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# Effects of temperature, proportion and organic loading rate on the performance of anaerobic digestion of food waste

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# ABSTRACT

In Brazil, a significant amount of organic waste is produced in households and restaurants. This study thus aimed to determine the ideal conditions for generating methane from the treatment of household waste by anaerobic digestion, under mesophilic (37 °C) and thermophilic (55 °C) conditions, to determine the maximum organic loading rate (OLR) in the reactors, and to evaluate kinetic parameters by statistical models: Modified Gompertz, First-Order, Logistic and Transference functions. The experiments were conducted in anaerobic batch reactors. Different proportions of pre-prepared waste (PPW)/leftover waste (LW) were used: 100/0, 75/25, 50/50, 25/75, and 0/100 and different ORL: 0.15; 0.30; 0.45; 0.60; and 0.90 g TVS (Total Volatile Solids).L<sup>-1</sup>.d<sup>-1</sup>. For both conditions, the optimal proportions of PPW/LW were 100/0 and 75/25 %. Under mesophilic condition, the best results were observed (869 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>). The maximum organic load was 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup>. The best data adjustment was performed by the Transference function.

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# 1. Introduction

The use of fossil fuels is responsible for the atmospheric emission of most of the Greenhouse Gases of anthropic origin. The negative impacts associated to this energy source could be reduced through a better use of renewable energies. In this sense, biomass and organic wastes play a fundamental role in mitigating these adverse effects and may contribute to energy production [1].

According to the Food and Agriculture Organization of the United Nations (FAO) and the Pan American Health Organization (PAHO), about 1.3 billion tons of food are lost per year in the segments of agricultural production, post-harvest, and processing, or wasted in retail sales and final consumption through the food supply chain for human consumption. In Latin America and the Caribbean in 2016, about 127 million tons of food were wasted [2].

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In Brazil, there are estimated 200 g per capita a day of food waste in the areas of agricultural production, post-harvest and processing [3], which generates about 15.3 million tons a year. Food waste in retail activities and final consumption generate organic matter of 51.4 % of the total gravimetrical composition of the Municipal Solid Waste (MSW) in the country [4]. This represents about 40.2 million tons a year. Gathering losses and wastes in Brazil, approximately 55.5 million tons a year of organic waste are generated.

The data collection/survey conducted by the National Sanitation Information System [5] inferred that, in 2016, from the total amount of organic waste collected in Brazilian cities, 59.0 % are disposed in landfills, 9.6 % in controlled landfills, 10.3 % in dumping grounds, 3.4 % are taken to composting units and 17.7 % of the municipalities have not provided any information. Considering the negative environmental impacts of disposing of organic wastes in landfills, of incineration, and the low energy yield from composting food waste [6,7], anaerobic digestion has been proposed as an alternative for generating clean energy and for treating waste with high moisture content [8].

In comparison to other processes, anaerobic digestion has the advantages of adapting to different work scales, treating a wider

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range of substrates, even with high or low moisture and impurities content [9], resulting in low environmental impacts [10] producing material able to be used as biofertilizers [11] and having high energy recovery potential [11–14].

Although anaerobic digestion is a process widely employed in treating residual waters and sewage sludge, the treatment of food waste still faces technical and economic challenges in many countries. Among the latter, there is the instability of the process, the accumulation of volatile fatty acids (VFA), high cost of transportation and operation [15], reduced efficiency in the production of biogas and in the removal of volatile solids, the long time required for stabilizing the organic matter [16], high organic carrying rate and reduced percentage of solids applied to reactors [17], besides the difficulty in controlling the origin and the seasonality of the substrates [18].

Globally, there is an estimated production of 2 billion tons a year of MSW, with a content up to 53 % of biodegradable organic wastes (defined by the organic fraction of municipal solid waste (OFMSW)), which mainly come from restaurants and households, and have high treatment cost, estimated in about U\$ 900 billion [19]. Thus, not only does the recovery of energy and nutrients from food waste constitute substantial economic opportunities, but also performs an essential requirement for the development of society [15].

In this sense, this paper aimed to determine the ideal condition for treating household waste by anaerobic digestion, to evaluate the potential of methane generation under mesophilic (37 °C) and thermophilic (55 °C) conditions, to determine the maximum organic loading rate to be used in the reactors and to evaluate kinetic parameters by statistical models: Modified Gompertz, First-Order and Logistic and Transference functions.

# 2. Material and methods

# 2.1. Inocula source and substrates

Two granular sludges from Upflow Anaerobic Sludge Blanket reactors have been used as inocula sources : (1) mesophilic for treating sewage sludge from a poultry farm in the city of Tietê/SP, Brazil; and (2) thermophilic for treating vinasse from sugarcane and alcohol production (Pradópolis/SP, Brazil). Granular sludges (1) and (2) were maintained at 4 °C until acclimatization. During acclimatization, the inocula were kept at 25 °C for 60 days with additions of acetate (2 g.L<sup>-1</sup>) every seven days for maintaining the methanogenic activity of microorganisms.

