Characterization of macadamia oil (macadamia integrifolia) obtained under different extraction conditions

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ABSTRACT

The aim of this study was to evaluate how the pretreatment of macadamia nuts, with oven or microwave, followed by oil extraction by mechanical press (screw or hydraulic) affect the yield and chemical characteristics of macadamia oil. The combination of the pretreatments and mechanical oil extractions was done as follows: oil extract only by screw press, only by hydraulic press, heating by microwave followed by screw press, heating in oven followed by screw press, heating by microwave followed by hydraulic press, and heating in oven followed by hydraulic press. To characterize the oils, the following analyzes were performed: yield, peroxide value, free acidity, iodine and saponification value, fatty acid composition and oxidative rancidity. The oils extracted by hydraulic press combined with microwave had the highest yield, the best oxidative stability and lowest peroxide value. The results suggest that the combination of microwaves and hydraulic press extract oils with quality superior to the other treatments studied.

Keywords: Screw press; Hydraulic press; Nut; Heat; Microwave

INTRODUCTION

The extraction of oil from nuts and seeds is one of the most critical steps in processing, as it determines the quality and quantity of oil extracted. The oil extraction process can be carried out in different ways, one of them being the solvent extraction. Solvent extraction is a process that transports mass from one phase to another. The liquid solvent comes into contact with the lipid matter, interacting with it and subsequently dragging the lipids out of the solid matrix (Araújo et al., 2019).

The use of organic solvents to extract oil from seed and nuts has been much questioned. Organic solvents in the food industry have some disadvantages, such as health and safety problems. The use of flammable and toxic solvents causes adverse environmental effects, besides deterioration of bioactive compounds because of the high-temperature processing (Cheng et al., 2011) and the potential of solvent residue in the final product, causing health issues.

Several extraction techniques that replace organic solvents have been researched to achieve more ecological methods of extraction. Safe extraction methods have been used for extracting oils from seeds, such as supercritical fluid extraction and hydraulic and screw presses (Hamed et al., 2017). Although supercritical fluid extraction does not use solvent and is capable of extracting higher quality oil, it is still very expensive to use on a large scale. Thus, extraction by pressing is the best option to extract good quality oils at an affordable price (Roncero et al., 2016).

Mechanical methods are very suitable to replace the extraction of oil by organic solvents. They have the advantage of low cost, producing a higher quality oil, with low concentrations of free acidity, and the possibility of the easy use of its cake compared to when extraction is done in solvents (Nde and Foncha, 2020).

The screw and hydraulic pressing are considered adequate methods of mechanical extraction for small and medium scale production levels since it is...
environmental friendly and safe to extract functional oils for the dietary supplement market (Hamed et al., 2017).

The disadvantage of mechanical extraction methods is that they provide a lower yield when compared to solvent methods of oil extraction (Santoso et al., 2014). Optimizing the conditions of mechanical extraction methods is important to increase oil yield and quality, in addition to saving time, energy and lowering the costs of the entire process (Satyannarayana et al., 2018; Nde and Foncha, 2020).

The use of pretreatments in nuts and seeds is an alternative to make mechanical oil extraction more effective. Cooking is a type of pretreatment that, although it allows an increase in yield, also could be responsible for the degradation of the oil (Rombaut et al., 2015), which is uninteresting in the final quality of the product.

Alternatives such as microwaves have become increasingly popular for its technical and economic benefits of replacing the use of hydrocarbon solvents (Cirimmina et al., 2016) and heating process. The microwave applied in the nuts as a pretreatment to cold pressing is able to significantly enhance oil yield (Güneşer and Yılmaz, 2017). The microwaves generate high frequency waves, which break the cells by shock induction. The waves penetrate into the material and vibrate the molecules, provoking a rapid heating and subsequent rupture of the cells (Aguilar-Reynosa et al., 2017), which may help to improve oil extraction efficiency.

To the best of our knowledge, there is no study comparing different pretreatments of macadamia nuts combined with cold extraction of its oil. The objectives of this study were, therefore, to evaluate how the pretreatment of macadamia nuts with oven or microwave followed by oil extraction by mechanical presses (screw or hydraulic press) affect the yield and chemical characteristics of macadamia oil.

