Enhancement of methane production of microwaved pretreated biowaste at different enthalpies

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Abstract:

The aim of this study was to enhance the anaerobic biodegradability and methane production of a synthetic Organic Fraction of Municipal Solid Waste (OFMSW) with high lignocellulosic content by assessing microwave at different enthalpies. The pretreatment was tested on two different amounts of substrate (0.25 and 0.50 kg) with different irradiation times (ranging between 120 and 960 s). Biochemical Methane Potential (BMP) assays were performed for 21 days. The cumulated methane production and the energy balance of the pretreatments were used to evaluate the efficiency of microwaving on the anaerobic digestion process. Results showed a BMP_{21} increasefor all the tested conditions with the highest increase of about 30.7% and 32.7% recorded for the substrates subjected to the most intense treatments (1632 kJ/kg).Furthermore, with the increase of the energy demand of the pretreatment a relative increase in biogas production was observed. Despite this beneficial effect, the total energy balance was always registered negative, due to laboratory scale conditions.

Keywords:

Anaerobic digestion, Biogas, Energy balance, Microwave, Organic Fraction of Municipal Solid Waste, Thermal pretreatment.

1. Introduction

Bioenergy is defined as a renewable form of energy derived from organic materials with lower emissions than traditional fossil fuels [1]. Nowadays the scientific community is focused in drawing new borders for the production of biofuels as bioethanol [2, 3]and biogas from organic recalcitrant and non-recalcitrant materials in order to gain valuable renewable energies. Under this perspective Anaerobic Digestion (AD) is an efficient organic waste treatment that has gained interest during the last years as it recovers energy in the form of biogas for use in combined heat and power plants.

The Organic Fraction of Municipal Solid Wastes (OFMSW) often includes a high content of lignocellulosic fiber that is not readily digestible. Plant fiber of yard waste typically comprises around 30% hemicellulose, 45% cellulose, and 25% lignin on a dry weight basis [4]. The encasing of cellulose and hemicelluloses in lignin may considerably restrict anaerobic degradation in which the limiting factor is the hydrolytic phase due to constrained accessibility of particulate substrates by enzymes and/or the complexity of compounds that need to be hydrolyzed [5]. The rupture of the complex structure is essential for enzymatic attack and efficient bioconversion to processes such as hydrolysis, fermentation and biomethanogenesis. Pretreatments of OFMSW can be used to solubilize organic matter prior to AD in order to improve the overall AD process in terms of faster rates and degree of OFMSW degradation, thus increasing methane production [6, 7]. Moreover, substrate pre-treatments have been shown to be a useful step to enhance aerobic biodegradation processes as composting [8]and for pathogens destruction [9].

Several methods have been assessed for their technical feasibility at pre-treating residues[10]. These include enzymatic[10], chemical [12], ultrasonic [13,14], thermal [15, 16], hydrothermal [17, 18]and microwave treatments. The present research focuses its attention on this latter method in order to study the anaerobic biodegradability and methane production of a rich lignocellulosic OFMSW by assessing Microwaving (MW).

Microwave irradiation is an electromagnetic radiation with a wavelength between 1 mm and 1 m, corresponding to an oscillation frequency of 300–0.3 GHz[19, 20]. Domestic "kitchen" microwave ovens and industrial microwave generators are generally operating at a frequency of 2.45 GHz with a corresponding wavelength of 12.24 cm and energy of 1.02·10-5 eV [8, 19]. MW is an alternative method to conventional thermal pretreatments as it is able to synthesize organic molecules. The cell liquor and extracellular organic matter within polymeric network can release into the soluble phase increasing the ratio of accessible and biodegradable components. The main factors influencing the treatment are temperature, power and irradiation time. The range of application of the power is between 440-500 W [21-23] and 1250 W [9, 20, 24] while the applied temperature covers a wide range of values: from 30°C [25] to 175°C [24]. The irradiation time is generally found to be in the order of few minutes (1-10 minutes) even if some works present irradiation time higher than 40 minutes[7, 24]. Although this, MW with high temperatures (above 170°C) and long irradiation time could lead to the formation of refractory compounds inhibiting the digestion [7, 24].

The enhancement of biogas production due to the application of pre-treatments is generally analyzed through Biochemical Methane Potential (BMP) tests [7, 8, 17, 20-26].