Food waste generated at the university restaurant (UNESP, campus of Rio Claro/SP, Brazil) was used as substrate. The total mass collected (% on a natural moist basis) consisted of PPW (Pre-Preparation Wastes) from the food (kale – 12 %, zucchini – 5%, escarole – 5%, eggplant – 9%, chayote – 10 %, cabbage – 10 %, carrot – 11 %, and

beet – 10 %) and LW (Leftover Wastes) from meals (rice – 11 % and beans – 14%). The typical PPW/LW ratio generated in the restaurant is 75/25 %.

The mixtures of wastes were diluted in the proportion 1:1 (1.0 kg of waste for 1.0 L of distilled water), crushed in industrial blender to reduce the size of the particles, and kept at -20 °C. The inocula and substrates were characterized by solids (TS - total solids, TVS - total volatile solids, and TFS - total fixe solids) pH, BOD (Biochemical Oxygen Demand) and COD (Chemical Oxygen Demand) according to APHA [20]; total alkalinity, and volatile fatty acids [21], total carbohydrates [22], and C/N ratio [23].

# 2.2. Biochemical Methane Potential (BMP) tests under mesophilic and thermophilic conditions

The BMP tests were conducted in triplicates in anaerobic batch reactors (500 mL with 250 mL of reactional volume). Five mixtures of food wastes were tested in the proportions PPW/LW: 100/0 (M1), 75/25 (M2), 50/50 (M3), 25/75 (M4), and 0/100 % (M5). The compositions of substrate (S) and inoculum (I) are shown in Table 1. A mass ratio of 1.5 g substrate (in TVS) for 1.5 g inoculum (TVS) were added for the tested conditions.

The initial pH was kept at 7.0 with a sodium hydroxide solution (0.1 N) and sulfuric acid (0.1 N). The system was buffered with monobasic potassium phosphate (300 mg.L<sup>-1</sup>) and dibasic potassium phosphate (400 mg.L<sup>-1</sup>) according to Aquino et al. [24].

The headspace of the reactors was filled with Nitrogen gas (100 %) for 20 min to maintain the anaerobic conditions. They were kept at 37  $\pm$  1 °C (mesophilic test) and at 55  $\pm$  1 °C (thermophilic test). They were sealed with rubber lids, plastic thread with central opening, and with a coat of silicon over the lids surface.

The efficiency of the anaerobic digestion process was evaluated by the removal contents of COD (colorimetry by closed reflux) and TVS (gravimetry) according to APHA [20] and daily monitoring of methane production until stabilization (at 20 days of testing).

For quantifying the methane volume produced in the assays, the liquid volume displacement method was used. The average methane produced by the "white" reactors (inocula + water), due to their endogeny, were discounted according to Eq. 1 [25]:

$$BMP = \frac{CH_4 \ accumulated \ volume \ (mL) \ - \} white}{g \ TVS \ added}$$
(1)

2.3. Biochemical Methane Potential tests with increase in the organic loading rate in the reactors

The inoculum source, temperature, and the mixture were tested, presenting higher efficiency in the treatment of the wastes

#### Table 1

Composition of the reactional means used in the BMP tests under mesophilic and thermophilic conditions.

COMPONENT	FOOD WASTE MIXTURES					
	M1 (100 % PPW)	M2 (75 % PPW + 25 % LW)	M3 (50 % PPW + 50 % LW)	M4 (25 % PPW + 75 % LW)	M5 (100 % LW)	
Mesophilic Test						
PPW (mL)	97.0	72.7	48.5	24.3	-	
LW (mL)	-	3.1	6.1	9.2	12.3	
Inoculum (mL)	47.0	47.0	47.0	47.0	47.0	
Water (mL)	106.0	127.2	148.4	169.5	190.7	
Thermophilic Test						
PPW (mL)	97.0	72.7	48.5	24.3	-	
LW (mL)	-	3.1	6.1	9.2	12.3	
Inoculum (mL)	50.0	50.0	50.0	50.0	50.0	
Water (mL)	103	124.2	145.4	166.5	186.7	

and in the methane production. This time, different ORL were applied (0.15; 0.30; 0.45; 0.60; and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>) to verify the possible interferences of this factor in the process. The assembling procedure of this test followed the same steps as the BMP tests aforementioned, according to Table 2. The performance of this test was evaluated under the same parameters adopted in the previous tests.

# 2.4. Data treatment

The result of the parameters analyzed was treated by statistical methods. The hydrolysis this is the limiting phase of the anaerobic digestion process and based on this, researchers have modeled batch BMP data using first-order hydrolysis models and obtained valuable interpretations about hydrolysis kinetics [26]. In this study, four modified models were used to adjust the data and to estimate the performance of the kinetic parameters (rate and maximum methane production, and time to start the methane production): Modified Gompertz, First Order, Logistic and Transference functions.

The Modified Gompertz (GM) model (Eq. 2) assumes that the rate of methane production in batch reactors corresponds to the specific growth of methanogenic bacteria. It is an empirical model of non-linear regression used to estimate the accumulated methane production [27].

The Logistic function (LG) assumes that methane production is proportional to the maximum rate of methane production. This model is used to estimate the parameters of anaerobic fermentation and methane production. In this case, a modified version of LG function was used (Eq. 3) [28].

