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Effects of Microwave Heating on Biogas Production, Chemical Oxygen Demand and Volatile Solids Solubilization of Food Residues

Ackmez Mudhoo, Pravish Rye Moorateeah, Romeela Mohee

***Abstract*—**This paper presents the results of the preliminary investigation of microwave (MW) irradiation pretreatments on the anaerobic digestion of food residues using biochemical methane potential (BMP) assays. Low solids systems with a total solids (TS) content ranging from 5.0-10.0% were analyzed. The inoculum to bulk mass of substrates to water ratio was 1:2:2 (mass basis). The experimental conditions for pretreatments were as follows: a control (no MW irradiation), two runs with MW irradiation for 15 and 30 minutes at 320 W, and another two runs with MW irradiation at 528 W for 30 and 60 minutes. The cumulative biogas production were 6.3 L and 8.7 L for 15min/320 W and 30min/320 W MW irradiation conditions, respectively, and 10.5 L and 11.4 L biogas for 30min/528 W and 60min/528 W, respectively, as compared to the control giving 5.8 L biogas. Both an increase in exposure time of irradiation and power of MW had increased the rate and yield of biogas. Single-factor ANOVA tests ($p<0.05$) indicated that the variations in VS, TS, COD and cumulative biogas generation were significantly different for the pretreatment conditions. Results from this study indicated that MW irradiation had enhanced the biogas production and degradation of total solids with a significant improvement in VS and COD solubilization.

***Keywords*—**microwave irradiation, pretreatment, anaerobic digestion, food residues.

I. INTRODUCTION

SOLID wastes generation rates have become a major concern worldwide and different issues about their disposal are still arising. Solid wastes have an average composition, on a weight basis, of 45% of yard wastes, 26% of food wastes, 13% of plastics, 12% of paper, 3% of textiles and 1% of metals whereby the vegetable and food wastes account for more than 50% of the waste stream [1]. Food wastes is a fraction of solid wastes which can be defined as organic residues that have been generated by handling, storage, sale, preparation, cooking or serving of foods and which are intended or required to be discarded [2]. Food wastes can be categorised as pre-consumer food waste is food waste that is thrown away by staff within the control of the food service operator, and as post-consumer food waste is food which was primarily sold or served to customers, guests, students, patients and visitors but in the end is discarded as food wastes.

A. Mudhoo is Lecturer in the Department of Chemical and Environmental Engineering, Faculty of Engineering, University of Mauritius, Reduit, Mauritius (phone: +2307584279; fax: +2304657144; e-mail: a.mudhoo@uom.ac.mu).

P.R. Moorateeah is with the Chemical and Environmental Engineering Department, Faculty of Engineering, University of Mauritius, Reduit, Mauritius (phone: +2307014573; fax: +2304657144; e-mail: pravish.moorateeah@umail.uom.ac.mu).

R. Mohee was with the Chemical and Environmental Engineering Department, Faculty of Engineering, University of Mauritius, Reduit, Mauritius as Full Professor of Chemical and Environmental Engineering. She is now National Research Chair in Solid Waste Management in Mauritius (phone: +2304037857; fax: +2304657144; e-mail: rmh@uom.ac.mu).

Both aerobic and anaerobic treatments have been relatively promising solutions for decreasing the amount of solid wastes by providing treatment of a wide spectrum of wastes streams and at the same time producing renewable energy.