The objective of this work is therefore to study the enhancement of the anaerobic biodegradability and methane production of a synthetic OFMSW with a rich lignocellulosic content (M) by assessing microwave at different enthalpies. As such, BMP assays were performed for 21 days. The cumulated methane production (BMP_{21}) and the energy balance of the pretreatment were used to evaluate the efficiency of MW onAD process.

2. Materials and Methods

2.1 - Biowaste and inoculum

M, a syntheticsample of OFMSW with high lignocellulosic content, was assessed. M was characterized by (on weight basis, % w/w): fir sawdust (10%), grass (30%), carrot (10%), cabbage (10%), spinach (10%), cooked meat (7.5%), raw meat (7.5%) and cooked pasta (15%). Pasta and meat were cooked for 10 minutes and then strained. For each fraction proteins, carbohydrates, lipids and fibers contents were known. Their average values expressed in % w/w were respectively 6.1%, 4.0%, 12.1% and 11.0%. In order to reduce the particle size to 3 mm diameter each fraction was treated in a food processor and sift with a strainer. Supplemental tap water was then added to M leading to a mash in order to guarantee a total solids (TS) content suitable for wet technology range (8-12% TS). Dilution ratios was determined as 1.7 l/kg. The mash was then stored at 4°C until use. M characterization is presented in Table 1. Digested sludge from an anaerobic reactor treating OFMSW was used as the mesophilic inoculum. It had a pH of 7.85 while TS and Total Volatile Solids (TVS) contents were about 3.3±0.1% (w/w) and 62.1±0.1% on TS basis respectively.

2.2 - Microwave pretreatment

A commercial domestic microwave oven (2450 MHz frequency, 850 W) was used to irradiate M mash. The microwave heating was performed in batch placing the substrate in a closed vessel to avoid losses caused by hot spot formation during the treatment [19, 22]. In order to evaluate the pretreatment efficiency, different amounts and irradiation times were tested leading to different applied enthalpies. Substrates are then stored at 4°C until use. Pretreatment conditions (samples amount, irradiation time, enthalpy of the pretreatment and reached temperature) and the characterization of microwaved samples (TS, TVS/TS and pH) are presented in Table 1.

Table 1. Pretreatment conditions (samples amount, irradiation time, enthalpy of the pretreatment and reached temperature) and substrates characterization (TS, TVS/TS and pH).

	Sample amount [kg]	Time [s]	Enthalpy [kJ/kg]	T [°C]	TS [%]	TVS/TS [%]	pH
M	0	0	0	0	8.45 ± 0.22	96.39 ± 0.08	3.69 ± 0.01
MW 500/4	0.50	240	408	96.0	8.60 ± 0.24	96.63 ± 0.09	3.65 ± 0.01
MW 500/8	0.50	480	816	99.3	9.41 ± 0.09	96.74 ± 0.11	3.64 ± 0.01
MW 500/16	0.50	960	1632	100.8	12.89 ± 0.03	97.37 ± 0.13	3.28 ± 0.01
MW 250/2	0.25	120	408	85.5	8.95 ± 0.07	96.65 ± 0.07	3.68 ± 0.01
MW 250/4	0.25	240	816	96.4	9.54 ± 0.31	96.74 ± 0.14	3.65 ± 0.01
MW 250/8	0.25	480	1632	98.7	12.98 ± 0.29	96.62 ± 0.13	3.49 ± 0.01

2.3 - Analytical parameters

TS, TVS and pH were determined in order to characterize the inoculum and each substrate according to standard methods[27]. According with [28], due to the acidic condition of each substrate, TS determination was performed at 90°C instead of 105°C until constant weight in order to avoid the volatilization of volatile fatty acids (VFA).

2.4 - Specific energy demand

The specific energy demand (E_D) was calculated taking into account the power of the microwave heating system as well as the exposure time applied for each treatment. E_D (kJ/kgTVS) was calculated according to (1):

$$E_D = \frac{P_D \cdot t_D}{M_{TVS}} \qquad (1)$$

where:

- P_D: power of microwave generator or hot plate (kW);
- t_D: exposure time (s);
- M_{TVS}: mass of treated mash (kgTVS);

2.5 - Energy demand and profit of the pre-treatment

According with [25]specific energy profit of the pre-treatment E_T (kJ/kgTVS) was calculated taking into account the E_D of the pretreatment, the energy produced in the form of biogas and the theoretical amount of energy produced in the form of heat (2):

$$E_T = E_B + E_Q - E_D \qquad (2)$$

where:

- E_B: amount of energy produced in the form of biogas after subtracting the energy generated by raw substrates (kJ/kgTVS);
- E_Q: amount of energy produced in the form of heat (kJ/kgTVS);
- E_D: amount of energy used for samples pretreatment performed in certain conditions (kJ/kgTVS).

