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Characterization, antioxidant capacity, and application of a microcapsule with essential oil from *Campomanesia adamantium* fruit residue in a cosmetic product

Caracterização, capacidade antioxidante e aplicação de microcápsula com óleo essencial de resíduo de fruto de *Campomanesia adamantium* em um produto cosmético

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ABSTRACT

Essential oils (EOs) are continuously explored from new sources, while encapsulation emerges as a promising strategy for their application in different fields. The aim of this study was to characterize microcapsules (MCs) containing EOs extracted from *Campomanesia adamantium* fruit residues, evaluate their antioxidant capacity and incorporate them into a cosmetic formulation. Two microcapsules (MCs) containing EO were prepared by complex coacervation with gelatin (G) and gum arabic (GA) in two ratios: MC1 (G:GA 1:2) and MC2 (G:GA 2:1). The chemical composition, EO retention, encapsulation efficiency, morphological features, solubility, hygroscopicity, thermal properties, infrared spectroscopy, and antioxidant capacity were analyzed. EO and MC were incorporated into an anti-aging cosmetic formulation, and the organoleptic characteristics, stability (4, 25, and 45 °C), spreadability, and microbiological quality of the cream were evaluated for 90 days. MC1 exhibited superior properties due to the greater proportion of gum arabic, which provided thermal protection, better water solubility, lower hygroscopicity, and a less porous surface. The MC1 and MC2 antioxidant capacities were similar to those of free EO, consistent with the results of the retention rates and encapsulation efficiency. Infrared spectroscopy confirmed the encapsulation, maintaining the main constituents of EO (α-pinene, limonene, β-ocimene, and β-caryophyllene). Incorporating the MCs and EO into a cosmetic cream resulted in the formation of a stable emulsion with good spreadability and consistent organoleptic properties over 90 days, suggesting that *C. adamantium* residue can be used in cosmetic formulations.

Index terms: Agrobiodiversity; bioactive compounds; *Cerrado*; *guavira*; volatile oil.

RESUMO

Os óleos essenciais (OEs) são continuamente explorados a partir de novas fontes, enquanto o encapsulamento surge como uma estratégia promissora para sua aplicação em diferentes campos. O objetivo do estudo foi caracterizar microcápsulas (MCs) contendo OE extraído de resíduos de frutos de *Campomanesia adamantium*, avaliar sua capacidade antioxidante e incorporar em uma formulação cosmética. Duas microcápsulas (MCs) contendo OE foram preparadas por coacervação complexa com gelatina (G) e goma arábica (GA) em duas proporções: MC1 (G:GA 1:2) e MC2 (G:GA 2:1). Foram analisadas composição química, retenção de OE, eficiência de encapsulamento, características morfológicas, solubilidade, higroscopicidade, propriedades térmicas, espectroscopia de infravermelho e capacidade antioxidante. OE e MC foram incorporados em uma formulação de creme cosmético antienvelhecimento, na qual a estabilidade (4, 25 e 45 °C), espalhabilidade, características organolépticas e qualidade microbiológica foram avaliadas por 90 dias. A MC1 exibiu propriedades superiores devido à maior proporção de goma arábica, que forneceu proteção térmica, melhor solubilidade em água, menor higroscopicidade e uma superfície menos porosa. As capacidades antioxidantes de MC1 e de MC2 foram semelhantes às do OE livre, consistentes com as taxas de retenção e eficiência de encapsulamento. A espectroscopia de infravermelho confirmou o encapsulamento, preservando os principais constituintes do OE (α-pineno, limoneno, β-ocimeno e β-cariofileno). A incorporação das MCs e do OE em um creme cosmético resultou na formação de uma emulsão estável com boa espalhabilidade e propriedades organolépticas consistentes ao longo de 90 dias, sugerindo que o resíduo de *C. adamantium* pode ser usado em formulações cosméticas.

Termos para indexação: Agrobiodiversidade; compostos bioativos; cerrado; guavira; óleo volátil.

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Introduction

Brazil is renowned internationally for its rich diversity of medicinal plants that contain bioactive substances with nutritional, pharmacological, and cosmetic utility (Vale et al., 2021). The fruit of *Campomanesia adamantium* (Cambess O. Berg), commonly known as guavira, is extensively found in the Brazilian Cerrado, and its pulp is used in juices, sweets, and other preparations (Cardoso, 2021). These fruits have various biological activities, such as an antiproliferative effect on melanoma cells in mice (Silva et al., 2018) and protection against oxidative stress by the pulp (Araújo et al., 2023). Additionally, fruit peels can inhibit cyclooxygenase 1 and 2 enzymes (Lescano et al., 2018) and have antihyperalgesic, antidepressant, and anti-inflammatory effects (Souza et al., 2017). During the

industrial processing of guavira, the peel and seed residues, which are generally discarded, contain bioactive compounds with beneficial properties, particularly essential oils (Alves et al., 2013; Bin et al., 2024; Viscardi et al., 2017).