Anaerobic digestion (AD) is a promising solution for both waste management and energy production [3] because of its economic and environmental benefits [4]. Feedstock characteristics, lignocellulose content, reactor design and operation conditions affect the design and performance of an anaerobic process [5]. The first step of AD is hydrolysis and consists in the breakdown of cellulose, hemicellulose and lignin into monomers by the enzymatic action of hydrolytic fermentative bacteria. The resulting products are then converted during the acidogenesis process by acidogenic bacteria to acetic acid in the absence of oxygen. The final phase involves the action of methanogens on the acetic acid to produce biogas having a methane content of 40-60% by volume [6]. Food waste can also be digested anaerobically for biogas production [7]. The recalcitrant lignocellulosic content of the food wastes needs to be pretreated and then hydrolyzed during the rate determining hydrolysis step [8] before being digested by the enzymes/microorganisms [9]. Pretreatment is needed because the sugars necessary for biochemical reactions are trapped inside the crosslinking structure of the lignocellulose, thus inhibiting the process and decreasing yield [10]. To solve this problem and reduce the high cost of hydrolysis associated with anaerobic digestion processes, various physical, physicochemical, chemical and biological pretreatments have been actually employed [11]. Pretreatment methods such as microwave (MW) irradiations, sonication and electron beam irradiations (EB) are gradually immerging in the application of (green) radiation technology for biomass pretreatment. MW irradiation at low frequency has been shown to aid significantly in breaking down the complex floc structures of secondary sludge in order to cause the complex organic molecules of intracellular and extracellular components to unfold, denature, decrease in their size and eventually become more biodegradable [12]. Low-frequency MW irradiation pretreatment is also a heating technique offering advantages over conventional heating in terms of decreased process time, increase yields and environmental compatibility [13] and a more uniform heating of polar molecules while using less energy [14]. Bearing in mind the promise of MW irradiation, this study hence investigated the effects of MW irradiation as a pretreatment technique for the anaerobic digestion of food residues. The specific objectives were to set up of biochemical methane potential (BMP) assays using the microwave-heated substrates; to analyse the effects of different MW irradiation conditions on the AD process of the food residues; assessing the solubilization of the food residues during the AD process by monitoring total solids (TS), volatile solids (VS) and chemical oxygen demand (COD); and determining the cumulative biogas evolution from the BMP assays with and without MW irradiation.

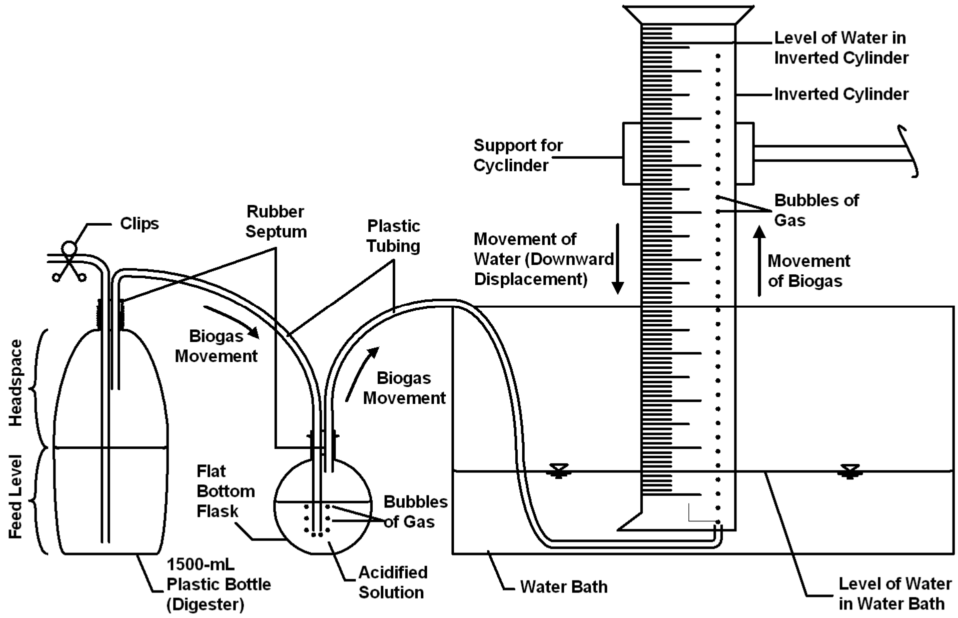

Fig. 1 Digester and biogas collection system comprising the biochemical methane potential assay

