Original Article
Food Science and Technology
ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)
DOI: https://doi.org/10.5327/fst.14523

Extraction of stem oils from *Cinnamomum cassia* and NMR characterization to produce nutraceuticals

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Abstract

Functional foods offer health benefits as a function of their bioactive compounds, beyond their basic nutritional functions. They can, for example, reduce the risk of chronic degenerative diseases such as cancer, diabetes, and cardiovascular damage, among others. Nutraceuticals can be produced using the bioactive compounds present in these foods. Essential oils are used according to the need and/or specific characteristics of their active ingredients. The objective of this study was to extract essential oil from the stem of *Cinnamomum cassia* by hydrodistillation employing the Clevenger apparatus. The results show the extraction of a dense yellow oil. This oil was characterized by solution nuclear magnetic resonance using the ^{1}H and ^{13}C nuclei. The results showed a correlation in 92% of cinnamaldehyde. The nutraceutical containing this oil was obtained from the nanoprecipitation process, generating a nanoencapsulation of the oil in the polycaprolactone. This new material was characterized by scanning electron microscopy. The micrography showed the homogeneous nanostructure of the nanoencapsulated material.

Keywords: cinnamaldehyde; nutraceutical; nanoencapsulation; scanning electron microscopy**.**

Practical Application: The article brings innovative content regarding the extraction of cinnamon oil. This was extracted from the stem of the plant and characterized by the NMR technique.

1 INTRODUCTION

Cinnamon is a spice obtained from the inner bark of several species of plants of the genus *Cinnamomum* (Lauraceae family), which is used in both sweet and savory foods. The term "cinnamon" also refers to the brownish color of the spice after it is ground. *Cinnamonum* oils are extracted by the distillation of the leaves or bark of the plant, which produce volatile components. The main *Cinnamomum* oils found in the world market are *Cinnamomum verum* oils: *C. cassia* and *Cinnamomum camphora*. Cinnamomum species are generally mined in minimal amounts (Guenther, 1950).

Cinnamon bark oil has a delicate spice aroma and a sweet, spicy taste. Its main constituent is cinnamaldehyde and other minor components that transmit its characteristic aroma and flavor. It is mainly employed in the flavoring industry, where it is used in seasonings for meats, sauces and pickled vegetables, bakery products, confectionery, and cola-type drinks; to add flavor in cigarettes; and in dental and pharmaceutical preparations (Coppen, 1995; Guenther, 1950).

Studies developed by the American Heart Association showed that cinnamon can decrease the risk of cardiovascular damage caused by a high-fat diet, activating antioxidant and anti-inflammatory molecules in the body, demonstrating one of the benefits of cinnamon for humans. (Moncada et al., 2022; Rahmatullah et al., 2009; Unlu et al., 2010; Zanardo et al., 2014; Ziegenfuss et al., 2006). Other cinnamon applications have also been studied, such as antimicrobial, antioxidant, bactericidal, antidermolytic, and antifungal actions (Doyle & Stephens, 2019; Faix et al., 2009; Makimori et al., 2020; Ranasinghe et al., 2002; Unlu et al., 2010; Shi et al., 2021).

Traditional methods of extracting essential oils use aggressive solvents under agitation and heating, which are distilled to later separate the oil, through the "Soxhlet" device. In these steps, many toxic residues are produced, in addition to requiring a large amount of energy to extract the oils. Therefore, new studies for the extraction of essential oils have used green solvents (ecological solvents) instead of the more aggressive ones, such as petroleum derivatives, thus directly impacting people's quality of life, as they generate less toxic waste and consequently less environmental pollution. Extraction using green solvents requires a microwave-assisted hydrodistillation technique (In fact, the effectiveness and the rapidity of the microwave-assisted hydrodistillation extraction were due to the instantaneous production of radiation of the electromagnetic waves and quick heat of the plant material. The continuous increase in the thermal stress and the internal pressure in the cell lead to the breakage of the cell walls to release the volatile compounds from the inside to the outside of the cell or sonohydrodistillation. The sonohydrodistillation was particularly efficacious due to the

Received 22 Feb., 2023
Accepted 18 Jun., 2023
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principle of the cavitation phenomena, which helps the penetration of the solvent (distilled water) to the plant material. The violent collapse of the bubbles led to the macro-turbulences and the micro-mixing produced by the acoustic cavitations in an alternating pressure field. This phenomenon caused the cell disruption and augmented the mass transfer intensification from the cell content to the solvent (distilled water), and several articles have studied the efficiency of these techniques (Al-Ajalein et al., 2023; Benmoussa et al., 2022; Chen, 2021; Jeyaratnama et al., 2016; Kim, 2017).