**MATERIALS AND METHODS**

**Materials**
The commercial samples of macadamia nuts were obtained from the market of Lavras - MG, Brazil. A screw press (Yoda, MQ0001) and a hydraulic press (Marcon, MPH-15) equipped with an oil extraction cylinder (6 cm diameter × 7 cm height) was used to extract the macadamia oil. A microwave oven (Philco, PME22, microwave frequency 2450 MHz), laboratory oven (Medicate, MD 1.1), petri dishes (100 mm of diameter and 15 mm high). Solvents (99%) and other chemicals (analytical grade) were purchased from Sigma-Aldrich (São Paulo, Brazil).

**Pretreatments and extraction of macadamia oil**

**Pretreatments:** The nuts were separated into three parts: 200 grams of which did not undergo any pretreatment, 200 grams only under pretreatment with oven and 200 grams only microwave pretreatment.

Firstly, 200g of the nuts were placed in Petri dishes (100 mm of diameter and 15 mm high) and subjected to the pretreatments by microwave oven at maximum potency with exposure time of 1 min. The second pretreatment consisted of heating 200g of macadamia in the laboratory oven at 100ºC for 5 minutes. After receiving the pretreatments, the nuts were submitted to the oil extraction.

**Extraction of macadamia oil:** Two types of oil extraction of were carried out: with screw press and hydraulic press. The nuts were divided into 6 groups of 100 grams each. Each group went through a type of mechanical extraction, resulting in 6 treatments: oil extracted by screw press without any pretreatment (SP), oil extracted by hydraulic press without any pretreatment (HP), microwave pretreatment followed by screw press extraction (MSP), oven pretreatment followed by screw press extraction (OSP), microwave pretreatment followed by hydraulic press extraction (MHP) and oven pretreatment followed by hydraulic press extraction (OHP), as shown in Table 1. After the oil extraction, all the samples were centrifuged at room temperature for 5 min at 4000 rpm (relative centrifugal force of 2150g) to separate the fine particles of cake and stored at 4°C until further analysis.

**Characterization of macadamia oils**

After the macadamia oil extraction the samples were characterized chemically. All analyzes were performed in triplicate according to the following parameters:

**Yield of macadamia oil**
The yield analysis of the macadamia oil was calculated by the following formula:

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\text{Yield of macadamia oil} = \frac{\text{Mass of oil}}{\text{Mass of dry matter}} \times 100
\]

**Table 1: Processes used in the extraction of macadamia oil**

| Treatments | Extraction type of macadamia oil |
|------------|---------------------------------|
| SP         | Oil extracted by screw press without any pretreatment |
| HP         | Oil extracted by hydraulic press without any pretreatment |
| MSP        | Pretreatment with microwave followed by screw press extraction |
| OSP        | Pretreatment with oven followed by screw press extraction |
| MHP        | Pretreatment with microwave followed by hydraulic press extraction |
| OHP        | Pretreatment with oven followed by hydraulic press extraction |

SP = oil extract by screw press without any pretreatment, HP = oil extracted by hydraulic press without any pretreatment, MSP = pretreatment with microwave followed by screw press extraction, OSP = pretreatment with oven followed by screw press extraction, MHP = pretreatment with microwave followed by hydraulic press extraction and OHP = pretreatment with oven followed by hydraulic press extraction.
Yield (%) = (Weight of oil x100)/Weight of macadamia sample.

**Peroxide value**
Expressed in milliequivalents active oxygen contained in 1 kg of oil, according to AOCS method Cd 8b-90.

**Free acidity**
Expressed as the percentage of free oleic acid, according to the AOCS method Ca5a-40 (2004).

**Fatty acid composition**
Samples were transesterified into methyl esters using potassium hydroxide in methanol and n-hexane, according to AOCS method Ce 2-66. The methyl esters were analyzed by gas chromatography (GC-2010 - Shimadzu) equipped with a flame ionization detector and an SPTM-2560 capillary column (100 mm × 0.25 mm × 0.2 μm). The standard used was a mixture of 37 methyl esters (Supelco 37 Component FAME Mix). The following operational parameters were used: split injection mode, split ratio 1:100; 1 μL injection volume; 260°C detector temperature; 260°C injector temperature; oven temperature program: held at 60°C for 1 minute, ramped at 4°C·min−1 to 140°C, held for 5 minutes; ramped at 4°C·min−1 to 240°C, held for 30 minutes. Peak identification was resolved by comparing the retention times of the fatty acid methyl ester standards with the retention times of the observed peaks. Quantification was done by normalization (%).