The use of food waste, including underutilized plant parts, is essential for full utilization, supporting the circular economy and bioeconomy (Gonçalves & Maximo, 2023). One way to add value to this waste is by extracting essential oils (Teigiserova et al., 2021; Visakh et al., 2022). Essential oils (EOs) are hydrophobic and volatile liquids composed of mixtures of substances produced by plants, which mainly include terpenes and terpenoids (Masyita et al., 2022), and they are highly valued products because of their active ingredients and aromas, with applications in various industries (Al-Refaie, Mehyar, & Shahein, 2023). These ingredients can perform biological activities such as antioxidant (Ferreira et al., 2021), antitumor (Weimer et al., 2021), anti-inflammatory (Viscardi et al., 2017), antifungal (Borotová et al., 2022), analgesic (Viscardi et al., 2017), antibacterial (Borotová et al., 2022; Mohammadi, Ghaboos, & Almasi, 2023), insecticidal (Silva et al., 2022), and aromatic properties (Radünz et al., 2019). According to the Ministry of Industry, Foreign Trade, and Services (Brasil, 2023), the market for EOs, perfumes, and flavors increased by 24% in 12 months, with exports generating 560 million USD during this period.

Although EOs have a high value, their volatility and susceptibility to environmental factors make their use difficult (Himed et al., 2019; Wang et al., 2021), prompting the development of technological strategies to address these issues. EO encapsulation was proposed as a solution to preserve active ingredients and aromas (Sundar & Parikh, 2023), create barriers against degradation (Bastos et al., 2020), and enable the controlled release of bioactive compounds (Rutz et al., 2017).

Various encapsulation techniques have been used for EO, including complex coacervation (Hernández-Nava et al., 2020), spray drying (Balci-Torun, 2023), liposomes (Lohani et al., 2021), and ion gelation (Volić et al., 2018), among others (Sundar & Parikh, 2023). Complex coacervation is an effective method for encapsulating oils and lipophilic compounds through electrostatic interactions between polymers with opposite charges, forming a protective wall (Khatibi et al., 2021; Tavares & Noreña, 2020). The choice of polymers and encapsulation conditions plays an important role in ensuring the application potential of microcapsules (Bastos et al., 2020; Sundar & Parikh, 2023). The combination of gelatin and gum arabic is a classic system for complex coacervation and has been used successfully to form microcapsules for EO with high protection of the encapsulated material (Bin et al., 2024; Khatibi et al., 2021; Wang et al., 2021).

The cosmetics, perfume, and personal care industries are quickly adopting innovative technologies and scientific advancements to meet consumer demands for using safer and more natural products. The demand for innovative cosmetic products formulated with natural or nutraceutical substances is increasing (Associação Brasileira da Indústria de Higiene Pessoal Perfumaria e Cosméticos - ABIHPEC, 2024; Sharmeen et al., 2021). Owing to their biological properties and valuable aroma (Borotová et al., 2022), EOs are used in various cosmetic products, both in free and encapsulated forms (Carvalho, Estevinho, & Santos, 2016; Lohani et al., 2021; Sendi et al., 2023). A study (Bin et al., 2024) focused on optimizing the microencapsulation process of EOs extracted from *C. adamantium* fruit residues using complex coacervation with gelatin and gum arabic. These results encouraged further research on the application of microcapsules and the use of waste.

This study was conducted because research on the application of *C. adamantium* EO is scarce; the findings of this study may contribute to the field of cosmetics. The objectives of this study were to characterize the EO microcapsule from the fruit residue of *C. adamantium*, compare the antioxidant capacity of free EO and microencapsulated oil, and assess whether EO and microcapsules can be incorporated into a cosmetic formulation.

Material and Methods

Plant material and extraction of EO

The fruits of *C. adamantium* were obtained in the city of Ponta Porã, Mato Grosso do Sul, Brazil (latitude 22° 32’ 09”; longitude 55° 43’ 33”). One sample was deposited in the herbarium of the Federal University of Grande Dourados, Dourados, Mato Grosso do Sul, Brazil (DDMS 4602 – Sisgen n° A37EC3E). The fruits were manually pulped, and the residue (peel and seeds) was placed in a dehydrator with an airflow of 0.5 m/s at 50 °C and a maximum humidity of 10% (about 24 h). The residue was crushed in a multiprocessor (Mondial® Turbo Pratic MP-16-B, Bahia, Brazil), and the EO was extracted from this residue by hydrodistillation in a Clevenger apparatus for 150 min, following the method described by Viscardi et al. (2017). The EO was transferred to an amber glass bottle containing anhydrous sodium sulfate to remove moisture and stored at –18 °C until use.

Microencapsulation

The microcapsules were prepared by complex coacervation and subsequently freeze-dried using gelatin (G; 1% w/v) and gum arabic (GA; 1% w/v) as wall materials, following the methodology described in another study (Bin et al., 2024). Two formulations of microcapsules, MC1 (G:GA 1:2) and MC2 (G:GA 2:1), were selected based on a study that investigated the polymer ratio and the amount of EO in microcapsule preparation (Bin et al., 2024). The concentration of EO used did not differ between the two formulations (40.3%, w/w). Moreover, two oil-free control formulations, known as CT1 (G:GA 1:2) and CT2 (G:GA 2:1), were developed using the same method in which the proportions of the wall materials were taken into account.