II. Materials and Methods

A. BMP assays and substrates

The objective of this study was to analyze the effects of MW irradiation pretreatment on food wastes under anaerobic conditions. For this purpose ten (10) 1.5-litre capacity BMP assays were set up (Fig. 1). The digesters used in the BMP assays were wrapped in black to prevent any photochemical effects on the AD process. Light is not lethal to methanogens but does inhibit the methanogenic process. Hence, AD should be performed in complete darkness [15]. Five BMP assays were used to monitor biogas production using a water displacement gas collection system, and the remaining assays being run in parallel were used solely to draw slurry samples for analytical testing of COD, TS and VS contents. The biogas from the digester was bubbled through an acidified solution (dilute sulphuric acid was used) to ensure that all the biogas generated was collected during the downward displacement of water [16]. All BMP assays were purged with nitrogen gas before being sealed to ensure anaerobic conditions. The substrate used for the whole study consists of mainly of residues of fried noodles (more than 70% by mass in mix fed to assays) and the rest comprising cooked chicken pieces, carrots cubes and flakes of cabbage leaves. These food residues were chopped and grounded in a in a mechanical grinder (Moulinex, TYPE 276, France) so as to obtain a homogeneous paste. By reducing the size of the food residues, a lower solids retention time could be obtained. The bacterial inoculum used in all BMPs was acclimated sludge obtained from an anaerobic digester from the Department of Chemical and Environmental Engineering at the University of Mauritius. This batch digester had been in operation for over 6 months using cow dung and market wastes as substrates.

The TS of the inoculum was 91.7±1.6%. A substrates paste to inoculum to water ratio of 1:2:2 was used throughout the experiments.

B. MW irradiation pretreatment conditions

The relatively low cost of modem domestic microwave ovens makes them reasonably readily available to academic and industrial chemists and engineers. However, somewhat surprisingly only a relatively small number of organic synthesis and biochemical research groups have reported their use. Besides that variable power levels are produced by simply switching the magnetron on and off, there are a number of useful reactions that have been carried out in a domestic microwave oven.

The literature shows a number of published methods for the safe direst use and/or modification of domestic microwave ovens for mediating chemical and/or biochemical reactions. In this study, a modern household 20-L capacity microwave oven (model Trust TMW- 200 M) with a maximum power input of 800W and operating at 2.45GHz was used to irradiate the food residues paste (i.e. substrates).

The first part of the experimental runs consisted in irradiating 220.04±5.09 g of substrates paste at a microwave power input of 320 W (i.e.40% × 800 W = 320W) for two time durations of 15 and 30 minutes in a propylene container adapted to the oven used in this work.

For the second part, the substrates were irradiated for 30 and 60 minutes at a microwave power input of 528W (66% × 800W = 528W). During the irradiation processes, the propylene container had been covered with the same polymer material so as to avoid any loss in moisture.

C. Analytical techniques

All analytical tests for TS and VS were carried out thrice a week. COD determination was performed once every week and biogas production was monitored daily. All analytical tests were done in triplicates. TS was determined using a gravimetric technique based on the final constant dry mass remaining after oven drying of samples at 105^{o}C for 18-24 hours in a hot-air drying oven (Memmert, model 800, Schwabach, Germany). VS content was determined by the loss on ignition BS 1377 method by burning the sample at 550^{o}C for 2 hours in an air-muffle furnace (Fisher Model 750-58, Fisher Scientific, Pittsburgh, PA). All masses were read using a top pan balance (model Mettler PM3000) with an accuracy of ±0.1 g. COD was determined using the standard potassium dichromate (VI)/ferrous ammonium sulphate titration method as per APHA [17].

D. Statistical analyses

For each of the parameters monitored during the AD process, error bars were inserted in their values by computing these errors from the absolute and relative errors ($\pm\Delta E/E$). Using SPSS Statistics 17.0, a Single-factor ANOVA test was performed on the net changes in TS, VS and COD to assess the effect of the pretreatment conditions on the AD process dynamics. Non-linear regression was employed to model cumulative biogas production by the modified Gompertz equation.

III. Results and Discussions

A. Effect of MW irradiation on TS and VS

From Fig. 2(a) and Fig. 2(b), it is observed that there was a decrease in TS and VS for both MW pretreated and non-pretreated substrates. Similar decreases in solids contents have also been reported by Meynell [18] and Uzodinma and Ofoefule [19] who explained that during anaerobic digestion, degradation caused by anaerobes would decrease the net available amount of TS and VS of organic wastes.