**Saponification value**
Saponification value defined by the amount in milligrams of potassium hydroxide needed to saponify 1 g of oil or fat, calculated using the fatty acid composition by AOCS method Cd 3a-94.

**Iodine value**
Expressed as the amount of iodine absorbed by 100 g of sample, calculated using the fatty acid composition according to AOCS method Cd 1c-85.

**Oxidative Rancidity**
Expressed as the induction period in hours determined using 3 g of sample in Rancimat apparatus at 110°C with an airflow rate of 20 L·h−1 and 60 mL of distilled water in vials containing electrodes, according to AOCS method Cd 12b-92.

**Statistical analysis**
Statistical analyses were performed using SISVAR (Ferreira, 2011). Data are expressed as mean ± SD from triplicates. One-way ANOVA was used and differences between means were calculated using Tukey’s test and statistical significance was set to p < 0.01.

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**RESULTS AND DISCUSSION**

**Yield of macadamia oil**
The yield of the oils extracted under different conditions ranged from 27.87 to 48.05%. The MHP sample obtained the highest yield (48.05%), followed by OHP (45.25%), HP (45%), OSP (38%), SP (28.64%), and lastly MSP (27.87%) (Fig 1).

The findings showed that the yield of macadamia oil was influenced both by the type of extractor and by the pretreatment applied to the nuts before the oil is extracted. This is in accordance with Navarro and Rodrigues (2018), who explained that the extraction and yield of macadamia oil are affected by the temperature and pretreatment given to the material. Navarro and Rodrigues (2016) showed that this nut oil may vary from 33-65%, depending on the variety and extraction method.

The extraction of macadamia oil only by hydraulic press (HP) and hydraulic press combined with different pre-treatments (MHP and OHP) was the extraction method that presented the highest yield. Despite the industrial advantages of continuous extraction, oil samples extracted by screw press (SP, MSP and OSP) obtained a much lower yield than via hydraulic press.

Yusuf (2018) states that by hydraulic pressing, oil yield is enhanced by increased mechanical pressure on the material. Furthermore, the high pressure through a hydraulic press, combined with pretreatments (oven or microwaves), obtained a higher yield than only hydraulic pressing on the macadamia nuts.

Microwaves used as pretreatment for oil extraction demonstrated to be a quick and inexpensive technique.
It was quite effective when followed by hydraulic pressing, being the pretreatment that had the highest yield of macadamia oil. Rincón et al. (2016) stated that microwave pretreatment is an alternative to conventional heating since it is a source of energy supplied by an electromagnetic field that leads to rapid heating throughout the material thickness with reduced thermal gradients, resulting in better yield compared to classical thermal pretreatment.

According to Tewari et al. (2016), when a material is placed in a very high frequency electromagnetic field (microwave), there is a rapid change in direction of the field, causing the orientation of the dipoles to change. The microwave acts on different positive and negative ends that oscillate to align with the microwave alternating current. These high-speed oscillations cause friction, which heats the food.

Although the OHP sample obtained lower yield compared to MHP, OHP presented the highest oil extraction when compared with samples that did not receive pretreatments (HP and SP). The findings indicate that pretreatment with oven increases the oil yield, therefore it is a pretreatment alternative for oil extraction. Ramos et al. (2017) explained that pretreatment may be associated with the breakdown of the cellular structure and consequent release of the oil. This can be observed upon when combining the oven pretreatment with hydraulic or screw pressing, the oil yield was higher than the samples that did not received pretreatment (HP and SP).

Aguilar-Reynosa et al. (2017) state that microwave pretreatment has advantages over an oven. This is because the generation of heat from the microwave has more energy efficiency, reducing the contact time of the food and the energy source, avoiding overheating and reducing the degradation of the thermolabile compounds.

The pretreatments applied in the nuts increased the yield in both types of mechanical extraction. This result is in accordance with those of Wroniak et al. (2016), who explained that despite the advantages of mechanical extraction due to less degradation of bioactive compounds in the oil, since high temperatures are not used, low yields require complementary methods to become industrially viable. Therefore methods that apply microwave pretreatment become very interesting, and this method decreases the effect of degradation by heating.