Characterization of microcapsules

Chemical composition and retention rate of the EOs in the microcapsules

To analyze the chemical composition of the microencapsulated essential oil (MEO), the microcapsules were initially mixed with hexane (50 mg/mL) and placed in an ultrasonic bath for 10 min. The organic fraction containing the EO was collected, and the solvent was evaporated under a fume hood. After drying, the sample was dissolved in hexane, and the newly extracted EO was characterized by gas chromatography coupled with mass spectrometry (GC-MS) on a chromatograph (Shimadzu QP2010 Plus, Shimadzu, Tokyo, Japan). Chromatographic separation was performed using a DB-5 column (J & W Folsom, California) with 5% phenyl-dimethylpolysiloxane (30 m × 0.25 mm × 0.25 mm) under the following conditions: helium carrier gas (99.999%) at a flow rate of 1 mL/min and a 1 µL injection volume split ratio of 1:10. The column temperature started at 50 °C and was increased to 250 °C at 3 °C/min. The injector, injector transfer line, and detector were maintained at 250, 290, and 290 °C, respectively. The MS scan parameters included an ionization voltage of 70 V and a mass variation of 50–600 Da over a 0.3 s interval. The retention indices were calculated using a mixture of normal alkanes (C_7–C_{40}) as an external reference. The components were identified by comparing the mass spectra of the samples with the spectra available in the NIST21 and WILEY229 libraries (Adams, 2007).

The EO retention rate in the microcapsules (%) was determined by considering the initial amount of EO (g) in each formulation using Equation (1).

$$\text{Retention rate}(\%) = \frac{M_f}{M_i} \times 100 \quad (1)$$

Here, M_f represents the mass (g) of EO extracted from the microcapsule (%) and Mi represents the mass (g) of EO incorporated into the microcapsule formulation.

Encapsulation efficiency

The encapsulation efficiency was determined using a modified method described by Badke et al. (2019). The procedure involved separating the outer essential oil (EO_E) from the microcapsules before extracting the inner essential oil (EO_I). A 500 mg sample of microcapsules was mixed with 5 mL of hexane, and the mixture was gently stirred for 15 s. An aliquot of 2 mL of the hexane fraction containing the EO was removed. The procedure was repeated three more times to obtain the EO_E fraction. The remaining microcapsules were treated with 20 mL of hexane to extract the EO_I. The mixture was shaken for 2 min, left in an ultrasonic bath for 30 min, transferred to a container with a lid, and incubated for 24 h with constant stirring (250 rpm). After incubation, the mixture was vacuum-filtered using a calibrated filter crucible with a porous plate and washed three times with 5 mL of acetone and 5 mL of hexane to remove the EO_I. The crucible with the retained material was dried at 70 °C until a constant mass was obtained. The mass of the EO_I was quantified based on this mass and the original mass of the sample. The total essential oil (EO_T) content was determined by including the EO_E in the extraction process. This method minimized errors caused by the volatilization of EO. The encapsulation efficiency was calculated using Equation 2 (Ferreira & Nicoletti, 2021).

$$\text{Encapsulation Efficiency}(\%) = \frac{EO_I}{EO_T} \times 100 \quad (2)$$

Here, EO_I and EO_T indicate the mass (mg) of the inner essential oil extracted from the microcapsules and the total essential oil present in the microcapsules, respectively.

Solubility, hygroscopicity, and water activity

The water solubility of the microcapsules was determined using the gravimetric method (Cano-Chauca et al., 2005), with modifications. Briefly, a sample of 500 mg of microcapsules was homogenized with 50 mL of distilled water on an orbital shaking table at 200 rpm for 30 min and then centrifuged at 3000 rpm for 5 min. Next, a 25 mL aliquot of the supernatant was transferred to a Petri dish and dried in an oven with continuous air circulation at 105 °C until it reached a constant mass (mg). The solubility was determined based on the initial sample mass (mg) dissolved in a 25 mL aliquot using Equation 3.

$$\text{Solubility}(\%) = \frac{\text{Mass}_{\text{dried}} - \text{Mass}_{\text{dish}}}{\text{Mass}_{\text{initial sample}}} \times 2 \times 100 \quad (3)$$

The hygroscopicity of the sample was determined following the method described by Cai and Corke (2000). First, a sample of 500 mg of microcapsules was weighed and stored in a saturated sodium chloride environment (76.8% equilibrium relative humidity) at 25 °C for seven days in triplicate. Then, the samples were weighed every 24 h until their mass no longer changed. Hygroscopicity was expressed as a percentage (%) of adsorbed moisture and calculated using Equation (4).

$$\text{Hygroscopicity}(\%) = \frac{\text{mass of adsorbed water}(\text{mg})}{\text{mass of sample}(\text{mg})} \times 100 \quad (4)$$

The water activity of the microcapsules was determined by directly reading the value from the Aqualab Pre Dew (USA) water activity meter at 25 °C.

Morphology and particle size

The morphology of the wet microcapsules was analyzed using an optical microscope (Nikon Eclipse E200, Japan) coupled with a photographic camera (Moticam 2300, 3 M Pixel, Nikon, Japan) and the Motic Image Plus software (100x magnification). The images were used to measure the average diameter (μm) of at least 300 particles (Ferreira & Nicoletti, 2021). The morphology of the freeze-dried microcapsules was analyzed by scanning electron microscopy (SEM). The microcapsules were placed on double-sided carbon tape, attached to aluminum pins, and metalized with a thin layer of gold (JEOL microscope, model JSM-6380LV, Tokyo, Japan). The images were captured with a voltage acceleration of 15 kV at a working distance of 11 mm and magnified at 800x and 3000x.

Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA)

The absorption spectra of the EO, wall material, and microcapsule samples were recorded using a Fourier transform infrared (FTIR) spectrophotometer (Shimadzu IR Affinity-1, Tokyo, Japan) using tablets dispersed in potassium bromide. The samples were analyzed at wavelengths of 4000–500 cm^{-1}, a resolution of 40 cm^{-1}, and 32 scans (Rohman & Man, 2010).

The TGA curves of the EO, wall material, and microcapsule samples were generated with a TGA Q500 (TA Instruments, EUA) using a platinum crucible. The parameters used for the analysis were as follows: nitrogen atmosphere, speed of 60 mL/min, temperature range of 25–700 °C, and heating speed of 10 °C/min.

Antioxidant capacity

The antioxidant capacity was determined using the ferric reducing antioxidant power assay - FRAP (Oliveira et al., 2019) and the $ABTS^{\bullet+}$ radical neutralization assay (Magalhães et al., 2020) in free EO and microencapsulated EO (MEO). Synthetic antioxidant standards, such as ascorbic acid (0.2 to 8.0 μg/mL) and BHT (5 to 500 μg/mL), were used as positive controls.

The antioxidant capacity of the MEO was evaluated after extracting the EO, following the method described by Yang et al. (2014), with modifications. An aliquot of 500 mg of microcapsules was added to 20 mL of ethanol and vortexed for 2 min. Then, the mixture was placed in an ultrasonic bath for 30 min and centrifuged at 10,000 rpm for 15 min. The supernatant was transferred to a 25 mL volumetric flask, and the volume was made up of ethanol.

Ferric reducing antioxidant power assay (FRAP): FRAP reagent was prepared from 25 mL of sodium acetate buffer (0.3 M, pH 3.6), 2.5 mL of 2,4,6-tri(2-pyridyl)-s-triazine (TPTZ) solution (10 mM), and 2.5 mL of ferric chloride (20 mM). Aliquots of 90 μL of EO (250–2000 μg/mL) or MEO (100–1000 μg/mL) were mixed with 270 μL of distilled water and 2700 μL of FRAP reagent. The mixture was incubated at 37 °C for 30 min, and the absorbance was recorded at 595 nm (Libra S60PC spectrophotometer, Biochrom, England). The antioxidant capacity of the samples was calculated using the equation obtained from the standard curve of ferrous sulfate, with concentrations ranging from 0.5 to 2 mmol/L ($y = 0.0228x - 0.1392$; $R^2 = 0.9999$). Ethanol was used as a negative control. The results were expressed as mmol ferrous sulfate/g.

$ABTS^{\bullet+}$ assay: $ABTS^{\bullet+}$ radical solution was prepared by mixing 5 mL of ABTS solution (7 mM) with 88 μL of potassium persulfate (140 mM) and keeping it in the dark at room temperature for 12–16 h. Next, 1 mL of the mixture was diluted with ethanol until the absorbance was 0.70 ±0.05 at 734 nm (Libra S60PC spectrophotometer, Biochrom, England). Aliquots of 100 μL of EO (250–2000 μg/mL) or MEO (100–1000 μg/mL) were mixed with 2900 μL of the $ABTS^{\bullet+}$ radical solution. After stirring, the mixture was incubated in the dark for 40 min until the absorbance was read at 734 nm. Ethanol was used as a negative control. The antioxidant capacity was expressed as the inhibition of the $ABTS^{\bullet+}$ radical (%I) at 1000 μg/mL free EO or MEO (Equation 5). The inhibition capacity of 50% of the $ABTS^{\bullet+}$ radical (IC_{50}) by free EO and MEO was also determined by plotting the percentage of inhibition of the $ABTS^{\bullet+}$ radical (%I) versus sample concentration (μg/mL).

$$\text{Inhibition of } ABTS^{\bullet+}\,(\%) = \frac{(ABS_{CT} - ABS_{SP})}{ABS_{CT}} \times 100 \quad (5)$$

Here, ABS_{CT} represents the negative control absorbance and ABS_{SP} represents the sample absorbance.

Cosmetic formulation incorporating EO and microcapsules

The formulation of the anti-aging cream cosmetic containing the EO and the microcapsules is shown in Table 1. The composition of the nonionic cream base (Lot 13024/2023, Botanik Cosmetics, Campo Bom/RS, Brazil) includes water, cetostearyl alcohol, ceteareth 20, glyceryl stearate, soy glycerides, shea butter, ascorbyl palmitate, citric acid, capric and caprylic acid triglycerides, cyclopentasiloxane, dimethicone, phenoxyethanol, methylisothiazolinone, propylene glycol, and disodium EDTA. Along with retinol, vitamin E, hyaluronic acid, and Longevicell® (INCI: Water & Hydrolyzed Myrtus Communis Leaf Extract), 1% of the EO extracted from the *C. adamantium* residue and 3% of the microcapsules containing the EO were incorporated into this formulation. The active ingredients of the cosmetic incorporated into the formulation are responsible for antioxidant (vitamin E) activity, hydration (hyaluronic acid), cell renewal (retinol), and cell longevity (Longevicell®) (Souza & Antunes Junior, 2020).

Table 1: Antiaging cream formulations containing EO and *C. adamantium* EO microcapsules.