The median lethal dose (LD50) gives a measure of the immediate or acute toxicity of a chemical in a particular animal species being tested and also gives an idea of the margin of safety. The basic idea of this test was to force-feed the healthy animals enough drugs to kill approximately 50% of them. The LD50 test is expected to give an idea at what dose cinnamaldehyde produces side effects if any and at what dose 50% of the animals will be killed. LD50 studies of cinnamaldehyde seem to have no toxic effects even with the administration of 0.4 g/kg body weight and effective dose, suggesting that its margin of safety is much higher (Anand et al., 2010).

Anand et al. (2010) performed bioassay-guided fractionation of chloroform extract of *Cinnamomum Z.* and identified cinnamaldehyde as an active principle against diabetes. The detailed study was undertaken to elucidate its mode of antidiabetic action in streptozotocin (STZ)-induced diabetic rats. Their results indicated that the median lethal dose of cinnamaldehyde could not be obtained even with 0.4 g/kg body weight of its effective dose.

Babu et al. in 2007 investigated the hypoglycemic and hypolipidemic effects of cinnamaldehyde in STZ-induced diabetic rats. They extracted cinnamaldehyde from *Cinnamomum Z.*, and the results of the bioanalysis indicated that there was a decrease in plasma glucose levels. That oral administration of cinnamaldehyde produces a significant antihyperglycemic effect, lowers both total cholesterol and triglyceride levels, and, at the same time, increases high-density lipoprotein cholesterol in STZ-induced diabetic rats. In the acute toxicity study in male Wistar rats, graded doses of cinnamaldehyde were administered orally and the median lethal dose value was 1850737 mg/kg body weight. Based on this observation, doses below this threshold can be considered safe in mammals.

Therefore, nutraceuticals must be produced following these cinnamaldehyde concentrations at safe levels.

It is known that nuclear magnetic resonance (NMR) is a very good tool to characterize materials in solution (Batista et al., 2020) or solid state (Rocha et al., 2022). NMR is an absolute technique and can give fundamental and important information on chemical structure, through solution techniques, and from molecular dynamics when the samples were analyzed by solid-state measurements, due to the determination of relaxation parameters. In this sense, NMR gives much information on the molecular organization of materials.

The technique of NMR spectroscopy is one of the main spectroscopic techniques to identify and quantify organic components, and it is based on the study of the interaction between electromagnetic radiation, in the radiofrequency region, with certain atomic nuclei of matter, both subjected to a strong magnetic field. By NMR, it is possible to employ several types of nuclei found in nature, such as ^{1}H, ^{13}C, ^{31}P, ^{19}F, ^{14}N, and ^{15}N. The nuclei that are commonly available for the identification of organic materials are those of ^{1}H and ^{13}C. From the ^{1}H NMR spectrum, it is possible to determine the number of each different type of this nucleus in the molecular structure and also obtain information about the nature of the environmental ambient of each type. The ^{13}C NMR spectrum provides the determination of the organizational structure of carbon atoms in molecular chains. Each group occupies a certain position in the spectrum relative to a reference, and their relative proportion can be obtained by integrating their curves area within the spectrum. Generally, the analyses of ^{1}H and ^{13}C NMR spectra make possible to identify unknown compounds (Paiva et al., 2010; Silverstein et al., 2005).

The time-domain NMR (TD-NMR) technique is based on measuring the longitudinal, T_1, or spin-lattice relaxation times (the result of the existence of transient magnetic moments produced by the rotational and translational movements of neighboring molecules) and transverse, T_2, or spin-spin (related to the loss of coherence that is attributed the direct interactions between the individual magnetic moments of the spins in a sample). The T_1 and T_2 relaxation times are directly related to the mobility of water and fat in food, so it is widely used in quality control in the food industry (Steiner et al., 2010).

In this sense, cinnamon and its extracts were characterized by NMR, applying spectroscopic techniques (NMR) and time-domain techniques (time-domain NMR (TD-NMR)).

2 OBJECTIVE

In this context, this article aimed to extract cinnamaldehyde from the stem of *C. cassia* using green solvent, characterize it by NMR, and develop the nutraceutical from the nanoencapsulation of the extract in polycaprolactone (PCL).