From Fig. 2(a), it may be inferred that a decrease in TS concentration from 10% (day 1) to 5.2% (day 10) had occurred over the first 10 days of the anaerobic digestion. The variations in TS for both MW pretreated and non-pretreated substrates indicated that the BMPs assays were low-solid systems since the TS was less than 10% [20]. Also, the gradual decrease in TS over the first 10 days indicated that anaerobic bacteria were able to readily degrade the most easily hydrolysable components in the food residues.

However, the quasi similar rates of decrease in TS also hinted that microbial consumption of TS had seemingly progressed independent of microwave irradiation for the experimental conditions tested in this work. There was a net decrease in TS for the MW pretreated substrates as compared to the control as supported by the calculated net decreases in Table I.

According to Cheng and Liu [21], as MW irradiation is increased, the loss in total mass of substrates also increases. The results obtained were as follows: BMP assay 1 irradiated for 15 mins at 320W showed a net decrease in TS of 76.77%, BMP assay 2 which was irradiated for 30 mins at 320W had a net decrease of 82.14%, BMP assay 3 irradiated at 538W for 30 mins showed an overall decrease of 88.81% of TS and BMP assay 4 irradiated at 538W for 60 mins had an overall decrease in TS of 91.54%. MW irradiation has been reported to make organic molecules more bioavailable. In this line of thought, the higher the intensity of the MW irradiation, a higher fraction of biodegradable molecules has been released and become more susceptible to as food for biodegradation to the microorganisms [22]. Data in Fig. 2(b) shows a gradual decrease in VS over the 10 days. Compared to the control, there was also a decrease in VS for all MW irradiated substrates. The net decrease in VS for the BMP assays increased with an increase in MW specific energy and exposure time relative to the control (Table I). The total masses of substrates in the BMP assays were essentially equal at 220.04±5.09 g with an average moisture content of 85.07±3.22% but the microwave specific energies developed as a result of MW irradiation were different because irradiation powers and times were different.

TABLE I
Net Percentage Decrease in VS, TS and COD

Pretreatment conditions for substrates	Microwave Specific energy (J/g)	Decrease in VS (VS_d/%)	Decrease in TS (TS_d/%)	Decrease in COD (COD_d/%)
control	-	52.05	70.45	46.15
15 min, 320 W	122.71	58.01	76.77	46.43
30 min, 320 W	245.41	63.94	82.14	50.01
30 min, 528 W	4319.22	68.45	88.81	52.63
60 min, 528 W	8638.43	72.81	91.54	54.76

B. Effect of MW irradiation on COD

COD tests were performed for a period of 12 days and the results have shown a net decrease in COD for all the BMP assays (Fig. 3). This decrease may be explained following the degradation of organic matter to gaseous products from the slurries during anaerobic digestion. Eskicioglu *et al.* [23] and Beszédes *et al.* [24] reported that MW irradiation has the ability to damage activated sludge floc structure and cell membranes as a result of which the release of extracellular and intracellular compounds such as proteins, sugars, and nucleic acid is increased and contribute ultimately to the COD fraction of the substrates. When both irradiation time and power were increased, the initial COD for the four pretreated assays increased *vis-à-vis* the control (Fig. 3). The high values of COD in the MW pretreated samples thus clearly indicated the presence of an elevated concentration of organic matter in the system during digestion period. Thus, the extent of solubilization of COD was also much seemingly dependent on the MW specific energy with the highest solubilization being for the highest specific energy. Following the anaerobic digestion process, the decrease in COD over the 12 days were as follows: 46.2% (control), 46.4% (BMP assay 1), 50.0% (BMP assay 2), 52.6% (BMP assay 3) and 54.8% (BMP assay 4) (Table I). It was also observed that there was an increase of 23.1% COD from day 1 to day 2 for the control. This was probably due to the action of hydrolytic enzymes in breaking down the substrate for digestion which resulted in an accumulation of organic matter up to day 2 [25].