The Transference function (Eq. 4) (reaction curve model) assumes that any process can be analyzed as an input and output system. This modified model has been implemented in the adjustment of anaerobic digestion data [29].

Finally, the First Order model (Eq. 5) was calculated by the relationship between the concentration of COD or TVS and methane production. This model is the simplest; however, it does not predict the maximum biological activity rate and system failures. using the first-order kinetic model, several studies have obtained valuable interpretations of anaerobic digestion kinetics [26].

$$R_{CH_4} = P \exp\left[-\exp\left(\frac{R_m \cdot e}{P}(\lambda - t) + 1\right)\right]$$
(2)

$$R_{CH_4} = \frac{P}{1 + \exp\left(\frac{4.Rm(\lambda - t)}{P + 2}\right)}$$
(3)

$$R_{CH_4} = P \left[ 1 - \exp\left(\frac{R_m (\lambda - t)}{P}\right) \right]$$
(4)

$$R_{CH_4} = P.(1 - e^{(-K.t)})$$
(5)

#### Table 2

Composition of the reactional medium used in the BMP test under different organic loading rates.

COMPONENTS	0	ORGANIC LOADING RATES (g TVS.L <sup>-1</sup> .d <sup>-1</sup> )			
	0.15	0.30	0.45	0.60	0.90
PPW (mL)	36.4	72.7	109.1	145.4	218.1
LW (mL)	1.6	3.1	4.7	6.2	9.3
Inoculum (mL)	47.0	47.0	47.0	47.0	47.0
Water (mL)	165.0	127.2	89.2	51.8	0.0

Where:  $R_{CH_4}$ : accumulated methane yield in time *t* (mL); *P*: maximum methane production (mL);  $R_{max}$ : maximum methane production rate (mLd<sup>-1</sup>);  $\lambda$ : time to start the methane production (d); *t*: time (d); *e*: exp (1) = 2.71828; and k: apparent hydrolysis rate coefficient (1.d<sup>-1</sup>).

The models statistical indicators were evaluated by the relative root mean square error (rRMSE) (Eq. 6) and coefficient of determination ( $R^2$ ) [30]:

$$rRMSE = \left(\frac{1}{m}\sum_{j=1}^{m} \left(\frac{d_j}{Y_j}\right)^2\right)^{1/2}$$
(6)

where,  $d_j$  is the deviation between the *jth* measured and the predicted values,  $Y_j$  is the *jth* measured value and *m* is the number of data points. Eq. (6) employed a relative root mean square normalized deviation [31].

R-squared ( $R^2$ ) (Eq. 7) is a statistical measure of how well a predicted line approximates measured data. An R-squared value equal to 1 implies that the model provides perfect prediction, and 0 implies that there is no relationship between the measured and predicted value [30].

$$R^{2} = \left[\frac{m\left(\sum_{j=1}^{m} X_{j} Y_{j}\right) - \left(\sum_{j=1}^{m} X_{j}\right)\left(\sum_{j=1}^{m} Y_{j}\right)}{\sqrt{\left[m\sum_{j=1}^{m} X_{j}^{2} - \left(\sum_{j=1}^{m} X_{j}\right)^{2}\right]\left[m\sum_{j=1}^{m} Y_{j}^{2} - \left(\sum_{j=1}^{m} Y\right)^{2}\right]}}\right]^{2}$$
(7)

where, Xj is the jth predicted value.

# 3. Results and discussion

## 3.1. Characterization of the inocula and substrates

The characterization of inocula (1) and (2) showed TVS contents of 81.65 % and 52.88 %, respectively (Table 3). The high TVS percentage in inoculum (1) showed a large amount of organic matter present in it with low mineralization rate, which can indicate the abundance of microorganisms. Conversely, inoculum (2) showed a smaller organic material percentage, with higher mineralization rate.

Granular sludges with organic content below 50 % present low capacity of anaerobic biodegradability [32]. Thus, inoculum (1) and (2) showed a high potential for being used in the tests with food wastes.

Inoculum (1) showed a Specific Methanogenic Activity (SMA) index of 0.1965 g  $\text{COD}_{\text{CH4},\text{g}}$  TVS<sup>-1</sup>.d<sup>-1</sup>, while inoculum (2) obtained 0.1559 g  $\text{COD}_{\text{CH4},\text{g}}$  TVS<sup>-1</sup>.d<sup>-1</sup>. For being considered active, the granular sludges must present SMA values about 0.1 to 0.3 g  $\text{COD}_{\text{CH4},\text{g}}$  TVS<sup>-1</sup>.d<sup>-1</sup> [33]. Thus, sludges (1) and (2) were considered active, with relevant conditions to be employed in bioreactors with organic wastes.

The two substrates showed pH of 4.53 and 4.73 for the wastes of the PPW and LW groups, respectively. Similar pH (4.66) was verified by Pavi et al. [34] during the production of biogas from food wastes. Yet, the pH values obtained in the present work fit the values for food waste estimated by Fisgativa et al. [35] (about 4.3–5.8).