Ingredients	
Essential oil from *C. adamantium*	1%
Microcapsules containing the EO of *C. adamantium**	3%
Retinol	2%
Vitamin E	2%
Hyaluronic acid	5%
Longevicell®	2%
Nonionic cream base	QS 100 g

*MC1 formulation microcapsules were used (G:GA 1:2; 40.3% EO). QS: quantum sufficit to 100 g.

Centrifugation test

A 5.0 g sample of cream was weighed and placed in a Falcon tube and centrifuged at increasing speeds of 980, 1800, and 3000 rpm for 15 min each time at room temperature (Agência Nacional de Vigilância Sanitária - ANVISA, 2004). If the sample was stable after centrifugation, further stability tests were conducted (ANVISA, 2004).

Accelerated stability tests

The accelerated stability tests were conducted as recommended in the Cosmetic Product Stability Guide (ANVISA, 2004). The cream was distributed in transparent glass flasks with a high-sealing plastic lid, keeping space in the container for gas exchange. The flasks containing the cream were placed at three temperatures: 25 ±2 °C (room temperature), 4 ±2 °C (refrigeration), and 45 ±2 °C for 90 days, with triplicates for each condition. Part of the cream prepared was separated for pH, spreadability, and centrifugation analyses. Cream samples were prepared every 30 days and stored in opaque, sealed polyethylene bottles for 90 days for comparison (reference cream).

Under the three temperature conditions, the cream was evaluated for appearance, color, odor, and pH after 1, 7, 30, 60, and 90 days. The appearance was analyzed as follows: 1: normal, no changes; 2: slightly separated, slightly precipitated, or slightly cloudy; and 3: separated, precipitated, or cloudy. The color assessment was visual and classified as follows: 1: normal, no change; 2: slightly modified; 3: modified; and 4: intensely modified. The odor intensity of the reference sample was evaluated using the same color criteria. The pH was determined by reading on a previously calibrated peagameter.

The spreadability of the cream under the three temperature conditions was evaluated after 1 and 90 days of cream preparation, following the method described by Borghetti and Knorst (2006). A sheet of millimeter paper was placed below a circular glass plate (diameter = 20 cm). A circular glass plate with a central hole of 1.0 cm diameter was placed on top. The hole was filled with the sample and leveled with a spatula. Then, the plate with the well was carefully removed. A circular glass plate (diameter = 15 cm; thickness = 3 mm) of known mass was placed on the sample. After 2 min, the average diameter covered by the spreadability of the cream was measured by taking the average of two opposite positions. The procedure was repeated, plates of known mass were added, and the diameter was measured until a constant value was obtained. The spreadability (Ei) was determined at 25 °C, calculated using Equation (6), and expressed as a function of the mass added.

$$Ei = \frac{d^2 . \pi}{4} \quad (6)$$

Here, Ei indicates the spreadability of the sample for mass i (mm^2) and d indicates the mean diameter (mm).

Microbiological control

Microbiological quality control was performed based on the specifications for nonsterile products of the Brazilian Pharmacopoeia (ANVISA, 2019). The mesophilic microorganisms, molds, and yeasts in the anti-aging cream were counted using the "spread-plate" surface seeding method. Coliform bacteria were detected using the Petrifilm technique. The analyses of *Pseudomonas* and *Staphylococcus aureus* were performed using the streak depletion method. Analyses were conducted in triplicate for each of the replicates (3) stored in a controlled environment (25 °C) for four periods (7, 30, 60, and 90 days).

First, 1.0 g of the sample was homogenized and diluted in 9 mL of commercial inactivator (Letheen, lot 81214, Newprov, Pinhais/PR) to neutralize any antimicrobial properties of the formulation products with preservative action. The mixture was homogenized for 5 min in a vortex shaker and incubated for 24 h at 30 °C, as recommended by the manufacturer. The procedure established by the Brazilian Pharmacopoeia was followed (ANVISA, 2019). Decimal dilutions were made with peptone saline (10^{-1}, 10^{-2}). To evaluate the content of total bacteria, 0.1 mL of the dilutions were seeded in a Petri dish containing nutrient agar. This volume was 1 mL for coliform analysis. The plates and Petrifilm were incubated at 32.5 ±2.5 °C. After 48 h, colony counts were performed, and the results were expressed as colony-forming units per gram (CFU/g). To assess molds and yeasts, the same procedure used for bacteria was used but with Sabouraud-dextrose agar growth medium. The plates were incubated for five days at 22.5 ±2.5 °C. Plates showing colony growth compatible with molds and yeasts were counted. The results were expressed as the mean value in colony-forming units/g cream (CFU/g).

A non-selective soy-casein enrichment medium was used to test for *Pseudomonas* and *S. aureus*. Briefly, 1 mL of the initial dilution was transferred to 9 mL of casein-soy broth and incubated for 48 h at 32.5 ±2.5 °C. For evaluating *Pseudomonas*

aeruginosa, the material was transferred from the non-selective enrichment broth to a Petri dish containing MacConkey medium and Luria Bertani Muller agar by streaking with a platinum loop. The dish was inverted and incubated for 48 h at 32.5 ±2.5 °C. In the case of colony growth, Gram identification and biochemical tests were performed. For *S. aureus*, the procedure was similar to that for *Pseudomonas*, but incubation was performed in a test tube containing mannitol agar for 72 h. The growth of yellow or white colonies surrounded by a yellow zone was investigated for the probable presence of *S. aureus*.