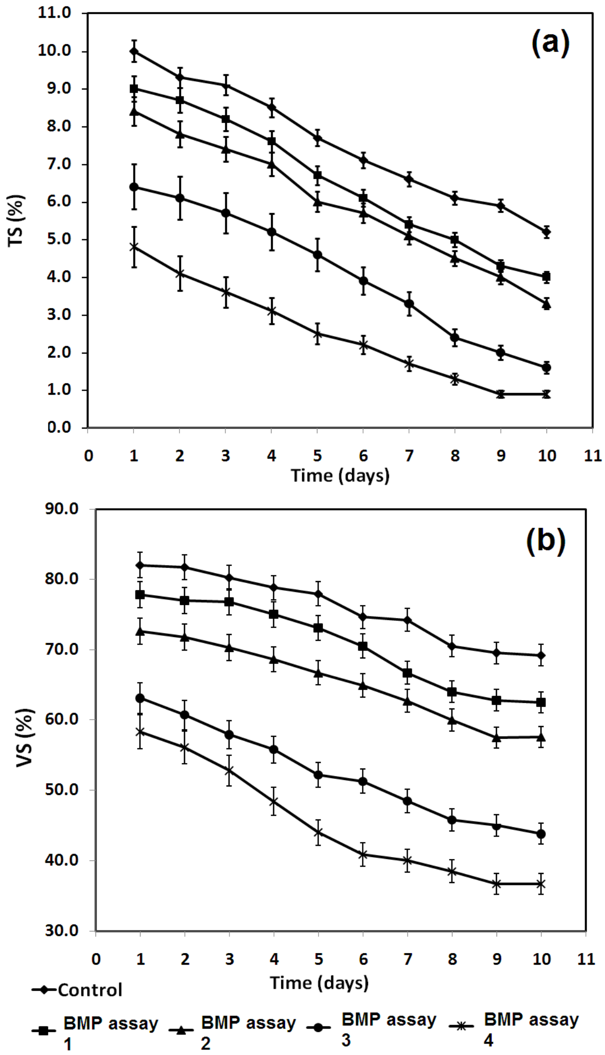

Fig. 2 Decrease in total solids (TS) and volatile solids (VS) for control experiment and MW irradiated test assays (BMP assay 1: 15min 320W, BMP assay 2: 30min 320W, BMP assay 3: 30min 528W, BMP assay 4: 60min 528W)

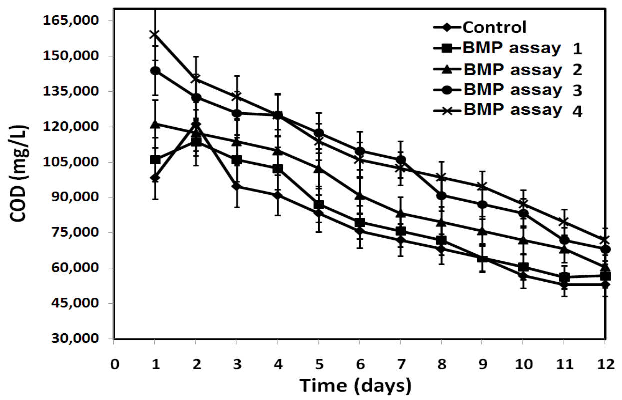

Fig. 3 Decrease in chemical oxygen demand (COD) for control experiment and MW irradiated assays (BMP assay 1: 15min 320W, BMP assay 2: 30min 320W, BMP assay 3: 30min 528W, BMP assay 4: 60min 528W)

C. Effect of MW irradiation on biogas production

From Fig. 4, it is observed that for all MW pretreated substrates, both the rates and cumulative biogas production were higher that for the control assay. The maximum cumulative biogas production were 5.80L (control), 6.30L (BMP assay 1 - pretreatment: 320W; 15 mins), 8.70L (BMP assay 2 - pretreatment: 320W; 30 mins), 10.50L (BMP assay 3 - pretreatment: 538W; 30 mins) and 11.40L (BMP assay 4 - pretreatment: 538W; 60 mins) for a period of 12 days. These observations for larger cumulative biogas productions could be attributed to an increased availability of intracellular materials in the slurries of the BMP assays as a result of cell wall disruption by MW irradiation. Similar effects were observed and so explained by Eskicioglu *et al*. [22] and Ahn *et al*. [26].