E_B was based on an average CH_4 energetic value of 37 kJ/dm^3 and BMP_{21}. E_Q (kJ) was calculated as follows (3):

$$E_Q = M_{TVS} \cdot C_p \cdot \Delta T \qquad (3)$$

where:

- M_{TVS}: the mass of substrate equivalent to unit of volatile solids;
- C_p: the specific heat capacity of substrates (kJ/kg·°C);
- ΔT: the temperature difference between the mash after pretreatment and the temperature (37°C) of the mesophilic digestion.

C_p was based on ratio of water and solids. The values of C_p used for calculations amounted to 4.18 and 1.95 kJ/kg°C for water and solids respectively [25, 29].

2.6 - BMP assays

BMP assays were performed for 21 days in order to determine the BMP_{21}. BMP_{21} were expressed as biogas volume (L) produced and measured at normal conditions (T=273.15 K, P=1 atm) per kg of TS and TVS. The analysis were conducted using a modified method of [30]following the basic guidelines and advices included in [31]. The BMP was determined using stainless steel bottles (1 L, 2 bar proof pressure) manufactured at DIEF (Department of Industrial Engineering of Florence,[32]) incubated in a water bath at 37°C tightly closed by a special cap provided with a ball valve to enable the gas sampling. After set-up the bottles were flushed with inert gasto ensure anaerobic conditions in the headspace of the batches.

The triplicate of the different substrates previously described was performed. Each reactor was loaded with different amounts of substrate, depending on the characteristics of the materials, to achieve a concentration of substrate of about 2 gTVS/100 ml solution in each batch. This concentration is a compromise of, one hand, the need to use a large sample to have a good representativeness and to get a high easy-to-measure gas production, and, on the other hand, to avoid too large and impractical volumes of reactors and gas production and keep the solution dilute to avoid inhibition from accumulation of VFA and ammonia [33]. The inoculum to sample ratio was kept under 10:1 weight ratio. This was set as the optimal ratio according to previous researches performed on the same substrate and according with [30]for fresh feed-in substrates. In order to determine the background methane production a blank assay with only the inoculum was done in triplicate. The inoculum response toward a “standard” substrate (control vessels) was checked in duplicate with cellulose with a concentration of 1gTVS/100 mL solution. The inoculum was degassed for 5 days in order to deplete the residual biodegradable organic matteruntil the achievement of an endogenous metabolism phase[31].

Biogas production was estimated by measuring the pressure in the head space of each reactor and then converting to volume by the application of the ideal gas law. Pressure was measured using a membrane pressure gauge (Model HD2304.0, Delta Ohm S.r.L., Italy). The measured values of pressure were converted into biogas volume as following (4):

$$V_{biogas} = \frac{P_{measured} \cdot T_{NTP}}{P_{NTP} \cdot T_r} \cdot V_r \qquad (4)$$

where:

- V_{biogas}: volume of daily biogas production, expressed in Normal litre (NL);
- $P_{measured}$: headspace pressure before the gas sampling (atm);
- $T_r$andV_r: temperature (K) and volume (L) of the reactor’s headspace;
- T_{NTP} and P_{NTP}: normal temperature and pressure, (273.15 K and 1 atm respectively).

The gas produced was routinely analysed using an IR gas analyser (ECOPROBE 5 – RS Dynamics). The bottles were daily shaken to guarantee homogeneous conditions in the assay vessels [31].

The BMP was determined as the cumulate biogas production, calculated as the sum of the daily volumes, divided by the TS and the TVS content contained in each batch. Results reported at normal temperature and pressure were obtained after 21 days and presented as GB_{21} (gas production sum,[34]) and BMP_{21}.