**Peroxide value and free acidity value of macadamia oil**
The Table 2 presents the peroxide value and free acidity for macadamia oil samples extracted by the different methods. The peroxide value ranged from 0.81 to 6.33 meq O$_2$/kg. The sample that presented the lowest peroxide value was MHP (0.81), followed by HP (0.95), OSP (2), OHP (2.99), MSP (4.33) and SP (6.33). The samples that presented the lowest values of peroxide (MHP and HP) were not significantly different (p <0.01).

The samples MHP and HP obtained a much lower peroxide value than the samples that underwent a pretreatment with the oven (OHP and OSP). The samples that underwent the screw press treatment (OSP, MSP and SP) also had a higher peroxide value than those extracted by hydraulic press (MHP and HP).

This may be explained considering the fact that the pretreatment with the oven led to prolonged heating and, consequently, greater oil oxidation of the OHP and OSP samples, increasing the peroxide value. Moreover, oil samples extracted by screw presses are slightly heated when the extractor screw comes into contact with the sample. Heating occurs due to friction between the screw of the extractor and the sample, increasing the peroxide value in the samples OSP, MSP and SP. This heating does not occur in the hydraulic press.

Despite the different results, none of the samples exceeded the peroxide value of 10 meq/kg, the maximum established by the legislation (Brasil, 2005; Codex Alimentarius – FAO, 2015).

Regarding pretreatment, microwaves seem to lead to lower formation of the peroxide compounds in the oil,
a result that may vary according to the potency of the microwave oven. Rękasa et al. (2017) found that the higher the microwave power, the higher the peroxide value in macadamia oils. This occurs because the increase in microwave power is related to the increase in temperature due to agitation of the molecules, leading to oxidation of the oil. Also, the peroxide value increase significantly after 3 minutes of pretreatment.

Kaijser et al. (2000) reported that macadamia has an advantage of much lower peroxide formation compared to other nuts, because the nuts are protected by a thick pericarp, which decreases peroxide formation. Wall (2010) stated that the presence of phytochemical compounds in macadamia nuts also helps to protect the grains against oxidation reactions, as well as having human health benefits and prolonging walnut shelf life.

The values of free acidity for OSP (0.39% of oleic acid), MSP (0.46%), SP (0.36%) and HP (0.51%) did not present significant differences (p>0.01). The samples extracted by hydraulic pressing with pretreatments, MHP (0.64%) and OHP (0.67%), obtained higher free acidity values, and did not differ significantly by Tuckey’s test (p>0.01).

Considering that macadamia nuts have a profile rich in unsaturated fatty acids, the peroxide value and free acidity are important parameters to measure lipid degradation, indicating macadamia oil quality (Canneneddu et al., 2016). For free acidity, Codex Alimentarius and Brazilian law establishes that cold pressing for unrefined oils should have a maximum of 4.0 mg KOH/g and refined oils at a maximum of 0.6 mg KOH/g, so all samples are within standards.

Rodrigues et al. (2005) found that macadamia oil from hydraulic pressing has maximum acidity of 0.24%. This result is lower when compared to our findings. The high acid content is related to stored poorly macadamia (Rodrigues et al., 2005), as happens with commercial samples.

The samples that obtained less free acidity formation were the samples that received screw press press extraction (MSP, OSP and SP), with no significant difference among them by the Tuckey’s tests (p> 0.01).

Free acidity are the hydrolysis products of oil and fat oxidation during long-term storage or processing at elevated temperatures during heating (Mahesar et al., 2014). It is noted that the type of extraction, and not the pretreatment, influenced the increase in free acidity. Through gas chromatography (Table 3), it is possible to observe a higher unsaturated profile of the fatty acids present in samples that have been extracted by hydraulic pressing (HP, MHP and OHP) that directly influence the formation of free acidity.

### Fatty acid profile

The macadamia oils extracted by different methods, using pretreatment or not, displayed a diversified fatty acid profile (Table 3). The acids C14:0, C16:0, C18:0, C16:1n7, C18:1n9c, C18:2n6c, C18:3n3, C20:1n9 and C18:3n3 were identified. The results revealed that extraction with screw and hydraulic pressing, set with pretreatments or not, did not influence the fatty acid profile of the oils. This result is of great importance, since it indicates that the best treatment applied for the extraction of macadamia oil may be done considering other factors, such as the highest yield and less oil degradation.