A further analysis of the biogas production in relation to the decreases recorded for VS, it can be observed that that the amount of biogas production has increased for larger decreases in VS degradation (BMP assay 4 had the highest biogas yield of 11.40L for the corresponding highest decrease in VS). This observation has also been shared by Kennedy *et al*. [14]. From Fig. 4, it is also observed that the initial lag phase for MW pretreated substrates was shorter than that of the control. This may be due to the readily more available soluble organic matter obtained from enhanced cell wall disruption caused by the MW irradiation. Also, with reference to Table I and the cumulative biogas production profiles in Fig. 4, the higher the MW irradiation specific energy, the higher have been the biogas production rates and yields. BMP assay 1 one has a specific energy of 122.71 J/g and produced 6.30 L of biogas whereas BMP assay 4 with the highest specific energy of 8638.43 J/g produced 1.81 times more biogas. The latter observations and inferences were supported by a kinetic analysis of the biogas production data for the control and four BMP assays (Fig. 4).

A review of literature has shown that the kinetic analysis of biogas, methane and biohydrogen production has been mostly performed using the modified three-parameter Gompertz equation (minimizing the sum of squared errors) for fitting the experimental data of accumulative biogas, methane and hydrogen production [27]-[29]. Equation (1) gives the modified Gompertz equation where $B(t)$ is the cumulative biogas production (L) during the fermentation time t(day), P the (maximum) biogas production potential (L), R_m the maximum production rate (L/day), λ the lag−phase time (day), and e is 2.7182818. The values reported in Table II for P, R_m and λ for data reported in Fig. 4 were determined by best fitting the experimental biogas producing data using the non-linear regression approach with the aid of the MATLAB 7.9 model software function at a 95% confidence interval.

$$B(t) = P \times \exp\left\{-\exp\left[\frac{R_m e}{P}(\lambda - t) + 1\right]\right\} \quad (1)$$

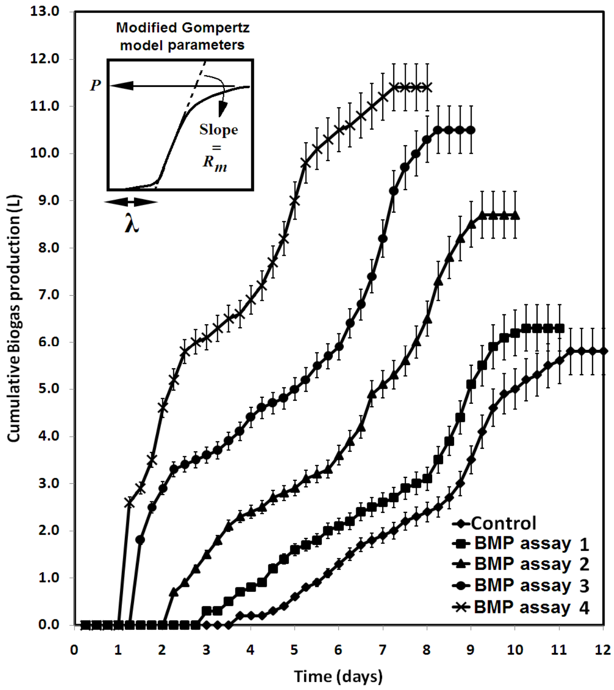

Fig. 4 Cumulative biogas production for control BMP assay and MW irradiation pretreated test assays (BMP assay 1: 15min 320W, BMP assay 2: 30min 320W, BMP assay 3: 30min 528W, BMP assay 4: 60min 528W)

TABLE II
RESULTS OF NON-LINEAR REGRESSION FOR CUMULATIVE BIOGAS PRODUCTION DATA FITTED TO THE MODIFIED GOMPERTZ EQUATION

Assay	*P* (L)	R_m (L/day)	*λ* (days)	R^2
control	5.582 ±0.811	0.961±0.026	5.13±0.11	0.9892
BMP assay 1	6.622 ±3.003	1.057±0.071	4.45±0.34	0.9823
BMP assay 2	9.372±3.627	1.367±0.080	3.17±0.31	0.9842
BMP assay 3	11.681±7.068	1.460±0.127	1.57±0.52	0.9560
BMP assay 4	12.201±0.537	1.583±0.237	1.37±0.24	0.9469

The values of R_m and λ in Table II clearly indicate that MW irradiation pretreatment had improved the rate of biogas production and reduced the lag−phase time. R_m for the control assay was 0.961 L/day and had a lag-phase time of 5.13 days whereas for the BMP assay with the highest specific energy (BMP assay 4), R_m has increased to 1.583 L/day and the lag−phase time decreased by 3.76 days.