The increase in BMP_{21} was calculated as given in the following equation (5):

$$\Delta BMP_{21}(\%) = \frac{(BMP_{21_{after\ pretreatment}} - BMP_{21_{before\ pretreatment}})}{BMP_{21_{before\ pretreatment}}} \times 100 \quad (5)$$

3. Results

3.1 - BMP_{21}

Fig. 1 represents the methane production profiles of each substrate, blank and control (cellulose) assays [31] on TVS basis. Table 2 shows the results of the BMP test with GB_{21}, BMP_{21} on TS and TVS basis and mean methane content.

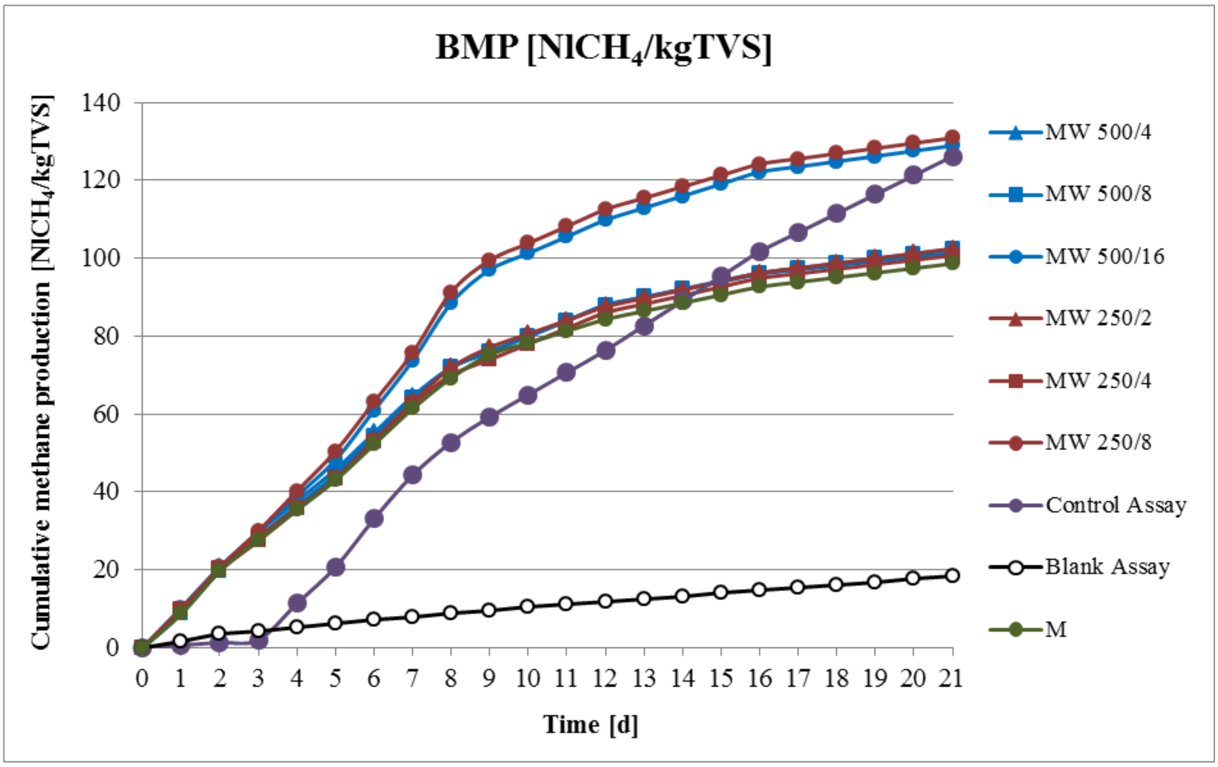

Fig. 1.Mean BMP test curves expressed in $NlCH_4/kgTVS$ for the 21 days with control and blank assays

Table 2. Anaerobic biodegradability assays results in terms of GB, BMP and mean methane content