Kaijser et al. (2000) and Malvestiti et al. (2017) found a similar profile of the main fatty acids present in these nuts. The findings are in agreement with Malvestiti et al. (2017), who stated that macadamia contains up to 78% monounsaturated fatty acids, which are represented mainly by oleic (49-65%), palmitoleic (17-30%), and palmitic (8-9%) acids.

The extraction by cold pressing was studied by Prescha et al. (2014), who verified that macadamia oil is rich in oleic acid. The authors found approximately 58.9% oleic acid and 18.1% palmitoleic acid, indicating the high levels of monounsaturated fatty acids in macadamia nuts. Madawala et al. (2012) stated that macadamia nuts oil has high levels of palmitoleic acid (16:1), approximately 18%, which is not

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**Table 3: Fatty acid profile of macadamia nuts extracted by different methods: screw and hydraulic press, without and with pretreatments using microwave or oven**

| Fatty acid         | SP | MSP | OSP | HP  | MHP | OHP |
|--------------------|----|-----|-----|-----|-----|-----|
| Myristic (C14:0)   | 0.89| 0.88| 0.88| 0.34| 0.34| 0.39|
| Palmitic (C16:0)   | 9.21| 9.45| 9.33| 8.66| 8.68| 8.71|
| Stearic (C18:0)    | 3.43| 3.56| 3.60| 2.82| 2.66| 2.83|
| Palmitoleic (C16:1n7) | 19.67| 20.44| 20.81| 18.26| 18.06| 18.26|
| Oleic (C18:1n9)    | 61.01| 60.68| 60.48| 63.56| 64.70| 63.80|
| Linoleic (C18:2n6) | 2.24| 2.06| 2.08| 1.45| 1.41| 1.44|
| Gondoic (C20:1n9)  | 2.81| 2.67| 2.68| 2.53| 2.51| 2.21|
| α-linoleic (C18:3n3) | 0.13| 0.15| 0.14| 2.38| 2.29| 2.36|
| Saturated          | 13.53| 13.90| 13.82| 11.81| 11.67| 11.93|
| Unsaturated        | 86.47| 86.10| 86.18| 88.19| 88.33| 88.07|

**Legend:** SP = oil extract by screw press without any pretreatment, HP = oil extracted by hydraulic press without any pretreatment, MSP = pretreatment with microwave followed by screw press extraction, OSP = pretreatment with microwave followed by hydraulic press extraction, MHP = pretreatment with microwave followed by hydraulic press extraction and OHP = pretreatment with oven followed by hydraulic press extraction.
common in many specialty oils such as walnut, halzenut, argan, avocado and grapeseed.

Souza et al. (2007) also showed similar amounts of saturated (16.22 - 16.91%) and unsaturated (82.35-83.61%) fatty acids. The authors agreeded that macadamia oil has higher levels of C20:0, C20:1, C22:0, C22:1 and C24:0 fatty acids than most vegetable oils. This profile is important, enabling this oil characteristic to be used to distinguish and find macadamia oils with possible adulterations.

**Saponification and Iodine Values**

The iodine value varied from 77.7 to 82.7 g I2/100g and the saponification value from 194.3 to 195.8 mg KOH/g (Table 2). In view of the results obtained, it is possible to note the low variation of the saponification and iodine values in different extraction methods. Both the hydraulic and the screw press extraction showed similar results, which was expected, since no variation was observed in the fatty acid profile (Table 3).

According to Kittiphoom and Sutasinee (2015), the saponification value is a measure of the alkali reactive groups in fats and oils. Glycerides containing short-chain fatty acids have a higher saponification value than those with longer-chain fatty acids. Moodley et al. (2007) found a saponification value of macadamia oil of 193.7 mg KOH/g oil, and for iodine a value of 78.3 g I2/100 g, indicating values very similar to the findings for all samples analyzed in the present study (approximately 194 mg KOH/g oil for saponification value and 75 g I2/100 g for the iodine value).