The determination of the series of solids from the substrates revealed a concentration of 19.59 ( $\pm$  1.02) g TVS.L<sup>-1</sup> to PPW waste and 122.23 ( $\pm$  0.68) g TVS.L<sup>-1</sup> to LW waste. This analysis emphasizes a content of solids about 6.7 times higher in the wastes of the LW group than in the ones of the PPW group. The

Table 3					
Characterization	of	the	substrates	and	inocula.

PARAMETERS	SUBSTRATES		IN	OCULA
	PPW	LW	Mesophilic	(2)Thermophilic
рН	4.53 (0.06)	4.73 (0.03)	7.1 (0.02)	8.2 (0.05)
TS $(g.L^{-1})$	19.59 (1.02)	128.38 (0.70)	39.13 (1.49)	56.00 (1.25)
TFS $(g.L^{-1})$	4.13 (0.20)	6.14 (0.04)	7.19 (0.45)	26.37 (0.12)
TVS $(g.L^{-1})$	15.46 (0.86)	122.23 (0.68)	31.94 (1.03)	29.62(1.12)
TVS/TS (%)	78.89 (0.57)	95.21 (0.02)	81.65 (0.45)	52.88 (0.83)
TFS/ST (%)	21.10 (0.57)	4.78 (0.02)	18.35 (0.45)	47.12 (0.83)
SMA (g COD <sub>CH4</sub> .g TVS <sup><math>-1</math></sup> .d <sup><math>-1</math></sup> )	-	-	0.1965	0.1559
Moist (%)	84.15 (0.01)	74.6 (0.01)	_	_
Carbohydrates (g.L <sup>-1</sup> )	17.28 (1.02)	9.67 (0.84)	-	-
TA (g CaCO <sub>3</sub> .L <sup><math>-1</math></sup> )	0.6 (0.15)	0.9 (0.02)	-	-
VFA (g HAc. $L^{-1}$ )	1.98 (0.03)	2.21 (0.02)	_	_
$COD(g.L^{-1})$	31.9 (1.26)	53.2 (2.40)	_	_
BOD $(g.L^{-1})$	28.8 (1.62)	43.6 (1.43)	_	_
C/N ratio (%)	23.5 (0.45)	29.1 (0.88)	-	-

\*SMA: Specific Methanogenic Activity; g HAc. $L^{-1}$  – g. $L^{-1}$  of equivalent acetic acid.

smallest percentage of TVS in the PPW group can be attributed to the more fibrous material content in its structure.

In terms of TVS percentage, both groups presented high contents, with 78.89 % ( $\pm$  0.57) and 95.21 % ( $\pm$  0.02) for the PPW and LW groups, respectively. The values agreed with those in the literature [36–38], which estimated an organic content (TVS) of about 87.1 % for vegetal wastes, 95.2 % and 92.3 % for food wastes, respectively.

The moisture contents were 84.1 % ( $\pm$  0.01) for the wastes of the PPW group and 74.6 % ( $\pm$  0.01) for the LW group. These values were comparable with Fisgativa et al. [35] in estimating moisture contents between 74–90%.

The total carbohydrates of PPW (17.28  $(\pm 1.02)$  g.L<sup>-1</sup>) were higher than LW (9.67  $(\pm 0.84)$  g.L<sup>-1</sup>). Presumably, the PPW group contained more sugars than LW.

The total alkalinity of both wastes was 0.6 ( $\pm$  0.15) and 0.9 ( $\pm$  0.02) g CaCO<sub>3</sub>.L<sup>-1</sup>. The acid pH and the low total alkalinity indicate a low tamponing capacity of the system during the anaerobic digestion process.

The PPW and LW mixture of wastes had COD concentration of 31.9 g.L<sup>-1</sup> (± 1.26) and 53.2 g.L<sup>-1</sup> (± 2.40), respectively. The BOD of the mixture wastes were 28.8 g.L<sup>-1</sup> (± 1.26) for PPW and 43.6 g.L<sup>-1</sup> (± 1.43) for LW. An extremely high organic content hinders the action of microorganisms and makes the anaerobic process unstable.

The waste mixtures had a biodegradability (BOD/COD) level of 0.9 ( $\pm$  0.12) and 0.81 ( $\pm$  0.08), for PPW and LW, respectively. The high biodegradability of both wastes was confirmed with BOD higher than 0.5 [39].

The C/N ratios of PPW and LW were 23.5 ( $\pm$  0.45) and 29.1 ( $\pm$  0.88), respectively. Ratios with values of 20 to 30:1 are ideal to substrates biodigestion [40–42]. These results thus confirmed the good performance in the food wastes biodigestion.

# 3.2. Biochemical methane potential tests under mesophilic and thermophilic conditions

The removal percentage of COD from the mixtures of food wastes presented a decreasing pattern (M1 > M2 > M3 > M4 > M5) independently of the test conditions (Fig. 1), which were of 84.4 (M1), 82.0 (M2), 80.6 (M3), 78.7 (M4), and 74.1 % (M5) under mesophilic conditions; and 77.7 (M1), 77.6 (M2), 76.6 (M3), 74.8 (M4), and 71.5 % (M5) under thermophilic conditions. In all the mixtures, greater COD removal was observed under mesophilic condition, possibly due to the higher activity in the mesophilic sludge, as pointed out by the SMA index of the inocula (Table 3).