	M	MW 500/4	MW 500/8	MW 500/16	MW 250/2	MW 250/4	MW 250/8	Control
CH_4 [%]	63.8 ± 1.8	62.4 ± 2.3	64.4 ± 2.3	65.4 ± 3.5	64.1 ± 2.1	63.6 ± 2.4	64.6 ± 2.4	49.1 ± 0.4
GB_{21} [NL/kgTS]	119.5 ± 1.6	122.0 ± 2.7	123.5 ± 0.1	154.8 ± 8.7	124.3 ± 7.5	131.8 ± 8.8	158.1 ± 11.5	185.6 ± 2.1
GB_{21} [NL/kgTVS]	173.8 ± 2.3	177.2 ± 3.9	177.3 ± 0.1	215.0 ± 12.1	179.1 ± 10.8	189.9 ± 12.6	221.3 ± 16.1	268.0 ± 4.0
BMP_{21} [$NLCH_4$/kgTS]	67.8 ± 1.6	69.9 ± 0.1	71.3 ± 0.9	92.8 ± 6.1	71.1 ± 5.5	75.2 ± 7.5	93.5 ± 9.2	87.4 ± 1.2
BMP_{21}[$NLCH_4$/kgTVS]	98.7 ± 2.3	101.5 ± 0.1	102.4 ± 1.3	129.0 ± 8.4	102.5 ± 7.9	108.4 ± 10.8	130.9 ± 12.9	126.2 ± 2.6
ΔBMP_{21} [%]	-	2.9	3.8	30.7	3.9	9.9	32.7	-

BMP assays showed the typical trend of the test withoutthe occurrenceof any acidification process. Control test showed a mean BMP_{21} values of 126.2 $NLCH_4$/kgTVS which underlines a good quality response of the inoculum to a standard substrate as cellulose. The mean methane content ranged between 62.4% and 65.4% for all the tested samples, typical of an energy rich biogas(values in theoptimal range defined by[19, 35]between 55%and 70%).

All treatments noticed an increase in methane production compared to the blank substrate M. This is in agreementwith what reported in previous batch studies [20, 23, 25]. In particular, the enhancement in methane production occurredat increasing applied enthalpies with the highest result observed for MW 250/8 with a mean value of 32.7%. Moreover,treatments performed on the smaller amount (0.25 kg) determined slightly higher methane productions compared to treatments performed on 0.50 kg of substrate. These behaviors are probably attributable to a better application of the treatment to the mass of substrate. In this way a strongersolubilisation effect is produced leading to a sample compositionwhich is more suitable for anaerobic bacteria.The solubilisation effect of the pretreatments was confirmed by the lower pH found after all treatments reported in Table 1 that could be associated to a release of organic acids during the process [17].

3.2 - Specific energy demand and profit of the pre-treatment

Energy efficiency is a crucial factor influencing the economic feasibility and justifying the substrate pre-treatment [25]. E_D, E_B, E_Q and E_T for the different treatments and substrates are presented in Table 3.

Table 3. Energy demand and profit of the pretreatments.

	E_B [kJ/kgTVS]	E_D [kJ/kgTVS]	E_Q [kJ/kgTVS]	E_T [kJ/kgTVS]
MW 500/4	105	5101	2944	-2052
MW 500/8	138	10202	3109	-6955
MW 500/16	1121	20404	3184	-16099
MW 250/2	142	5101	2420	-2539
MW 250/4	360	10202	2964	-6877
MW 250/8	1194	20404	3079	-16130

Analysing the specific energy balance, no energy profits were registered for all treatments. This was mainly due to the low increase in biogas production compared to raw substrate digestionand to laboratory scale conditions. The amount of E_B and E_Q was not enough to balanceE_D. Even if with negative results, lighter treatments showed better energetic response than more aggressive one due to a lower E_D of the treatment. Other studies showed relevant increase in total energy [25] leading to the conclusion that further investigations with different pre-treatment conditions and on pilot scale conditions are necessary to examine the feasibility of such pre-treatments on lignocellulosic OFMSW.

As the enhancement in methane production occurred at increasing applied enthalpiesE_B was plotted in function of the E_D leading to a parabolic trend with a coefficient of determination (R^2) of about 0.97 which guarantees a good approximation of the model (Fig. 2).

