample pretreated at 1000 W contained the highest TKN concentration which was 225 mg/L. The other MS samples pretreated at 500 and 800 W had TKN concentrations of 143.5 and 155 mg/L, respectively (Table 2).

Results of the analysis of untreated and microwaved MS samples of run 2 experiments in terms of COD_S/COD_T and VSS are presented in Figure 4a. At an ultimate pretreatment temperature of 96±20C, the level of particulate COD solubility resulted in 3.13±0.001, 2.69±0.33 and 3.48±0.25 fold increase of COD_S/COD_T ratios at MW intensities of 500, 800 and 1000 W, respectively compared to untreated. These results support the strong solubility effect of MWs on MS previously reported by Eskicioglu et al. [26], though the results of this study are slightly lower. As expected, volatilization of organics increased by increasing the MW exposure time. Solubility was always higher at 1000W than at 500W and 800W MW power intensities at the same contact time.

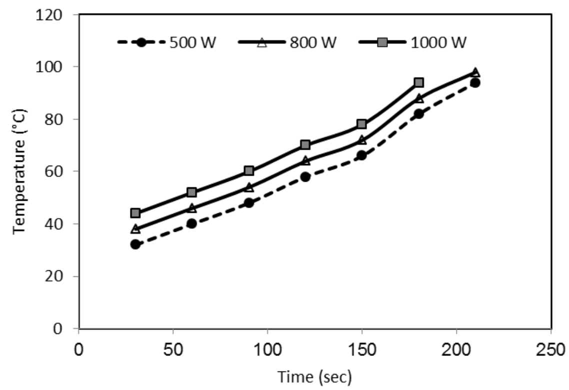


Fig. 3. Effect of MW intensity and contact time on sludge temperature

Table 2. Characteristics of treated mixed sludge for second run

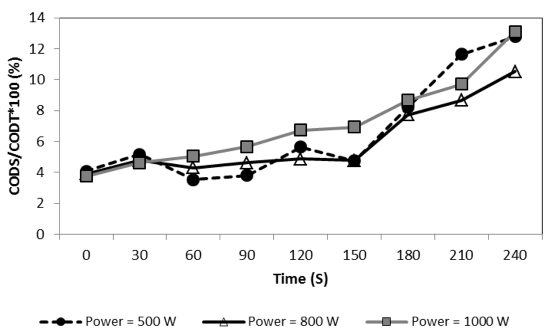
Parameter	I=500W	I=800W	I=1000W
TSS [%, w/w]	1.776	1.995	1.97
VSS [%, w/w]	1.012	1.215	1.215
VSS/TSS [%]	56.98	60.9	61.7
CODT [mg/L]	21295	28750	25550
CODS [mg/L]	2720	3025	3350
CODS/CODT [%]	12.78	10.52	13.11
Soluble Proteins [mg/L]	40.65	40.5	43
Soluble TKN [mg/L]	143.5	155	225

The findings are in agreement with those of Yu et al. (2009), who observed that COD_S/COD_T ratios increased with microwave intensity and contact time. The findings, however, do not agree with those of Eskicioglu et al. [18] and Park et al. [20], who observed that the COD_S/COD_T ratios increased as microwave intensity decreased.

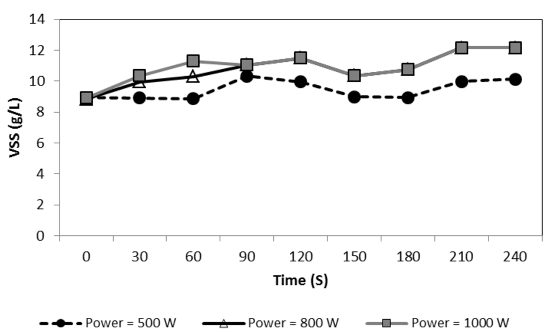
Variations in VSS concentrations of MS as a function of microwave intensity and contact time are shown in Figure 4b. Available results showed that VSS concentrations increase with the increase of microwave contact time. The percentage increase in VSS was higher at high MW intensity (1000W) than at MW intensity of 800W. This is due to the solubility of the organic matter.

At power intensities of 500, 800 and 1000W, temperature of 55±3 ▫ that has been reached after 120, 90 and 60 s, COD_S/COD_T, were insignificantly different (Figure 4a). Same trend has been reported for VSS. It is worth mentioning that sludge samples microwaved at the same temperature and at different MW intensities, consequently different exposure times, achieved almost equal degree of solubility, as expressed in terms of COD_S/COD_T and VSS. No increase in the values of VSS and VSS/TSS has been noticed by increasing the contact time more than 150 s. Therefore, it has been decided to select this contact time for BMP test to be more cost effective.

