

## **Biogas generation potential from anaerobic digestion of "Ponkan" and "Montenegrin" Tangerine peel waste**

Potencial de geração de biogás da digestão anaeróbia de resíduos de casca de Tangerina "Ponkan" e "Montenegrina"

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### **Abstract**

Due to the increase in human damage caused to the environment, there is a need for remediation measures and techniques to be applied in favor of a sustainable future for upcoming generations. One of the sectors that have potential in the use of low carbon economy stocks is agribusiness. In fact, part of agribusiness is fruit growing which is one of the sectors of greatest Brazilian economic prominence, with a wide variety of crops produced throughout the country and in different climates. In the food production chain, there are several post-harvest losses and the consequent generation of large amounts of waste. Anaerobic digestion presents itself as an excellent and promising option for the treatment of food waste, contributing to the fight against the increase of polluting emissions and generating biogas for use as thermal or electric energy or even as a fuel to replace fossil fuels. The anaerobic digestion of organic waste is a biological process characterized by the absence of oxygen which through the metabolic activities of microorganisms causes the complex organic material to be converted mainly into methane and carbon dioxide. In a scenario where the production of waste around the world is growing more and more, efficient waste treatment techniques are essential to ensure lower greenhouse gas emissions in the environment. In this study, the biodegradation capacity of the substrate formed with "ponkan" and "montenegrin" tangerine peels was investigated through BMP (Biochemical Methane Potential) tests with the determination of water content, total solids, and volatile solids in samples of laboratory under ideal conditions of pH, temperature, and humidity. The tests were carried out over a period of twenty-one days, having been found, in general, a greater production of gas until the twelfth day of testing, with a gradual reduction until stabilization around the twentieth day. The results obtained in laboratory show that there is a positive potential in the generation of biogas using tangerine peels in relation to the peels of other fruits researched in the literature (orange, banana, among others), with a biogas generation potential of 615.38 NmL/gSV for "ponkan" tangerine peel and 565.84 NmL/gSV for "montenegrin" tangerine peel. Brazil, due to its vast territorial extension and favorable climatic conditions, stands out in the international agricultural market and, therefore, holds great potential in the scenario of energy change. Despite the high potential of waste generated in the country, technologies for recovering organic matter are still incipient and even unknown. The results of this research are yet another contribution to reaffirm and publicize the benefits of treating organic matter via anaerobic digestion, with significant economic, social, and environmental advantages.

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**Keywords:** Anaerobic digestion; BMP test; Tangerine peel.

## Resumo

Com o crescente aumento dos danos antrópicos causados ao meio ambiente, surge a necessidade de medidas e técnicas de remediação a serem aplicadas em prol de um futuro sustentável para as gerações futuras. Um dos setores que apresentam potencial no emprego de ações da economia de baixo carbono é o agronegócio. Dentro deste está a fruticultura, que é um dos setores de maior destaque econômico brasileiro, com uma grande variedade de culturas, produzidas em todo o país e em diversos climas. Na cadeia produtiva do alimento ocorrem diversas perdas pós-colheita e a conseqüente geração de grandes quantidades de resíduos. A digestão anaeróbia, se apresenta como uma excelente e promissora opção para o tratamento de resíduos alimentares, contribuindo para o combate ao aumento de emissões poluidoras e gerando biogás para utilização como energia térmica, elétrica ou combustível em substituição aos combustíveis fósseis. A digestão anaeróbia de resíduos orgânicos é um processo biológico, caracterizado pela ausência de oxigênio, que através das atividades metabólicas dos micro-organismos faz com que o material orgânico complexo seja convertido principalmente em metano e dióxido de carbono. Em um cenário onde a produção de resíduos ao redor do mundo cresce cada vez mais, técnicas eficientes de tratamento de resíduos são fundamentais para garantir menores emissões de gases de efeito estufa no meio ambiente. Neste trabalho foi investigada a capacidade de biodegradação de substrato formado com cascas de tangerina tipos "ponkan" e "montenegrina" por meio da execução de ensaios BMP (Biochemical Methane Potential) com determinação dos teores de umidade, sólidos totais e sólidos voláteis em amostras de laboratório sob condições ideais de pH, temperatura e umidade. Os ensaios foram executados durante um período de vinte e um dias, tendo sido constatado, de modo geral, uma maior produção de gás até o décimo segundo dia de ensaio, com redução gradual até a estabilização em torno do vigésimo dia. Os resultados obtidos no laboratório mostram que há potencial positivo na geração de biogás utilizando cascas de tangerina em relação a cascas de outras frutas pesquisadas na literatura (laranja, banana, entre outras), com potencial de geração de biogás de 615,38 NmL/gSV para a casca de tangerina "ponkan" e 565,84 NmL/gSV para a casca de tangerina "montenegrina". O Brasil, devido a sua vasta extensão territorial e condições climáticas favoráveis, se destaca no mercado agrícola internacional e, dessa forma, detém grande potencial no cenário da mudança energética. Apesar do elevado potencial dos resíduos gerados no país, tecnologias de valorização de matéria orgânica ainda são incipientes e mesmo desconhecidas. Os resultados desta pesquisa constituem mais uma contribuição para reafirmar e divulgar os benefícios do tratamento de matéria orgânica via digestão anaeróbia, com significativas vantagens econômicas, sociais e ao meio ambiente.