3 EXPERIMENTAL

3.1 Sample preparation

The stem of *C. cassia* was used as the raw material. The stem was turned into powder using a Solab Cientifica knife mill, model GL30, series 0021, 300 w. The grains were sieved using two Bertel Indústria Metalúrgica LTDA grain fractionation sieves with openings of 80 mesh (6.35 mm) e 35 mesh (13.5 mm).

3.2 Essential oil extraction using the Clevenger apparatus

The essential oil was extracted by hydrodistillation using the Clevenger apparatus. The Clevenger apparatus consists of a 500 mL flask heated by a thermal blanket (Fisatom, model 52E, series 1595523, 200 W), where the generated steam is conducted through the system to a vertical condenser. The steam with the extracted oils is condensed and the liquid products are stored in a separator funnel with a valve, as shown in Figure 1. The oil sample extracted by the Clevenger apparatus will be designated as "Cinnamon oil extracted by the authors."

3.3 Nuclear magnetic resonance

3.3.1 Time-domain magnetic resonance

Time-domain relaxometry measurements were used to determine nuclear relaxation times by using the Oxford Instruments® MARAN Ultra equipment, operating at 0.54 T (23.4 MHz for the ^{1}H) equipped with an 18 mm probe at 30°C. The following two pulse sequences were used.

To obtain the longitudinal relaxation times (spin-lattice) of hydrogen for the sample phases, the sequence of small-angle flip-flop (SAFF) pulses (Cucinelli Neto et al., 2019) was applied, using the following parameters: 90° pulse (duration 7.5 µs), number of scans 16, and receiver gain 42 dB. The transverse relaxation times (spin-spin) of hydrogen for solid samples were applied to the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence using the following parameters: 90° pulse (duration 7.5 µs), time between echoes 600 (2t, µs), number of echoes 8192, points per echo 1, number of scans 4, recycle time 1, and receiver gain 40 dB.

3.3.2 Nuclear magnetic resonance spectroscopy

NMR spectra in solution were acquired in a Varian Mercury VX 300 using a 5 mm diameter probe. Both the extracted sample and the cinnamaldehyde standard were prepared soluble in deuterated dimethylsulfoxide (DMSO-d6). NMR spectra were acquired using the standard pulse sequence of the equipment for ^{1}H and ^{13}C nuclei. The acquisition parameters for the ^{1}H spectrum (299.9 MHz) were as follows: spectral width of 4,800 Hz; acquisition time of 2.5 s; pulse width calibration of 90°; delay time of 10 s; number of transients 16; and probe temperature of 40°C. The acquisition parameters for the ^{13}C spectrum (75.4 MHz) were as follows: spectral width of 18,868 Hz; acquisition time of 1.0 s; pulse width calibration of 90°; the delay time of 2.0 s; number of transients 1,200; decoupler mode set on throughout acquisition, and probe at a temperature of 40°C.

All spectra were processed using the program MestreNova 12.0 (MestreLab, Spain).

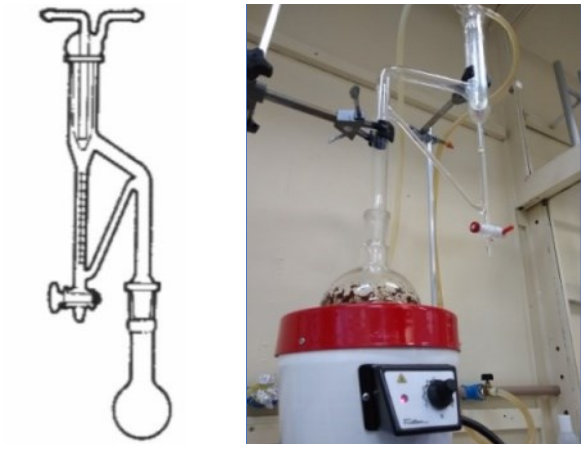

Figure 1. Hydrodistillation using the Clevenger apparatus.

3.4 Encapsulation process

The encapsulation methodology consisted of preparing two solutions: 100 mL of distilled water + 120 mg of Pluronic® and 50 mL of acetone + 200 mg of PCL polymer and 1 mL of extract extracted from cinnamon. Both remained under magnetic stirring until the total solubilization of the reagents. Thus, the solutions were combined in the same container and again remained for 100 h under constant agitation at room temperature, where nanoprecipitation occurred. After this period, the sample was placed in a freezer for 24 h at 62°C and finally in the lyophilizer under a pressure of 2,000 µHg at 47°C (Tavares et al., 2017).