Statistical analysis

The data were statistically evaluated by conducting analysis of variance (ANOVA) of three replicates. Data are expressed as the mean and standard deviation. When there were differences between groups, Tukey's multiple comparison test was used. It was considered significant when $p < 0.05$, $p < 0.01$, and $p < 0.001$ using the Statistica Software version 8.0 and GraphPad Prism software version 8.0 (GraphPad Software, Inc., La Jolla, CA, USA).

Results and Discussion

Microencapsulated essential oil composition

The main constituents identified in the free EO and the MEO as the average of two independent analyses are shown in Figure 1. The EO yield was 0.37 ±0.08% (w/w). The main constituents were limonene, α-pinene, β-ocimene, and β-caryophyllene, with percentages similar to those of free EO and MEO. For some constituents, the percentage in the encapsulated form was significantly different. The changes in some constituents might be due to the loss caused by volatilization during the complex coacervation phase and the presence of EO on the outside of the microcapsule wall (Campelo et al., 2017). The reduction of some constituents in the encapsulated form may be related to water adsorption, the type of polymer chains, and the different affinities of each compound for the encapsulating matrix, which affects diffusion through it (Locali-Pereira et al., 2020). Analogous behavior, with high retention of EO constituents in the microcapsule, has been reported in other studies (Campelo et al., 2017; Khatibi et al., 2021; Locali-Pereira et al., 2020).

Compared to those of the free EO, the limonene retention rates of microcapsules MC1 and MC2 were 89.9% and 90.2%, respectively. Limonene is a monoterpene hydrocarbon compound with anti-inflammatory (Yu, Yan, & Sun, 2017), antifungal (Dias et al., 2020), antitumor (Magalhães et al., 2020), neuroprotective (Eddin et al., 2021), and antioxidant (Olatunya & Akintayo, 2017; Yang et al., 2010) activities, indicating that its bioactive properties are important for health. Limonene also promotes re-epithelialization capacity by increasing collagen synthesis and improving wound healing in rat skin (Keskin et al., 2017). Limonene is a major constituent in the EO of fruits belonging to the genus *Campomanesia* (Chang et al., 2011; Marin et al., 2008; Viscardi et al., 2017).

Another main compound identified was α-pinene, which showed 99.9% retention in MC1 and MC2. Studies have shown antimicrobial (Locali-Pereira et al., 2020), antioxidant, gastroprotective, cytoprotective, and anticonvulsant activities (Salehi et al., 2019) associated with α-pinene. The other two main constituents were β-ocimene and β-caryophyllene.

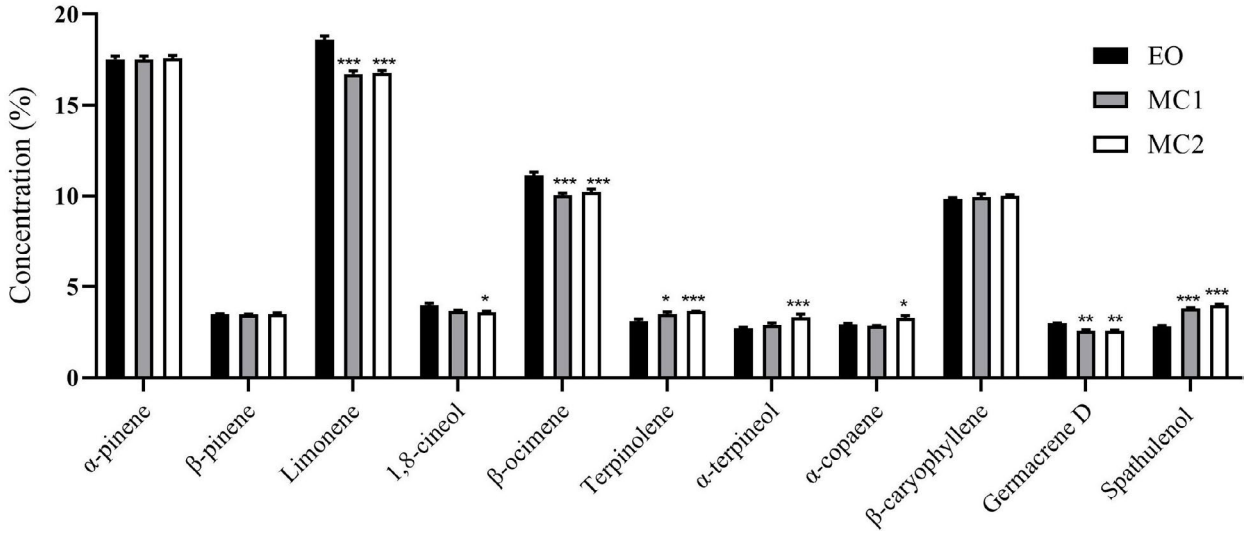

Figure 1: Main compounds of free and encapsulated EOs obtained from the fruit residues of *C. adamantium*. The values represent the mean and standard deviation of two independent analyses; *p < 0.05, **p < 0.01, and ***p < 0.001 (MC1 and MC2 versus EO). MC1: microcapsule 1 (G:GA 1:2, EO:40.3%); MC2: microcapsule 2 (G:GA 2:1, EO:40.3%); EO: essential oil.