**Palavras-chave:** Digestão Anaeróbia; Ensaio BMP; Casca de Tangerina.

## 1 Introduction

The constant increase in global warming that occurred after the middle of the twentieth century and intensified by anthropogenic actions based on the emission of greenhouse gases such as methane, is a reality that worries not only for the harmful damages to the environment and the quality of life of human beings, but also for the preservation of future generations (Silva & Paula, 2009). In the mid-1980s, with the Brundtland conference, the concept for “sustainable development” emerged which is defined as “meeting the needs of the present without compromising future generations” (BRUNDLAND, 1987).

In order to avoid excessive damage to the existence of human beings, recent studies by the Intergovernmental Panel on Climate Change (IPCC, 2018) examined the need to limit global warming to 1.5 °C instead of 2 °C, as previously predicted by the Paris Climate Agreement signed in 2015. According to the study, the absence of measures to be taken at present may generate extremely harmful damage to the environment, human beings, fauna, and flora of the planet.

Limiting global warming to 1.5 °C involves large reductions in the emission of methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>). Thus, a strong demand in the bioenergy sector is necessary, highlighting the importance of using appropriate management measures which contribute to minimizing the problem, as is the case with the use of anaerobic digestion as a treatment for organic solid waste.

The growing increase in the world population and the accelerated urbanization process of cities have resulted in the generation of excessive amounts of solid urban waste (Roth & Garcias, 2008), which, if poorly managed, contribute not only to the increase in damage caused to environment, but also inflict several problems in the social and economic aspect of modern society, thus aggravating the problem.

In Brazil, according to Paixão (2018), a large part of the solid waste generated is sent to sanitary landfills or dumps. Since these are inadequate destinations, it can lead to major socio-environmental impacts because in these types of provisions there are no adequate techniques to preserve the environment.

According to data from ABRELPE's "Panorama of Solid Waste in Brazil" (2015) in 2015, an amount of 72.5 million tons was generated with a coverage rate of 90.8% in the country. It represents 7,3 million tons of waste without collection and, consequently, with improper destination. According to Prates et al. (2019), even with the PNRS - National Solid Waste Policy - in force since 2010 (Brazil, 2010), a large part of the waste generated is still disposed of in landfills, without any previous processing.

In Brazil, the composition of USW (Urban Solid Waste) is quite heterogeneous and gravimetric analyzes reveal significant amounts of the fraction composed of organic materials, representing on average more than 50% of the collected waste (Zago & Barros, 2019). In addition, the agricultural sector also has a strong influence on the generation of OSW (Organic Solid Waste) given the fact that Brazil is a major power in the area. One of the branches of agriculture is fruit growing, a sector that generates significant amounts of biomass resulting from the harvest and processing of products in various sectors. According to Christo et al. (2018), more than 40% of food losses occur in the post-harvest and processing stage in developing countries.

Brazil is the world's largest producer of citrus fruits with tangerines being the second largest group of citrus fruits produced, behind only orange (Brackmann et al., 2007). According to IBGE data (IBGE, 2018), approximately 997 thousand tons of tangerines are harvested annually in the country.