It is also necessary to emphasize that so far, different MW pretreatment studies achieved different soluble to total COD ratios at similar MW temperatures depending on the ramp, heating/holding times, MW units used (domestic or closed vessel) as well as the characteristics of secondary sludge being pretreated (moisture content, SRT of the activated sludge process, and chemicals added for thickening or nutrient removal at the WWTP) [29].



(a)



(b)

Fig. 4. Effect of MW intensity (I), contact time (t) and temperature on MS hydrolysis

3.4. Results of BMP Test of Second Run

Enhancement of MS biodegradability can be evaluated by comparing biogas volumes produced from microwaved and untreated samples. MS samples microwaved at constant contact time of 150 s and three different power intensities (500, 800 and 1000W) were investigated using BMP test. Corresponding temperatures were 66, 72 and 78^{0}C, respectively.

For all sludge samples, MW pretreatment led to higher biogas volume produced than the untreated sludge ones, as shown in Figure 5. Due to proper acclimation of inoculum, none of the BMP bottles experienced an apparent lag phase at the beginning of the batch tests (Figure 5). Starting day 1, pretreated bottles had higher cumulative biogas production (CBP) rates than day 1; in particular MS microwaved at 1000W power intensity was the highest. Around the 20th day, CBP differences between the control and the pretreatment reactors were around 28.2, 44.9, and 52.1% for MS microwaved at 500, 800, and 1000W power intensities, respectively. As digestion continued, CBP differences were almost the same between treated and untreated samples. Results also indicate that within a practical digestion time of 33 days, MW pretreatment enhanced the ultimate biodegradability of MS. Ultimate (at the end of the 33 days) methane yields from the BMP assays indicated that batch reactors pretreated at power intensity of 1000W and temperature of 78□ achieved methane yields of 84% higher than that of the control sample. The fact that the rate of biogas production was higher for treated samples compared to the control one at the early stages of the BMP test also suggests that refractory organic compounds in the MS were converted into more easily biodegradable compounds. The results obtained indicated higher biogas production for all treated samples compared to untreated MS samples. It can also be concluded that increasing power intensity increased sludge biodegradability.

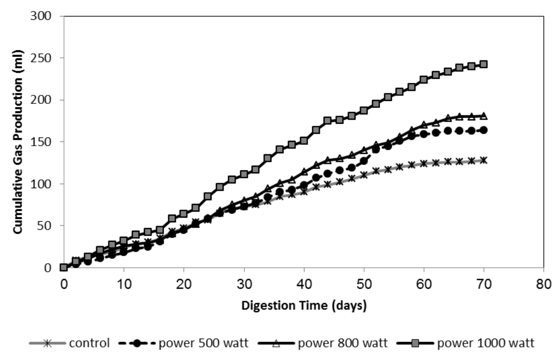


Fig. 5. Cumulative biogas productions from control and MS samples of run 2 experiments, power intensity = 500, 800 and 1000W

4. Conclusion

Based on the experimental results, the following conclusions can be drawn:

1- Sludge samples microwaved at the same temperature and at different MW intensities, consequently different exposure times, achieved almost equal degree of solubility, as expressed in terms of COD_S/COD_T and VSS. At power intensities of 500, 800 and 1000W, temperature of 55±3□, which has been reached after 120, 90 and 60 s, CODS/CODT and VSS were insignificantly different.

2- MW irradiation releases the organic matter in the floc. This has been confirmed by the results of the analysis of microwaved MS samples. At an ultimate pretreatment temperature of 96±20C, the level of particulate COD solubility resulted in 3.13±0.001, 2.69±0.33 and 3.48±0.25 fold increase of COD_S/COD_T ratios at MW intensities of 500, 800 and 1000 W, respectively compared to untreated.

3- The experimental results obtained in this work indicate that MS microwaved to 78^{0}C and power intensity of 1000W produced the greatest improvement in cumulative biogas production with 84% increase as compared to the controls after digestion for 33 days at sludge concentration of 1.46% TSS (w/w). Pretreatment significantly enhances the biodegradability of MS in the following steps of treatment, such as anaerobic digestion.

Nomenclature

BMP	Biochemical methane potential
CBP	Cumulative biogas production
COD_S	Soluble chemical oxygen demand , mg/l
COD_T	Total chemical oxygen demand, mg/l
MS	Mixed sludge
MW	Microwave
TSS	Total suspended solids, mg/l
TKN	Total kjeldahl nitrogen, mg/l
VSS	Volatile suspended solids, mg/l

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