A Durbin-Watson test was requested during the non-linear regression modeling to determine the random normal distribution of residuals (i.e. the difference between the estimated and the experimental data). The values of the Durbin-Watson statistic had varied from 0.242 to 0.258 for the control experiment through to BMP assay 4, respectively, and which were less than the lower critical values (D_L) which ranged from 1.42 to 2.56. This provided statistical evidence that the error terms were positively autocorrelated and all residuals had a random normal distribution of less than 5% random error. Based on the results of the statistical curve fitting, the modified Gompertz equation was observed to adequately describe cumulative biogas production with very high goodness of fit (R^2) values for low solids systems.

D. Statistical analyses and comparison of pretreatments

The effects of MW irradiation pretreatment conditions on the cumulative biogas production and net percentage changes in VS, TS and COD values (Table I) for the untreated and pretreated substrates have been compared through a Single–Factor ANOVA analysis (p<0.05). The ANOVA test statistics (Table III) have revealed that the null hypothesis made by ANOVA was rejected for TS, VS, COD and biogas production. Since the null hypothesis by ANOVA states that the datasets in the two columns are same and come from the same population, rejection of the null hypothesis implied that the pretreatment conditions have resulted in different sets of parameter variations for TS, VS, COD and biogas yields. This inference was supported by the test result F–values of the ANOVA tests for the 5 pretreatment conditions (treated and control) whereby the F–test statistic was greater than the critical value for F (F_{crit}) for all process parameters monitored over the duration of the AD processes till no more biogas was evolved.

TABLE III
SINGLE-FACTOR ANOVA RESULTS (p<0.05)

statistical parameter	VS	TS	COD	Biogas
F-statistic	11.26	10.41	12.02	15.88
F-critical (F_{crit})	10.13	10.13	10.13	10.13

IV. CONCLUSION

MW irradiation was found to have a positive effect on the overall anaerobic digestibility of the food residues. MW irradiation pretreatment enhanced the solubilization of VS and COD and concomitantly increased the rates and cumulative production of biogas.

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Ackmez Mudhoo, MPhil is Lecturer in the Department of Chemical and Environmental Engineering at the University of Mauritius. He obtained his Bachelors degree (B.Eng. (Hons.)) in Chemical and Environmental Engineering in 2004 and a Masters degree (Master of Philosophy) in Chemical Engineering in 2011, both from the University of Mauritius. His research interests encompass the design and analysis of composting processes and treatment of solid wastes and wastewaters by anaerobic digestion. Ackmez has 54 international journal publications, 5 conference papers and 5 edited books with CRC Press, RSC Publishing, Springer and Scrivener Publishing LLC to his credit on topics related to green chemistry and technology, bioremediation and anaerobic digestion. Ackmez serves as scientific reviewer for Waste Management, Critical Reviews in Biotechnology, Chemical Engineering Journal and Chemosphere.

Pravish Rye Moorateeah, BEng (Hons.) earned and holds a Bachelors degree (B.Eng. (Hons.) in Chemical and Renewable Energy Engineering from the University of Mauritius in 2012. His research interests are in biomass pretreatment for biogas production by using low frequency microwave irradiation to organic fractions derived from municipal solid wastes for subsequent anaerobic digestion.

Romeela Mohee, PhD is Professor of Chemical and Environmental Engineering. She has been Dean of the Faculty of Engineering and also with the Department of Chemical and Environmental Engineering at the University of Mauritius. She obtained her PhD at the University of Mauritius in 1998. Being a Graduate Engineer in Energy Engineering from the Institut National des Sciences Appliquées (**INSA**) de Lyon (France) in 1989, she has widely developed her research interests in waste containment, solid waste management and treatment by anaerobic digestion and composting, beneficial reuse of waste materials, and heat and mass transfer modeling. Professor R. Mohee has over 70 international publications on waste biotreatment, 5 books, numerous international conference papers and consultancy reports related to environmental sustainability and clean development. She is presently the National Research Chair in Solid Waste Management in Mauritius.