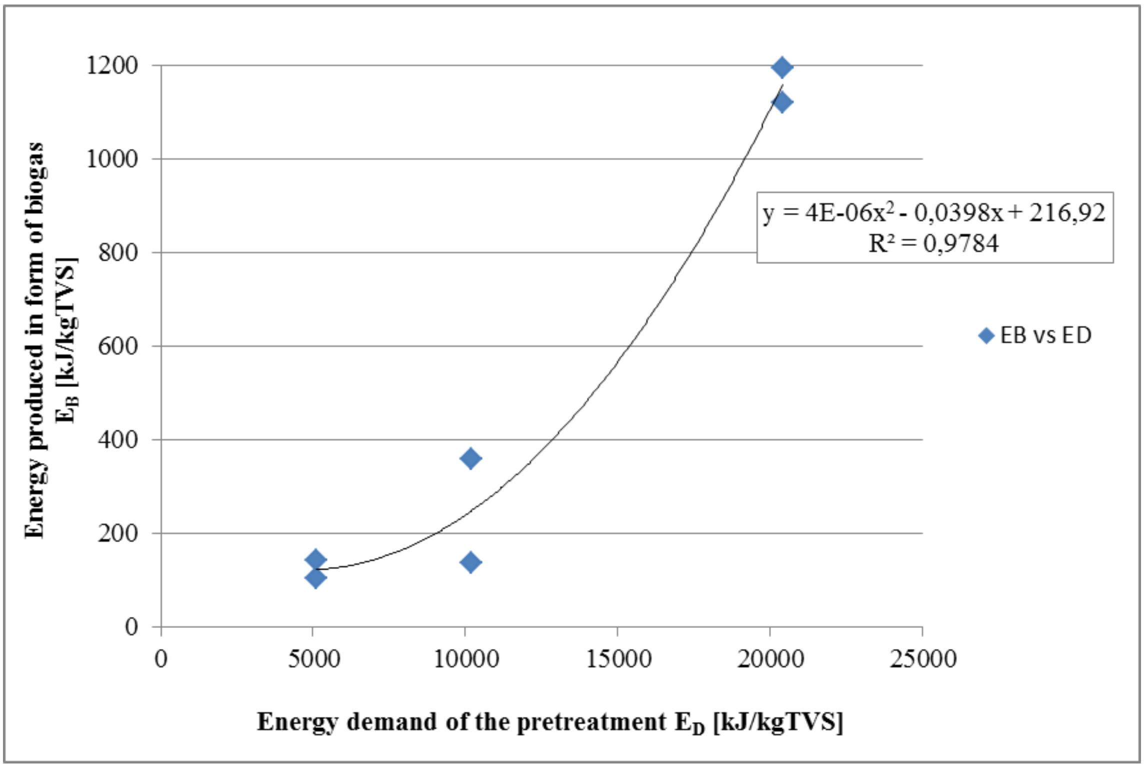

Fig.2. Correlation between the energy demand of the pretreatment (E_D) and the energy produced in form of biogas (E_B)

4. Conclusions

MW with the tested enthalpies (408, 816 and 1632kJ/kg) is an efficient pretreatment method to enhance methane production of rich lignocellulosic OFMSW. The research was carried out in batch mode for 21 days analysing GB_{21}, BMP_{21}, mean methane content and the energy balance of the treatment. Results showed a BMP_{21} increase for all the tested conditions compared to untreated samples with the highest increases of about 30.7% and 32.7% recorded for the substrates subjected to the most intense treatments (1632 kJ/kg).In particular, the enhancement in methane production occurred at increasing applied enthalpies with a parabolic correlation between the energy produced in the form of biogas and the energy demand of the pretreatment.This behavior is attributable to the solubilisation effect of the treatment that leads to a sample compositionmore suitable for anaerobic bacteria.

Despite this beneficial effect, no energy profit was recorded for any tested pretreatment due to the low increase in biogas production and to laboratory scale conditions.

Further investigations with different treatment conditions and on pilot scale are required to better probe the pretreatment efficiency on the AD of OFMSW.

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Nomenclature

BMP_{21}biochemical methane potential, $NLCH_4$/kgTS or $NLCH_4$/kgTS

CH_4methane content, %

C_p specific heat capacity, kJ/(kg °C)

$E_B$energy produced in the form of biogas, kJ/kgTVS

E_D energy demand of the pretreatment, kJ/kgTVS

E_Q energy produced in the form of heat, kJ/kgTVS

$E_T$energy profit of the pretreatment, kJ/kgTVS

GB_{21} gas production sum, NL/kgTS or NL/kgTVS

M_{TVS} mass of substrate, kgTVS

p pressure, atm

P_D power of microwave generator, kW

R^2 coefficient of determination

T temperature, °C

t_D exposure time, s

TS total solids, %

TVS total volatile solids, %

V volume, L

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