The compound β-ocimene has antiprotozoal activity against *Leishmania amazonenses*, with a selectivity index 469.5 times greater than that of meglumine antimoniate and 42.8 times greater than that of amphotericin B, which are reference medicines (Sousa et al., 2023). The compound β-caryophyllene has antioxidant (Nogueira Sobrinho et al., 2020) and antiproliferative (Irrera et al., 2020) activities. A topical formulation of β-caryophyllene improved wound healing in rat skin via antioxidant, anti-inflammatory, and re-epithelialization mechanisms (Gushiken et al., 2022). To summarize, the MEO results revealed that most of the constituents were retained, suggesting that their biological properties were stable and preserved.

Microcapsule characterization

The microcapsules were initially characterized in terms of their retention rate, encapsulation efficiency, water solubility, hygroscopicity, and water activity parameters, as shown in Table 2. The retention rate in the microcapsule formulation with a G:GA ratio of 1:2 (MC1) was not significantly different ($p > 0.05$) from that in the microcapsule with a G:GA ratio of 2:1 (MC2). Similar results were reported for thyme EO microcapsules, with 89% retention (Gonçalves et al., 2018), and for calendula oil, with 79% retention (Badke et al., 2019). The same wall material and complex coacervation encapsulation process were used in both studies. The encapsulation efficiency in both formulations (MC1 and MC2) was greater than 98%, which suggested protection of the EO and low impregnation on the outside of the microcapsule wall. In other studies, when gelatin/gum arabic pairs were used as encapsulants, the encapsulation efficiency was 98.7% with ginger EO (Ferreira & Nicoletti, 2021) and 91.2% with oregano EO (Hernández-Nava et al., 2020), similar to the results of this study.

The water solubility results (Table 2) indicated that the microcapsules containing a greater proportion of GA (MC1) showed greater solubility than MC2, a result confirmed by the solubility of the control microcapsules. Gum arabic can be used as an encapsulating material; it is a carbohydrate with high water solubility and low viscosity (Alves et al., 2014). The presence of hydroxyl groups in the carbohydrate structure facilitates interactions with the aqueous phase, while the protein chain containing polar and nonpolar amino acids interacts with the hydrophilic and hydrophobic phases, affecting the solubility of these polymers (Silva et al., 2015). Less soluble microcapsules may favor the release of the nucleus when in contact with an aqueous medium (Tavares & Noreña, 2020).

Compared to the control microcapsules, the microcapsules with EO had lower hygroscopicity, with significantly ($p < 0.05$) lower hygroscopicity in the MC1 formulation, which was prepared with more GA (Table 2). Tavares and Noreña (2020) stated that a lower porosity suggests lower hygroscopicity and longer dissolution time for the microcapsule. Gum arabic forms a thick wall, which contributes to less moisture absorption (Alves et al., 2014), whereas gelatin has hygroscopic characteristics (Araújo et al., 2020). Ginger EO microcapsules obtained by complex coacervation with different combinations of wall materials that included gum arabic presented similar hygroscopicity values, between 8.7% and 9.6% (Tavares & Noreña, 2020). The G:GA combination, which resulted in lower hygroscopicity (CT1 and MC1), resulted in greater water activity, although all formulations presented water activities lower than 0.4, which is important for conserving and preserving bioactive compounds (Martins et al., 2021). Thus, the greatest difficulty in eliminating or absorbing water is related to the lower porosity of the wall, which contains more GA.

Regarding size, the MC1 and CT1 microcapsules had diameters of 24.35 ±1.5 and 24.12 ±2.5 μm, respectively, whereas MC2 and CT2 had values of 35.73 ±4.5 and 34.44 ±5.2 μm, respectively. Therefore, the size of the microcapsules was influenced by the proportion of G and GA, with the microcapsules containing the highest GA content having the smallest size ($p < 0.05$). With a similar proportion (G:GA = 1:1.8), microcapsules with ginger oil also had the smallest size among the formulations evaluated (Ferreira & Nicoletti, 2021). The difference in size between the microcapsules in this study confirmed the results of water solubility, as the smaller the size, the greater the surface area available for hydration.

Table 2: Physical parameters of microcapsules containing EOs extracted from the fruit residues of *C. adamantium* (MC1 and MC2) and without EOs (CT1 and CT2).

	MC1	MC2	CT1	CT2
Retention rate (%)	76.99 ± 2.5^{a}	78.53 ± 2.4^{a}	n.a.	n.a.
Encapsulation efficiency (%)	98.65 ± 0.3^{a}	99.16 ± 0.2^{a}	n.a.	n.a.
Solubility (%)	24.75 ± 1.8^{b}	17.86 ± 0.6^{c}	34.83 ± 1.3^{a}	23.72 ± 1.5^{b}
Hygroscopicity (%)	4.12 ± 0.9^{d}	8.95 ± 0.2^{c}	13.83 ± 0.5^{b}	16.66 ± 0.7^{a}
Water activity	0.399 ± 0.023^{a}	0.284 ± 0.025^{b}	0.388 ± 0.016^{a}	0.153 ± 0.017^{c}

The results are expressed as the means and standard deviations. Different letters on the same line indicate a statistically significant difference ($p < 0.05$, Tukey's test). CT1 and CT2: control microcapsules without essential oil; MC1 and MC2: microcapsules with essential oil; n.a.: not applicable.