**Fig. 1.** Percentage of COD and TVS removal of anaerobic digestion from mixtures of food waste under mesophilic and thermophilic conditions. \*MT: Mesophilic Test; TT: Thermophilic Test.

The removal percentage of TVS (Fig. 1) was also superior in the mesophilic test (63.4 (M1), 73.3 (M2), 72.9 (M3), 76.8 (M4), and 68.1 % (M5)) than in the thermophilic one (62.3 (M1), 63.1 (M2), 64.7 (M3), 62.8 (M4), and 56.2 % (M5)). Mixture M1 probably presented a smaller TVS removal than mixtures M2, M3, and M4 in both test conditions due to the larger amount of fibrous material in this sample, which presents difficult degradation [43]. Mixture M5, which also presented smaller levels of TVS removal than M2, M3, and M4, presented this behavior due to the presence of oil in the leftover wastes, which may negatively affect the kinetics of the degradation reactions [44], interfering with the metabolic activity of the microorganisms. Thus, the facts previously described influenced the treatment of these waste mixtures by the anaerobic process.

The TVS removal under mesophilic (63.4–76.8%) and thermophilic conditions (56.2–64.7%) were relatively coherent with the rate of values observed by Bouallagui et al. [45] (58–75%), who evaluated the anaerobic digestion only for fruits and vegetables. However, in the present study, mixture M4 under mesophilic condition presented a TVS removal level of 76.8 %, slightly higher than [45].

There was a larger methane production and specific methane yield in the mesophilic condition test (1303 mL of  $CH_4$  and 869 mL of  $CH_4$ .g TVS<sup>-1</sup> (M1), 1256 mL of  $CH_4$  and 348–837 mL of  $CH_4$ .g TVS<sup>-1</sup> (M2), 1110 mL of  $CH_4$  and 740 mL of  $CH_4$ .g TVS<sup>-1</sup> (M3), 982 mL of  $CH_4$  and 654 mL of  $CH_4$ .g TVS<sup>-1</sup> (M4) and 523 mL of  $CH_4$  e 348 mL of  $CH_4$ .g

 $TVS^{-1}$  (M5)) than under thermophilic conditions (1103 mL of CH<sub>4</sub> and 735 mL of CH<sub>4</sub>.g  $TVS^{-1}$  (M1), 1004 mL of CH<sub>4</sub> and 670 mL of CH<sub>4</sub>.g  $TVS^{-1}$  (M2), 852 mL of CH<sub>4</sub>.g  $TVS^{-1}$  and 568 mL of CH<sub>4</sub>.g  $TVS^{-1}$  (M3), 750 mL of CH<sub>4</sub> and 500 mL of CH<sub>4</sub>.g  $TVS^{-1}$  (M4) and 506 mL of CH<sub>4</sub> and 338 mL of CH<sub>4</sub>.g  $TVS^{-1}$  (M5)) for all of the mixtures (Fig. 2). This could prove the metabolic activity of the microorganisms, according to the SMA level (Table 3).

A decreasing pattern in the generation and specific yield of methane was observed in the mixtures, in the order: M1 > M2 > M3 > M4 > M5, independently of the tested conditions, probably due to the total carbohydrates in the mixtures, which presented the same sequence highlighted in methane production.

Gou et al. [46] investigated the effect of different temperatures (35, 45, and 55 °C) in the digestion of food wastes and activated sludge. They observed a higher BMP value (400 mL of  $CH_{4.g}$  TVS<sup>-1</sup>) in the thermophilic condition. Superior BMP values were found in the present work for the mixtures from M1 to M4, both in mesophilic (654–869 mL of  $CH_{4.g}$  TVS<sup>-1</sup>) and thermophilic conditions (500–735 mL of  $CH_{4.g}$  TVS<sup>-1</sup>). However, mixture M5 presented inferior levels (338 and 348 mL of  $CH_{4.g}$  TVS<sup>-1</sup>) in both tested conditions.

The BMP values of all mixtures (348–869 mL of  $CH_{4.g} TVS^{-1}$ ) – mesophilic condition and 338–735 mL of  $CH_{4.g} TVS^{-1}$  were higher than the ones found by Santos [47], who studied the co-digestion of fruit and vegetable wastes + pruning and weeding wastes (257.4 mL of  $CH_{4.g} TVS^{-1}$ ). Guven et al. [48] obtained specific yield of 785 mL of  $CH_{4.g} TVS^{-1}$  in the monodigestion of OFMSW under mesophilic condition. These values were inferior in mixtures M1 and M2 (869 and 837 mL of  $CH_{4.g} TVS^{-1}$ ) in the mesophilic condition. This BMP difference between the studies was probably determined by the different wastes used.

# 3.3. Biochemical Methane Potential test with increase in the organic loading rates in the reactors

The mesophilic condition presented greater efficiency in treating wastes than the thermophilic condition, in terms of COD and TVS removal, methane yield and BMP value, corroborating the results of the SMA test for this condition. Thus, the organic carrying test was conducted under mesophilic condition.