In the fruit-growing sector, it is natural for losses to occur along the productive chain. According to Cypriano et al. (2017), approximately 50% of the fruit is discarded in the form of fruit pomace (composed of peel, seed, and pulp), which can be used to obtain products with higher added value. In view of the large population increase, a consequence for the coming years is an increase in per capita consumption of electricity in Brazil, making it necessary to search for electricity production in an alternative way to fossil fuels (Christo et al., 2018), in addition to more effective waste management solutions than landfill disposal.

Alternatives to landfill of waste tend to generate greater long-term benefits, such as ensuring the recovery of recyclable materials and the use of USW, thus reducing the environmental liability due to poor waste management. Thus, treatment technologies then

come as a remedy for these damages since they can be applied to maximize the waste potential.

Among the various types of solid waste treatment, anaerobic digestion represents an alternative as a biological treatment of organic waste. According to Amaral et al. (2004), its main benefit is the generation of biogas which can be used as a source of alternative renewable energy. Anaerobic digestion allows the reduction of the polluting potential and health risks of manure to a minimum, in addition to allowing the recycling of effluent which can be used as a biofertilizer.

Consistent with Gueri et al. (2018), food residues have a good potential to produce biogas since they present values above the average of most substrates when evaluating specific methane production. The estimation of this generation can be done through experimental studies carried out in bench reactors such as the biochemical methane potential (BMP) test. According to the authors, this test allows the substrate to biodegrade while, at the same time, measures the specific production of methane per unit of organic load (that is, by the amount of volatile solids).

Although not yet internationally standardized, the test is an analytical reference method when it comes to obtaining more information on the transformation of organic materials into methane. The process takes place under optimal conditions of degradation and can be considered an accelerated anaerobic digestion process.

This study aimed to evaluate the biogas generation potential from peel waste of two types of tangerine, the "ponkan" and the "montenegrin" species, through the BMP test. Thus, this research seeks to add more data to the literature in order to better understand the behavior of organic waste during anaerobic digestion.

## **2 Material and Methods**

### **2.1 Substrate preparation**

The substrate collection of the "ponkan" tangerine peel occurred through the purchase of the tangerines, with the peels separated and stored in the refrigerator. In order

to obtain the "montenegrin" tangerine residues, a supermarket in the south zone of Rio de Janeiro was contacted, which supplied the peels of the tangerines used for the production of natural juices.

The tangerine peels were subjected to a crushing process to reduce the size of the material and obtain a homogenized mass (Figure 1). Afterwards, such residues were also properly stored under refrigeration at  $-4\text{ }^{\circ}\text{C}$ .

Figure 1 - Crushing the substrate. A) Food multiprocessor; B) Sliced peel; C) Crushed peel.



## 2.2 Substrate characterization

The following physicochemical properties were determined: water content (w), total solids (TS), and volatile solids (VS) of the tangerine peels. Such information is essential for the evaluation of the biogas generation potential of the substrate.

## 2.3 Water content determination

To determine the water content of the "ponkan" and "montenegrin" tangerine peels, the procedure adopted by the Brazilian standard NBR 6457 (ABNT, 1986) was carried out. The samples, already crushed and homogenized, were separated and placed in porcelain crucibles. After mass constancy at a temperature of  $110\text{ }^{\circ}\text{C}$  in an oven, the water content based on dry weight was calculated according to Equation (1):

$$w = \frac{M_i - M_f}{M_f} * 100 \quad (1)$$

Where  $w$  is the water content (%);  $M_i$  is the mass of the residue in its initial state, that is, wet (g); and  $M_f$  is the mass of the residue after drying, that is, dry (g).

## 2.4 Total solids content determination

The procedure adopted by the Brazilian standard NBR 10664 (ABNT, 1989) was carried out. This procedure is similar to the previous one. However, for the calculation Equation (2) was used:

$$TS = \frac{M_f}{M_i} * 100 \quad (2)$$

Where  $TS$  is the total solids content (%).

## 2.5 Volatile solids content determination

This parameter determination was based on the WHO methodology (Standard Methods 2540G, 1992). The remaining mass used to determine the  $w$  and  $TS$  contents was introduced into a muffle furnace under the temperature of 550 °C for 5 hours. The volatile solids content was calculated based on Equation (3):

$$VS = \frac{P_1 - P_f}{P_1} * 100 \quad (3)$$

Where  $VS$  is the content of volatile solids (%);  $P_1$  is the initial mass of the dried sample in a kiln at 110 °C (g) and  $P_f$  is the final mass of the sample burned in a muffle furnace at 550 °C (g).