3.5 Characterization of encapsulated material

The characterization of the nanoencapsulation was carried out through micrography in a scanning electron microscope (SEM) using the TESCAN equipment, model MIRA fourth-generation LMU, equipped with a field-emission cannon (FEG, Schottky), secondary electron detector (SE), and backscattered electron detector (BSE).

4 RESULTS AND DISCUSSION

4.1 Essential oil extraction using the Clevenger apparatus

Cinnamon extraction was performed with 40 g of grains larger than 35 mesh (13.5 mm) and 300 mL of distilled water, producing 1 mL of essential oil after 3 h of distillation. The viscous oil extracted has a yellow color.

4.2 Nuclear magnetic resonance results

4.2.1 Time-domain nuclear magnetic resonance

Cinnamon powder samples were analyzed before and after the extraction of essential oils, the "Cinnamaldehyde Standard" and the "Cinnamon oil extracted by the authors." Figure 2 shows

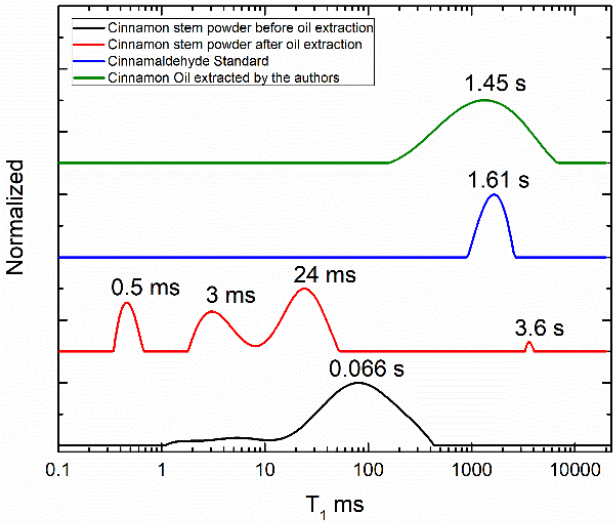

Figure 2. Longitudinal relaxation time curves (T_1).

the longitudinal relaxation time curves that have a time constant T_1. The results of the relaxation times such as T_1 and T_2 (time constant of spin-spin relaxation time) are listed in Table 1.

The curve referring to the cinnamaldehyde standard has a width at half height smaller than the other samples (cinnamon powder (before/after) and cinnamon oil extracted by the authors). This occurs due to the strength of the bonds between the molecules of the cinnamaldehyde sample, generating greater homogeneity in relation to the others (Cucinelli Neto et al., 2019). In the result of the cinnamon powder solid samples, only one predominant domain controls the relaxation process. In the curve referring to the powder before extraction, relaxation data of 66 ms can be seen, whereas in the cinnamon powder sample after extraction, other domains were formed (0.5, 3, 24, and 3,600 ms), which is due to the change in the structural organization, since the extract/water was withdrawn from the initial powder structure, generating new or multiples interactions causing a heterogeneity of the sample (Batista et al., 2020; Rocha et al., 2022; Steiner et al., 2010).

Due to the large amount of cinnamaldehyde in the analyzed cinnamon oil samples, the relaxation times varied around 1–2 s; however, the relaxation time of the oil sample extracted by the authors also maintained the same range (T_1=1.5 s and T_2=1.2 s), and the relaxation values are very similar to those of cinnamaldehyde standard (T_1=1.6 s and T_2=1.4 s), indicating a higher purity (Batista et al., 2020; Rocha et al., 2022; Steiner et al., 2010).

Table 1. Result of relaxation times through time-domain nuclear magnetic resonance.

Sample	T_1 (SAFF)	T_2 (CPMG)
Cinnamon powder before extraction	66.0 ms	-
Cinnamon powder after extraction	9.8 ms	-
Cinnamaldehyde Standard	1.6 s	1.4 s
Cinnamon oil extracted by the authors	1.5 s	1.2 s

4.2.2 Nuclear magnetic resonance

Figure 3 shows the identified result of the NMR spectrum of the cinnamaldehyde standard sample and its chemical shift characteristic of each nucleus (^{1}H or ^{13}C).

The cinnamaldehyde purity of this reference sample, which was calculated from the ^{1}H NMR by relative quantitation method (Bharti & Roy, 2012), was 95% of the molecular weight, where 5% refers to the solvent used (water and DMSO). Through NMR, it was possible to calculate the amount of cinnamaldehyde present in the cinnamon oil sample extracted by the authors, which was 92% of cinnamaldehyde.