For the BMP test with increase in OLR, mixture M2 was selected (75 % PPW + 25 % LW), which corresponds to the gravimetric composition of the wastes generated in the restaurant (75 % PPW + 25 % LW) and which presented results similar to the ones from mixture M1 (100 % PPW).



**Fig. 2.** Accumulated production of methane and BMP from food waste mixtures under mesophilic and thermophilic conditions. \*MT: Mesophilic Test; TT: Thermophilic Test.

ORL of 0.15 (82.1 %) and 0.30 gTVS.L<sup>-1</sup>.d<sup>-1</sup> (79.9 %) presented higher levels of COD removal than 0.45 (70.2 %), 0.60 (54.1 %), and 0.90 (39.9 %) (Fig. 3). The overloading of organic material in the reactors probably influenced the microorganism activity and the kinetics of the anaerobic degradation process.

Fernández et al. [49] verified the increase in the organic loading from 20 % to 30 % resulted in the decrease of more than 18 % in the efficiency of COD removal in anaerobic treatment of different loads of organic fraction municipal solid waste (OFMSW) (20 and 30 % TS). In the present study, also been observed a smaller efficiency of COD removal with the increase of the organic loading.

Comparing the performance of OLR of 0.15 and 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup> (which showed COD removal rates of approximately 80 %) with OLRs of 0.45, 0.60 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, the difference is approximately 12, 28, and 40 %, respectively. The fall in the COD removal may be due to the organic overloading in the reactors, which affected the metabolic activity in the microorganisms.

In terms of TVS, the OLR of 0.15 and 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup> presented removals of 79.5 and 80.1 %, respectively, i.e., high efficiency and very close contents, which highlights the adequate organic loads in the reactors. Conversely, for ORL equal to 0.45, 0.60 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, the TVS removals were of 54.4; 44.4; and 32.7 %, respectively, which shows that the increase in the organic loading in the reactors resulted in the fall in efficiency in the reduction of this parameter.

The removal verified at the OLR of 0.45 g TVS.L<sup>-1</sup>.d<sup>-1</sup> (54.4 %) was similar to the study by Pavi et al. [34] (54.4 and 54.6 %), which approached the anaerobic co-digestion of OFMSW + vegetal wastes under the proportion of 1/3, respectively. However, the OLR of 0.15 (79.5 %) and 0.30 (80.1 %) presented higher levels and the OLR of 0.60 (44.4 %) and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup> (32.7 %) presented inferior results. The overloading of organic matter in the reactors under OLR of 0.60 and 0.9 g TVS.L<sup>-1</sup>.d<sup>-1</sup> may have inhibited the methane generation.

High removals of TVS (83–91 %) had already been found in moist anaerobic digestion processes, with carrying level below 5% of total solids, with high TVS content and wastes with high biodegradability [50]. Thus, the results from the TVS removal at OLR equal to 0.15 and 0.30 g TVS.L<sup>-1</sup> were considered adequate.

Methane production was verified after 1 h of operation, as expected, since the hydrolysis and, consequently, the alcoholic fermentation of fruits and vegetables takes place in an accelerated way, due to the high biodegradability of these wastes [51]. Accumulated averages of 653, 1284, 1330, 1407, and 1741 mL of methane were observed for OLR equal to 0.15, 0.30, 0.45, 060 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, respectively (Fig. 4).



Fig. 3. Percentage of COD and TVS removal of different organic loading rates.



**Fig. 4.** Accumulated production of methane and BMP from food waste mixtures at different organic loading rates.

The methane produced in the reactors under OLR of 0.15 and 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup> presented linear relation; with the double of the organic loading, there was practically the double (1.97 times) of methane produced. However, for organic concentration higher than 0.15 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, there was a yield in the methane production of only 2.03; 2.15; and 2.66 times, respectively to OLR of 0.45, 0.60 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>. That is, with the increase of the organic concentration applied to the BMP test, the methane production did not have the same proportion. For OLR superior to 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, the methane generation was reduced by the overloading of the organic material in the reactors and a possible interference with the metabolic activity of the microorganisms.

OLR equal to 0.15, 0.30, 0.45, 0.60 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup> presented BMP values of 870, 868, 599, 475, and 391 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>, respectively (Fig. 4). However, OLR 0.15 and 0.30 g TVS.L<sup>-1</sup>. d<sup>-1</sup> presented very close levels, which emphasize the linearity between these conditions and indicate that, in concentrations up to 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, it was the optimal loading for the maximum performance of the anaerobic digestion of the food wastes approached in this study.

Haider et al. [52] evaluated the anaerobic co-digestion of food wastes and rice straw, with organic concentration of 2.0, 4.0, 8.0, 12.0, and 16.0 g VS of substrate and found a specific biogas yield of 557, 458, 267, 97, and 71 mL.g VS<sup>-1</sup>, respectively. The present study presented a similar behavior to that found by [47], being that the BMP value decreased with the increase of the organic loading in the reactors.