## 2.6 Biochemical Methane Potential (BMP) test

The assay method employed in this study was performed on the equipment made by Abreu (2014). It is recommended to consult the work of Abreu (2014) for the details of the equipment assembly.

However, for the test setup, the VDI 4630 standard was followed. As a recommendation of this standard, before the test setup, it is necessary to determine the amount of substrate and anaerobic sludge to be used. Among the restrictions imposed by the standard is preventing fermentation inhibition, thus, the substrate must not exceed the proportion presented by Equation (4):

$$\frac{TS \text{ substrate}}{TS \text{ sludge}} \leq 0,5; \quad (4)$$

The gas production yield of the substrate must be at least 80% of the total gas generated; and the amount of total solids must not exceed 10% to guarantee an adequate mass transfer.

Based on the restrictions and the physicochemical characteristics of the residue, the substrate mass and the amount of anaerobic sludge for digestion were calculated. Thus, for the "ponkan" tangerine, 10 g of substrate was added to 390 mL of sludge, while for the "montenegrin" tangerine, the values were 12 g and 388 mL, respectively. In the bottles containing only anaerobic sludge, the added value was 400 mL for both tests. Figure 2 shows the sample preparation process in the flasks. It should be noted that the tests were performed in triplicates.

Figure 2 - Preparation of the substrate-sludge mixture for the BMP test





With the substrates properly allocated in their flasks, the test proceeded with the pH measurement with the *Hanna* edge benchtop pH meter. It is important to note that during the entire test, it was only possible to measure the pH before and after the end of the test, that is, twenty-one days after the start, because after the coupling of the eudiometric tube to the reagent flask, the set does not allow continuous pH measurements during the process.

Finally, before being taken to the eudiometer, oxygen was extracted from the inside of the flasks through the addition of nitrogen gas, which led to the creation of an anaerobic environment for the degradation of the substrate.

Afterwards, the flasks were finally coupled to the eudiometer, starting the test with daily measurements of gas generation and future analyzes on the biogas generator potential of the tangerine peel residues. Figure 3 shows the complete system of the equipment used for the BMP test at the Laboratory of Geotechnics and Environment (LGMA) of PUC-Rio, to analyze the potential of biogas generation through anaerobic digestion of the tangerine peel.

Figure 3 - LGMA equipment used for the BMP test.



### 3 Results and Discussion

#### 3.1 Substrates characterization

Table 1 shows the results regarding the characteristics of the tangerine peels. For the evaluated "ponkan" tangerine peels, the average water content reached 304.9% and contents of 24.7% of total solids and 98.5% of volatile solids were determined. Similarly, for the "montenegrin" tangerine peels, the same parameters were determined noting slightly higher values of water content (316.4%) but with slightly lower values for TS and VS contents (24 and 96.6%, respectively) in relation to those found for the "ponkan" tangerine.

Table 1 - Tangerine peel test results.

Substrate	Input parameters		
	w (%)	TS (%)	VS (%)
"Ponkan" tangerine	304.9	24.7	98.5
"Montenegrin" tangerine	316.4	24	96.6

#### 3.2 BMP test

Table 2 presents the summary of the input and output data (after the 21-day test period) used for the BMP tests. Regarding the "ponkan" peel, the results of accumulated gas generation in the period of twenty-one days are shown in Figure 4. The generation curves for flasks 1, 2, and 3 show the behavior of microbial activity in anaerobic digestion.

In the first days the activity is intense, with a great slope of the curve that, over time, becomes softer from the tenth day, indicating the reduction of microbial activity in the generation of gas, which finally stabilizes around the twentieth day with little gas generation. Flasks 4, 5 and 6, in other words, flasks with only anaerobic sludge, were used as blank solutions, that is, to show how much biogas is generated only by the sludge and not by the substrate.