The iodine value indicates the degree of unsaturation of the fatty acids present in macadamia. The HP, OHP and MHP samples that had the highest content of unsaturation (Table 3) also had the highest iodine value. The findings are also in accordance with those of Rengel et al. (2015), who stated that macadamia oil has low values of polyunsaturated fatty acids, and more than 80% monounsaturated fatty acids, as evidenced in its composition obtained by gas chromatography (Table 3). This justifies the higher value of the iodine value of all samples of macadamia oil.

A study carried out by Navarro and Rodrigues (2016) cites of iodine content values between 72 and 78 g I2/100g for 4 species of the product, values being found in the present study are also within this range of variation.

**Oxidative Stability**

The oxidative stability of the macadamia oil samples extracted under different conditions ranged from 17.80 to 48.7 hours (Table 2). The most stable sample was the HP (48.7 hours), with no significant difference (p >0.01) compared to the MHP sample (47.52h), followed by MSP (35.24h). The results of the samples OHP (22.97h), SP (18.60) and OSP (17.80h) did not show significant differences (p > 0.01), presenting the lowest oxidative stability.

The treatments with highest oxidative stability were those that presented less heating during the extraction (HP and PM, followed by MSP). These results show the importance of choosing the adequate pretreatment to maintain oil quality. The extraction methods and the chosen pretreatment should not only increase the yield, but also avoid its degradation as much as possible.

The samples that received pretreatment with oven (OHP and OSP) showed very low oxidative stability when compared to other treatments. This was possibly due to the increase in temperature, since the higher the temperature, the faster the oxidation reaction rates.

All oils have a resistance to oxidation, which is related to several factors. One of those factors is the degree of unsaturation, in which the higher the number of unsaturations the faster the lipid oxidation. The oxidation starts slowly and then accelerates very quickly. The moment it starts to accelerate a lot is called Oxidative Stability, measured by the time the increase in conductivity occurs due to the formation of volatile acids (Garcia-Moreno et al., 2013).

The results obtained for all the oil samples were better than the results obtained by Waal (2010), who investigated the oxidative stability of 7 macadamia cultivars and obtained a maximum stability of 10.08 hours. This difference in results might be explained considering the solvent extraction techniques used by Wall (2010), which may have diminished the oxidative stability of the oil. In the Wall study, the author extracted the oil from the same nuts twice, increasing the yield and decreasing its oxidative stability. In this point, microwaves and oven applied in the nuts for a short period of time may have an advantage over solvent extraction, decreasing oxidation reactions.

Other authors also evaluated the oxidative stability of different oil types. Saoudi et al. (2016) found that heating decreases the oxidative stability of soybean oil, ranging from 3.29 to 0.63 hours after 24 hours of heating. Malheiro et al. (2012) stated that microwave heating induces oxidation advancement in olive oils, decreasing stability when compared to the control treatment, that did not receive microwave pretreatment. According to the findings of the present study, microwaves degraded the oil samples less when compared to oven heating.
CONCLUSIONS

Different pretreatments and extraction methods applied in macadamia nuts influenced the properties of the oil extracted from the nuts. The use of oven or microwave as pretreatments increased oil yield. The different pretreatment and extraction methods applied in the present study did not presented difference in the fatty acid profile of the samples. This result is very important, indicating that oven and microwave did not negatively influence this profile. Thus, other characteristics must be consider to evaluate the macadamia oil quality. The oils extracted by hydraulic pressing combined with microwave as pretreatment provided the highest yield, the best oxidative stability and lowest peroxide value. The results suggest that the combination of microwaves and hydraulic pressing extracts oils with quality superior to the other treatments studied.

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Authors’ contributions
Ana Paula Lima Ribeiro: Conceptualization; Methodology; Formal analysis; Investigation, Writing - Original Draft, Project administration, Data Curation, Funding acquisition, Supervision. Felipe Furtini Haddad: Investigation, Writing - Original Draft; Funding acquisition, Supervision, Data Curation. Talita de Sousa Tavares: Conceptualization; Investigation. Kassiana Teixeira Magalhães: Conceptualization; Investigation. Carlos José Pimenta: Conceptualization; Resources; Supervision. Cleiton Antônio Nunes: Conceptualization; Investigation, Resources, Data Curation, Supervision.

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