Liu et al. [53] evaluated the performance of the BMP test of food wastes by increasing the organic loading and reported that the anaerobic biodegradability of the wastes decreased with the increase of ORL. In the present work, a similar behavior was verified with the increase in the organic loading in the reactors.

ORL equal to 0.15 and 0.30 g TVS.L<sup>-1</sup> (870 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>) present BMP superior to the ones obtained by Yong et al. [54] (300–580 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>), Zhen et al. [55] (640 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>), Naran et al. [56] (481 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>), Koch et al. [57] (330–350 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>), Pavi et al. [34] (164–396 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>), and Guven et al. [48] (110–785 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>).

The BMP depends on the operational and environmental conditions of the tests, such as configuration of the continuous flux or batch reactors, moist or dry digestion, mesophilic or thermophilic condition, inoculum activity and the composition of organic wastes [58], which may hinder the comparison of the works.

# 3.4. Adjustment of data to models

The estimation of the kinetic parameters and data adjustment by applying the four modified models, Gompertz, First Order, Logistics and transfer functions, is summarized in Table 4, and Fig. 5 shows the model fit (solid line) with the experimental data from each assay (circles).

The Modified Gompertz model estimated the difference between the measured and predicted data of accumulated methane production of 1.42, 0.39, 0.94, 3.21 and 5.26 % for the organic loads of 0.15, 0.30, 0.45, 0.60 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, respectively. In the same order, the difference estimated by the First Order model was 0.60, 0.88, 0.40, 2.08 and 6.05 %. Meanwhile, the Logistic model estimated a difference of 2.10, 0.50, 0.23, 1.85 and 5.32 %, and the Transference function estimated a difference of 0.53, 1.76, 2.47, 4.72 and 1.42 %.

The estimation of the methane production values was variable, since for the organic loads of 0.15 and 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, the Transference function presented results closer to the data measured in the tests. For the organic loads of 0.30 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, the best

#### Table 4

Kinetic parameters calculated for organic loading tests by the models Modified Gompertz, First Order, Logistic and Transference functions.

		GM	FO	LG	TR		
0.15 g TVS.L <sup>-1</sup> .d <sup>-1</sup>							
	Measured (mL)	653.67	653.67	653.67	653.67		
$CH_4$	Predicted (mL)	644.38	649.75	639.95	650.17		
	Difference (%)	1.42	0.60	2.10	0.53		
Rm (mL	$d^{-1}$ )	354.69	-	409.19	549.28		
L (d)		-	-	0.02	-		
$K(d^{-1})$		-	0.8664	-	-		
R <sup>2</sup>		0.9813	0.9944	0.9698	0.9945		
rRMSE	1 - 1	0.1449	0.1049	0.1137	0.099		
0.30 g T	VS.L <sup>-1</sup> .d <sup>-1</sup>	1001.07	100107	100107	100107		
<u></u>	Measured (mL)	1284.67	1284.67	1284.67	1284.67		
$CH_4$	Predicted (mL)	1289.63	1295.94	12/8.23	1307.32		
D ( 1	Difference (%)	0.39	0.88	0.50	1.76		
Rm (mL	d ')	191.12	-	1/3.65	330.79		
L(a)		-	-	-	-		
$K(\mathbf{u})$ $\mathbf{p}^2$		-	0.2828	-	-		
K rDMCE		0.9801	0.9869	0.9729	0.9884		
	TVS I –1 d–1	0.5954	0.5454	0.5696	0.5562		
0.45 g I	Measured (mI)	1330.83	1330.83	1330.83	1330.83		
CH.	Predicted (mL)	13/3 3/	1336.00	1327.82	1363.60		
CI14	Difference (%)	0.94	0.40	0.23	2 47		
Rm (mI	$d^{-1}$	130.67	-	123.61	230.03		
L (d)		-	_	_	-		
$K(d^{-1})$		_	0.2047	_	_		
R <sup>2</sup>		0.9701	0.9450	0.9623	0.9795		
rRMSE		0.4857	0.4397	0.4797	0.4169		
0.60 g T	$VS.L^{-1}.d^{-1}$						
-	Measured (mL)	1407.33	1407.33	1407.33	1407.33		
CH <sub>4</sub>	Predicted (mL)	1452.52	1436.57	1433.34	1431.77		
	Difference (%)	3.21	2.08	1.85	4.72		
Rm (mL	$.d^{-1}$ )	107.98	-	106.33	172.97		
L (d)		-	-	-	-		
$K(d^{-1})$		-	0.1448	-	-		
R <sup>2</sup>		0.9627	0.9610	0.9592	0.9675		
rRMSE		0.5333	0.4904	0.5226	0.4581		
$0.90 \text{ g TVS.L}^{-1}.d^{-1}$							
	Measured (mL)	1741.67	1741.67	1741.67	1741.67		
$CH_4$	Predicted (mL)	1833.32	1636.28	1834.41	1766.35		
	Difference (%)	5.26	6.05	5.32	1.42		
Rm (mL	d <sup>-1</sup> )	74.96	-	74.49	81.54		
L (d)		-	-	-	-		
$K(d^{-1})$			0.1269				
R²		0.8472	0.8047	0.8469	0.8525		
rRMSE		0.8447	0.7573	0.7654	0.7012		

Rm: maximum methane production rate; L: time to start the methane production; k: apparent hydrolysis rate coefficient; R<sup>2</sup>: determination coefficient; rRMSE: relative root mean square error.