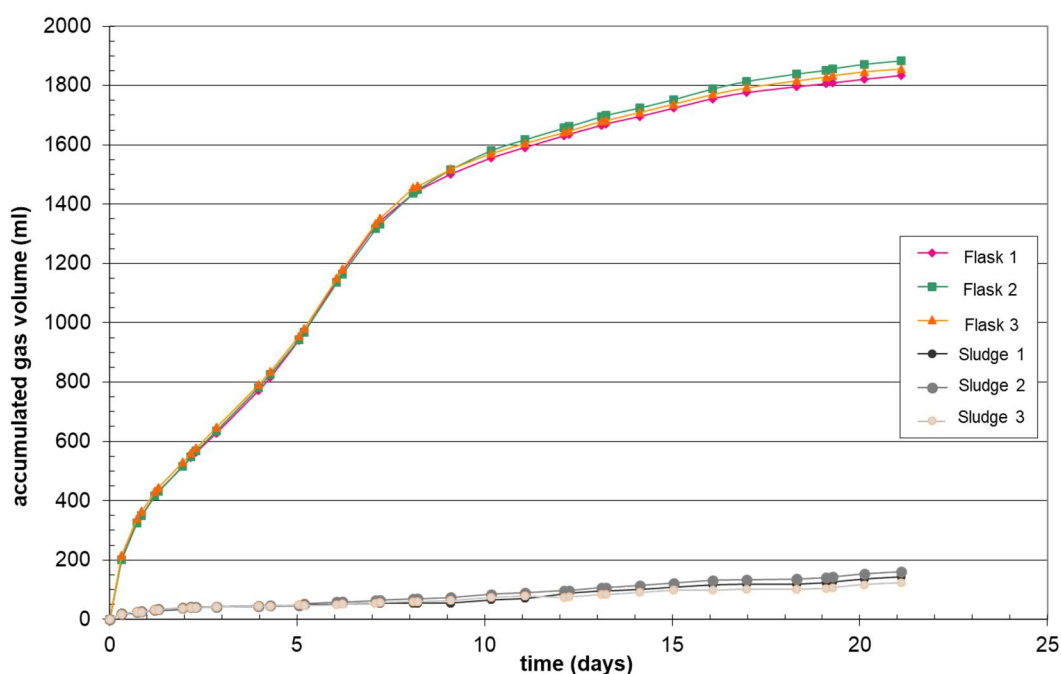
It is worth emphasizing the importance of measuring pH before and after the test, as the test configuration does not allow constant measurements. It is noted

that the pH values practically did not decrease after the test, remaining around the range between pH 7.3 and pH 7.8.

Table 2 - Summary of input and output data for the tangerine BMP test

Substrate	Input data	Output data	F1	F2	F3	F4	F5	F6
"Ponkan" tangerine	Substrate mass (g)	-	10	9.99	10.01	-	-	-
	Sludge mass (mL)	-	390	390	390	400	400	400
	Initial sludge pH	-	-	-	-	7.74	7.74	7.74
	Initial solution pH	-	7.59	7.59	7.59	-	-	-
	-	final solution pH	7.3	7.28	7.3	7.46	7.45	7.47
"Montenegrin" tangerine	Substrate mass (g)	-	12	12	12	-	-	-
	Sludge mass (mL)	-	388	388	388	400	400	400
	Initial sludge pH	-	-	-	-	7.92	7.92	7.92
	Initial solution pH	-	7.77	7.68	7.72	-	-	-
	-	final solution pH	7.32	7.34	7.35	7.46	7.49	7.48