To calculate the exact concentration in 1 mL of cinnamon oil extracted by the Clevenger apparatus, the analytical methodology by NMR based on the internal standard was applied (Claridge, 1999; Gödecke et al., 2013). The internal standard used for the analyses was N,N-dimethylformamide (DMF) (99.8%) due to its non-reactivity with cinnamon extract. The methodology consists of generating five samples of known concentration of the standard cinnamaldehyde P.A.

The calibration curve was generated from the ^{1}H NMR result, through the doublet (δ 9.67 and δ 9.70 ppm) of the aldehyde hydrogen of the cinnamaldehyde molecule in comparison with the singlet (δ 7.95 ppm) of the aldehyde hydrogen of the DMF internal standard molecule, as shown in Figure 4A. The calibration curve is represented in Figure 4B.

The quantification of the cinnamaldehyde concentration present in the cinnamon extract sample was calculated based on the linear equation of the calibration curve represented in Figure 4B. After preparing a new sample, containing an aliquot of the cinnamon extract with DMF (40%) and performing a new ^{1}H NMR, the cinnamaldehyde concentration was calculated, resulting in approximately 2 g/mL of cinnamaldehyde in the extract.

Anand et al. (2010) and Babu et al. (2007) revealed the potential of cinnamaldehyde for use as a natural oral agent, with both hypoglycemic and hypolipidemic effects. Based on the LD50 values of the cited references, nutraceuticals can be produced in safe concentrations of cinnamaldehyde.

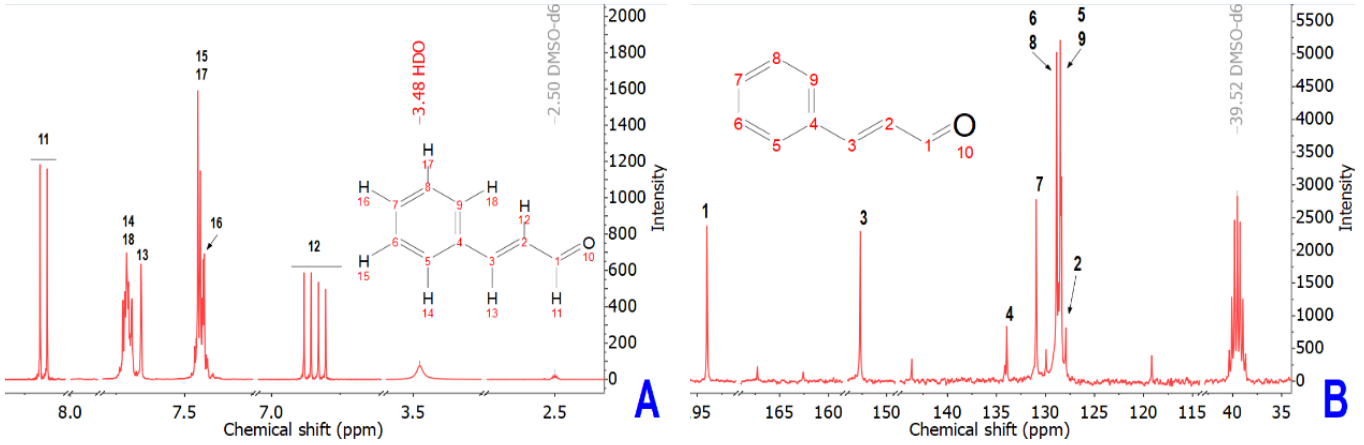

Figure 3. Cinnamaldehyde standard with DMSO-d6 solvent. (A) ^{1}H. (B) ^{13}C.