Fig. 5. Models fit of methane production of anaerobic test with different organic loading rates.

adjustment date was performed by Modified Gompertz model and, for ORL of 0.45 and  $0.60 \,\mathrm{g}\,\mathrm{TVS.L^{-1}.d^{-1}}$ , the greater proximity between theoretical and experimental data was observed by the application of the Logistic model.

In general, the percentage range of estimated values of methane production for the different OLRs was 0.39-5.26 (GM), 0.40-6.05 (FO), 0.23-5.32 (LG) and 0.53-4.72 (TR). Note that the largest difference values were observed in the organic load test of 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>, probably due to the higher organic load and process instability. The small difference between the theoretical values and the experimental values (less than 10 %) indicates that all the proposed models accurately predict the behavior of the reactors [59].

The Modified Gompertz, Logistic and Transference functions highlighted that the maximum methane production rate (Rm variable) followed a decreasing order of ORL: 0.15 > 0.30 > 0.45 > 0.60 > 0.90 g TVS.L<sup>-1</sup>.d<sup>-1</sup>. The largest range of Rm values were predicted by the Transference function (81.54–549.28 mL.d-1), followed by the Logistics (74.49–409.19 mL.d-1) and Modified Gompertz (74.96–354.69 mL.d-1) models. This analysis showed that the higher the organic load (SVT) applied to the reactors is, the lower the maximum methane production rate estimated by the models.

The time to start the methane production was negligible by all models, which indicates that the soluble material was quickly consumed by the anaerobic biomass [51,60].

The determination coefficient values (R2) were lower as the organic load was added to the tests. The models highlighted indices of 0.8472–0.9813 (GM), 0.8047–0.9944 (FO), 0.8469–0.9698 (LG) and 0.8525–0.9945 (TR). It is noticeable that, with the increase of the organic loading, the models employed for estimating the parameters presented the smaller correlation between the collected and the adjusted data.

The rRMSE indices followed values of 0.1449–0.8447 (GM), 0.1049–0.7573 (FO), 0.1137–0.7654 (LG) and 0.099–0.8525 (TR). For all the kinetic models, rRMSE indexes increased with increasing organic load.

The rRMSE indicates that the closer to 0, the greater the approximation between the observed data and the data calculated by adjusting the data. Thus, based on the kinetic study results, and statistical indicators ( $R^2$  e rRMSE), the model that showed the highest efficiency in data adjustment was the Transference function, followed by the First Order, Logistics and Modified Gompertz models. Note that all the models employed provided valuable kinetic data for the study of anaerobic digestion of food waste and that they should be used in other processes with the same purpose.

Kafle and Chen [29] compared three statistical models (Modified Gompertz, First Order and Chen and the Hashimoto) to predict the BMP of batch anaerobic digestion of five different livestock manures. The First Order model showed less difference between measured and predicted methane yield. The same behavior was observed in this work, comparing the same statistical models.

However, in the BMP tests conducted by Kafle et al. [61] and Kafle et al. [62], reported the Modified Gompertz model was a better model to predict the BMP compared to the First Order kinetic model. It is worth pointing out that each study has its peculiarities and that there is no consensus in the literature on which model to follow; it is necessary to test them until one that fits the work developed is found.

Donoso-Bravo et al. [60] also applied the same models as the present study to evaluate the effect of thermal pretreatment and the anaerobic degradation probe of sewage sludge. The authors also pointed out that all the models performed good data adjustment, but the Transfer function was the one that showed the least variation between experimental and theoretical data.

# 4. Conclusions

The mesophilic condition (temperature and inoculum) was the most adequate for treating the mixtures of food wastes for methane generation. In this condition, higher levels of COD and TVS removal, specific methane production were found.

Regardless of the experimental conditions, the percentage composition of food waste mixtures influenced the process. The highest average accumulated methane production followed the order of mixtures: M1 > M2 > M3 > M4 > M5.

Tests with increased organic load demonstrate that the maximum organic loading to be applied to the reactors to guarantee stability conditions and efficiency in the methane generation is up to  $0.30 \text{ g TVS.L}^{-1}$ .d<sup>-1</sup>, with high specific methane production up to 870 mL of CH<sub>4</sub>.g TVS<sup>-1</sup>.

The use of four simple models in the anaerobic digestion of food wastes showed to be a proper tool used to obtain performance parameters. The best data adjustment was performed by the kinetic model of the Transference function, followed by the First Order, Logistics and Modified Gompertz models. However, all the models highlighted that with the increase of the organic load in the tests, the proximity between the observed data and the predicted data was smaller.

In general, this study showed that it is possible to successfully treat food waste by anaerobic digestion process and to obtain high removal of the organic waste load and high methane yield, with potential for energy recovery.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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# Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.btre.2020.e00503.

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