Figure 4 - Accumulated biogas volume for "ponkan" tangerine peel



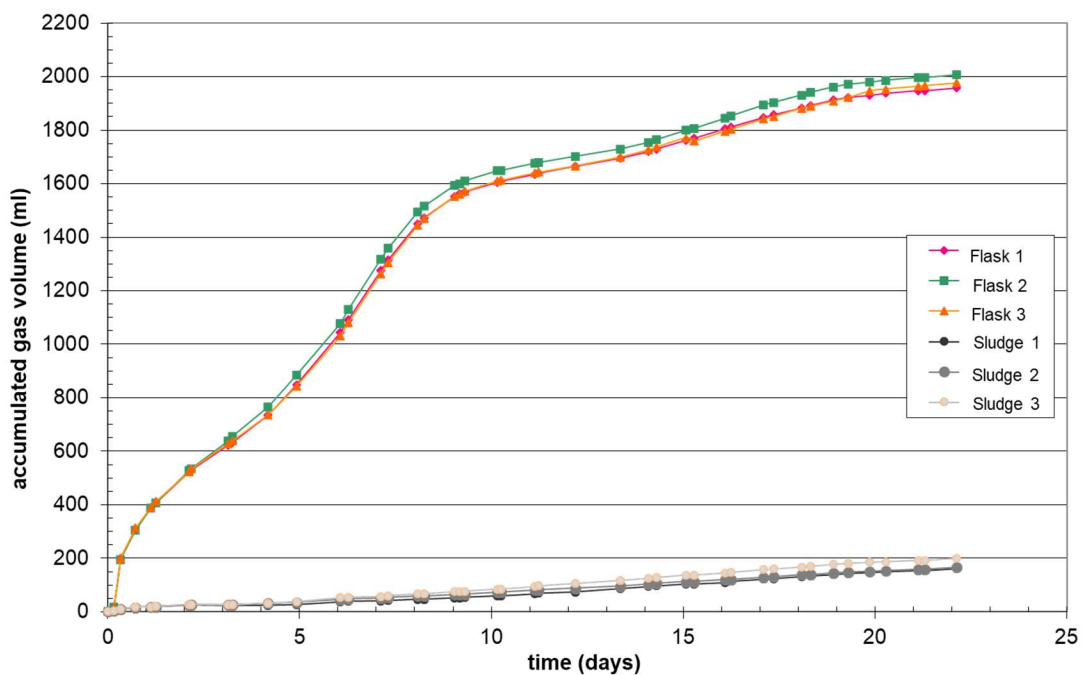
Thus, an average of 1,858 mL (or 1,621.4 NmL by conversion to normal temperature and pressure NTP) was obtained for accumulated gas volume for flasks 1, 2 and 3, that is, those with the mixture peel + sludge, and an average of 142.3 mL (or 124.2 NmL by

conversion to NTP) for flasks 4, 5 and 6, which contained only sludge. Correcting the volume in relation to the volume generated by the sludge, the volume of biogas generated was 1,497.2 NmL.

In terms of biogas generation potential, the value for the "ponkan" tangerine peel is approximately 615.38 NmL/gVS, or, in terms of total solids, 606.15 NmL/gTS.

The graphical result of the accumulated biogas volume for the "montenegrin" tangerine peel is shown in Figure 5. The analysis of the graph of accumulated biogas production allows to verify that around the fifth day there is still a marked generation of gas, with the slope of the curve decreasing from the twelfth day of testing, and stabilizing around the eighteenth day of testing.

Figure 5 - Accumulated biogas volume for the "Montenegrin" tangerine peel



An average of 1,979.5 mL (or 1,727.5 NmL by conversion to NTP) of accumulated gas volume was obtained in flasks 1, 2, and 3, which contained a mixture of peel + sludge, and an average of 175.7 mL (or 153.3 NmL by conversion to NTP) for flasks 4, 5, and 6, with only sludge. Correcting the volume in relation to the volume generated by the sludge, the volume of biogas generated becomes 1,574.2 NmL, on average, for the "montenegrin" tangerine peel.

The biogas generation potential of "montenegrin" tangerine was approximately 565.84 NmL/gVS, slightly less than for "ponkan" tangerine with a difference of 49.5 NmL/gVS. In terms of total solids, the potential of montenegrin tangerine was 546.6 NmL/gTS, slightly less than for the "ponkan" tangerine, with a difference of 59.5 NmL/gTS.

From the analysis of the results obtained, even with a greater generation of gas within the twenty-one days by the "montenegrin" tangerine peel, it has a lower potential for biogas generation compared to that of the peel of the "ponkan" tangerine. Some factors capable of justifying this issue are related to the fact that "ponkan" tangerine has better physical properties than "montenegrin" tangerine. For instance, for the same TS content, it presents a slightly higher VS content (2%) than the "montenegrin" tangerine.

Table 3 compares the biogas generation potentials of some fruits found in the literature with the tangerines studied in this research. Although each author expressed the biogas potential through a certain unit, to facilitate the comparison of the results of the different fruits, in this study the units were converted to NmL/gVS, with the exception of those results expressed in terms of TS, in which case the original unit was maintained since in these studies details about the VS values were not presented to make the conversion.