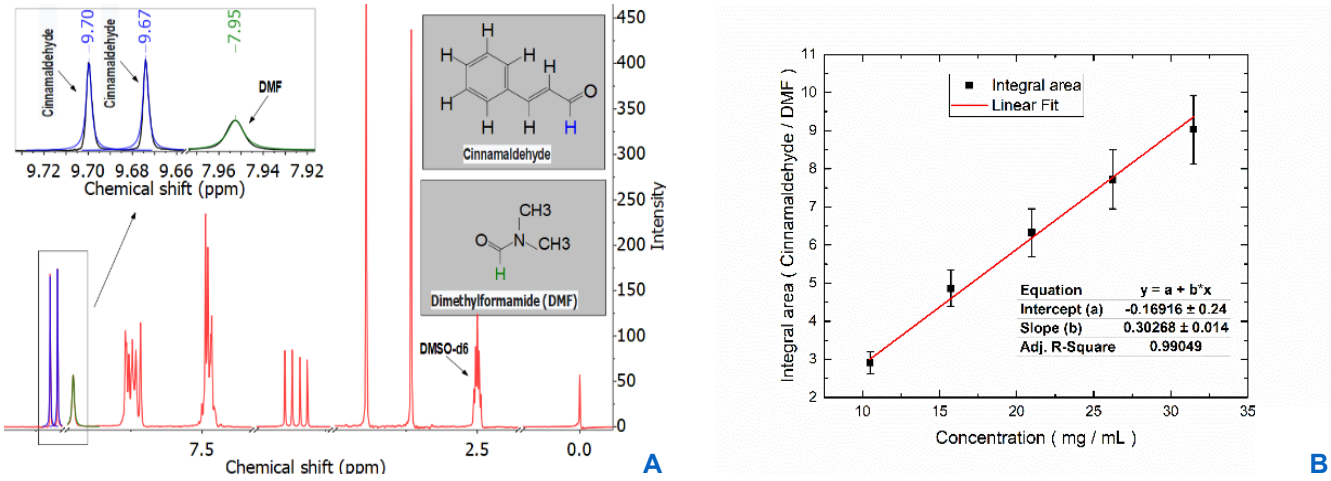

Figure 4. [1]H NMR result and the generated calibration curve. (A) NMR with the chemical shifts of the aldehyde cinnamaldehyde hydrogens P.A. and DMF aldehyde. (B) Calibration curve.

4.3 Characterization of the encapsulated material

The characterization of the nanoencapsulation was carried out through SEM. Figure 5 shows the morphology of micelles generated by nanoencapsulation of the extract in PCL. In Figure 6, a magnification at 100,000× reveals a micelle containing the internal oil.

Essential oils have low conductivity, and therefore, when present in micelles, oils accumulate electrons, revealing greater brightness in SEM micrographs (Goldstein et al., 2018). Thus, the presence of several micelles with the nanoencapsulated oils in Figures 5 and 6 can be observed.

Ferreira et al. (2022) proposed a new drug based on the PCL matrix and with nanoencapsulation of tea tree oil of the genus *Melaleuca alternifolia* and *Melaleuca linariifolia*, with the objective of combating the human papillomavirus. The authors chose *Melaleuca* oil due to its antioxidant, antifungal, antibacterial, anti-inflammatory, and anti-hyperproliferative properties. Their studies indicate that the PCL matrix acted as a cryoprotective agent for the nanoencapsulated oil with controlled release. Likewise, the nutraceutical based on the PCL matrix with nanoencapsulation of cinnamaldehyde oil will have a controlled release, obtaining the benefits of cinnamon essential oils for a longer time.

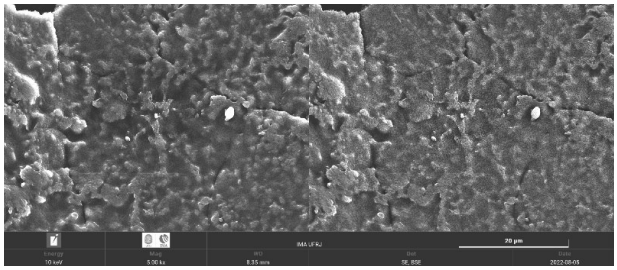

Figure 5. Micrograph performed on SEM-FEG with 5,000× magnification, with the left image generated by SE and the right images generated by BSE.

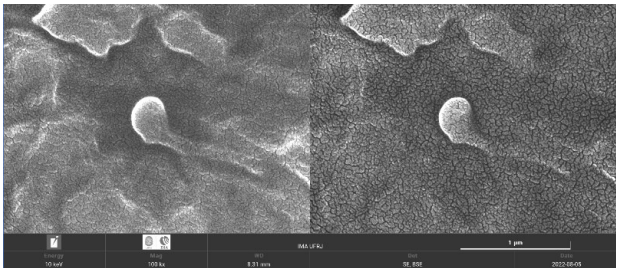

Figure 6. Micrograph of a micelle at 100,000× magnification, with the left image generated by SE and the right image generated by BSE.

5 CONCLUSION

The main objective of this study was achieved. The *C. cassia* stem extract was characterized by NMR, which allowed the precise identification of the cinnamaldehyde concentration present in the extracted sample. The generated nutraceutical presented a good homogeneity due to the multiple interaction formed as shown by SEM.

ACKNOWLEDGMENTS

The authors are grateful to Fundação de Amparo à Pesquisa do Estado do Rio de Janeiro - E-26/210.142/2019, for their financial support.

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