Table 3 - Comparative table of biogas potential of different fruits

Substrate		Biogas Potential	Unity	Author
Fruit	Fruit Parts			
Tangerine	"Ponkan" peel	615.38	NmL/gVS	This research
		606.15	NmL/gTS	
	"Montenegrin" peel	565.84	NmL/gVS	
		546.6	NmL/gTS	
	Peel (unspecified)	424.12	NmL/gVS*	GUNASEELAN, 2004**
Orange	Pomace	288	NmL/gVS	SANTOS <i>et al.</i> , 2018
	Pre-treated peel	478	NmL/gVS	CARVALHO <i>et al.</i> , 2017
	Peel	397.07	NmL/gVS*	GUNASEELAN, 2004**
Banana	Peel	212.93	NmL/gTS*	SOUZA <i>et al.</i> , 2013
	Peel	571.44	NmL/gVS*	ACHINAS <i>et al.</i> , 2019
	Peel + Stalk + Leaves + Pseudostem	269.05	NmL/gTS*	SOUZA <i>et al.</i> , 2010

Apple	Polpa	610.88	NmL/gTS*	COALLA <i>et al.</i> , 2009
Grape	Pomace	174.54	NmL/gVS*	BESINELLA <i>et al.</i> , 2017
Mango	Peel	322.89 – 456.41	NmL/gVS*	GUNASEELAN, 2004**
Pineapple	Peel	311.55	NmL/gVS*	
Melon	Peel	210.4	NmL/gVS*	
Lychee	Seeds	62.31	NmL/gVS*	
	Peel	93.73	NmL/gVS*	
Cherry	Seeds	27.14	NmL/gVS*	ZHAO <i>et al.</i> , 2016
Maracuya	Peel	170	NmL/gVS*	
Avocado	Peel	78.54	NmL/gVS*	
Dragon fruit	Peel	170.17	NmL/gVS*	

\* Values adapted to the units of this research; \*\**apud* LUCENA, 2016.

It is evident from this comparison that the characteristics of each fruit influence the process of anaerobic digestion in different ways. It should be noted that the peels are the parts of the fruit that tend to generate the greatest amount of gas, while the seeds have practically no generation.

Gunaseelan (2004, *apud* LUCENA, 2016) carried out a BMP test with several fruits, including citrus fruits like tangerine. Although the type of tangerine was not specified, the author reached results of 0.486 m<sup>3</sup>/kgVS for the analyzed fruits. Converting to the units used in this research, the value reaches 424.12 NmL/gVS, below the 615.38 NmL/gVS obtained with the "ponkan" tangerine peels and 565.84 NmL/gVS of the "montenegrin" tangerine determined in this study. This fact indicates the possibility that another type of tangerine was used. The organic load content of the tests used by Gunaseelan (2004) must also be observed.

Santos *et al.* (2018) carried out tests with orange pomace and inoculum, reaching results considered efficient in terms of production, potential, and methane generation. According to those authors, the configuration of the orange pomace mixed with the inoculum showed an accumulated volume of biogas of 1,748.0 NmL. Regarding the inoculum without the addition of orange pomace, an accumulated volume of biogas of 308.0 NmL was generated. Note that, when comparing with the tangerines studied here, the gas generation values were close (1,497.2 NmL and 1,574.2 NmL). In terms of biogas generation

potential, Santos et al. (2018) reached the result of 288 NmL/gVS, that is, 327.38 NmL / gSV below the "ponkan" peel and 277.84 NmL/gVS below the "montenegrin" peel.

Finally, Carvalho et al. (2017) in studies with pre-treated orange peel with the addition of sewage sludge obtained a biogas potential of 478 NmL/gVS, that is, 137.38 NmL/gVS below the "ponkan" tangerine and 87.84 NmL/gVS below the "montenegrin" tangerine. Such information reinforces the importance of tangerine as a fruit with a potential similar to orange as a substrate for anaerobic digestion and consequent biogas generation.

## **4 Conclusions**

This research carried out an analysis of the biochemical potential of biogas from an anaerobic digestion process of two types of tangerine. The results of laboratory experiments obtained with peels of "ponkan" and "montenegrin" tangerines showed a potential for biogas generation of 615.38 NmL/gVS and 565.84 NmL/gVS, respectively. The difference found can be considered as reflecting the physical-chemical characteristics, which showed little difference and which, in turn, influence their ability to generate gas during anaerobic digestion.

Furthermore, the biogas potential values obtained indicate that such substrates have a high potential when compared to other fruit peels. Therefore, the anaerobic digestion of the tangerine peel is a great solution in the management of solid waste, since its application results in good efficiency in the degradation of organic matter and fine energy use with the biogas generation, being then not just a way to manage waste as well as an option to generate clean energy.

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