Among the encapsulating carbohydrates used, gum arabic is an excellent wall material because of its emulsifying properties and ability to form a thicker wall (Alves et al., 2014). Gelatin is a biocompatible polymer that is soluble at body temperature and is biodegradable; these properties are of interest for pharmaceutical and food applications (Wang et al., 2016). Thus, gelatin and gum arabic can serve as efficient wall materials for encapsulating volatile substances, as demonstrated, for example, in the encapsulation of EO from *Lippia turbinata*, which allows its controlled release and maintenance of its antifungal activity (Girardi et al., 2017), and in the use of food films with microencapsulated ginger EO, which maintain its antibacterial and antioxidant capacity for a long period (Wang et al., 2021). In this study, the proportions of gum arabic and gelatin, as well as the added concentration of EO, effectively retained the guavira EO in the microcapsules.

Microcapsule morphology

The morphological structure of wet and freeze-dried microcapsules is shown in Figure 2. The wet microcapsules (Figure 2A) were spherical with different sizes and tended to agglomerate. They exhibited complete wall formation, indicating effective protection of the encapsulated material, which corroborated the encapsulation efficiency and retention rate values. Similar morphology was observed in *Zataria multiflora* EO microencapsulated with the same wall materials using a complex coacervation method (Khatibi et al., 2021). SEM images at 800x magnification (Figure 2B) revealed a porous surface due to water sublimation during freeze-drying (Rutz et al., 2017). At 3000x magnification (Figure 2C), smoother surfaces with few pores were observed; the MC2 formulation showed greater porosity, which indicated a less compact microstructure. This was attributed to the higher gelatin content in MC2, making it more hygroscopic and resulting in a porous morphology. These characteristics matched expectations for EO particles obtained through complex coacervation and freeze-drying (Araújo et al., 2020; Khatibi et al., 2021; Sendi et al., 2023).

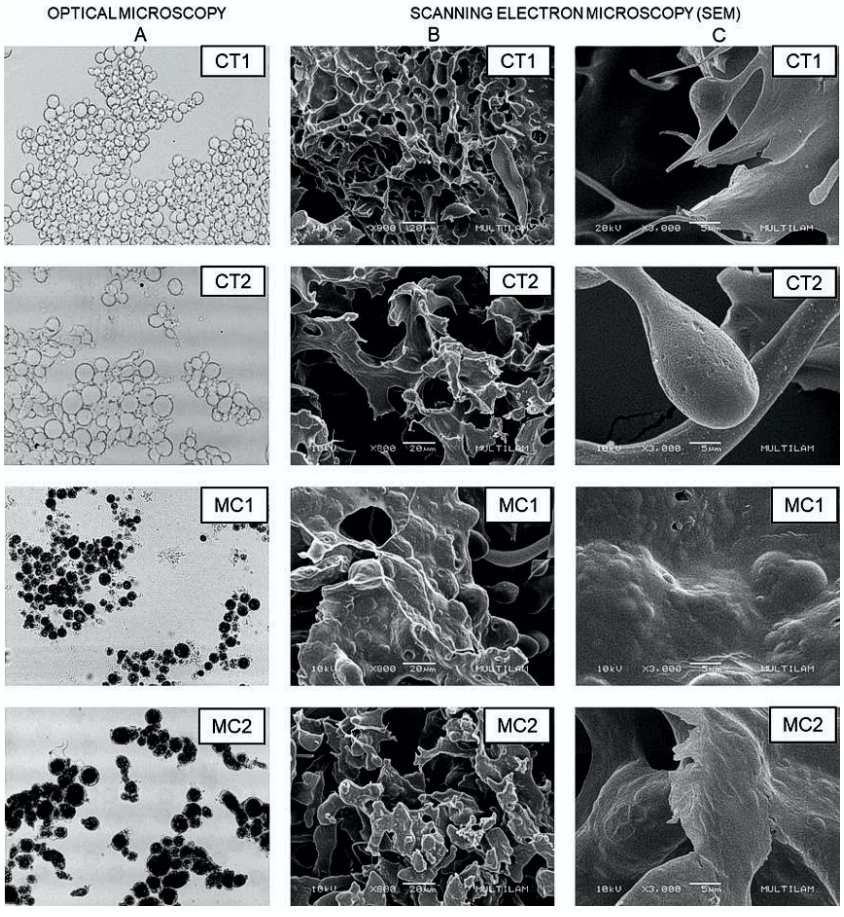

Figure 2: Optical microscopy (column A: ×100 magnification) and scanning electron microscopy (columns B: ×800 magnification and C: ×3000 magnification) images of microcapsules without EO (CT1 and CT2) and with *C. adamantium* EO (MC1 and MC2).

Fourier transform infrared spectroscopy (FTIR)

The Fourier transform infrared spectroscopy spectra of the EO, microcapsules with EO (MC1 and MC2), control microcapsules (CT1 and CT2), gelatin, and gum arabic are shown in Figure 3.

The EO spectrum displayed absorption bands at 2925, 1644, 1450, 1375, and 887 cm^{-1}. The spectrum exhibited characteristic peaks for limonene (887 cm^{-1} and 1644 cm^{-1}) (Araújo et al., 2020; Lima et al., 2021), which is a major component of the OE. The bands at 2925, 1450, and 1375 cm^{-1} suggested the presence of aliphatic chains (stretching in CH, CH_2, and CH_3, respectively) (Lima et al., 2021). The presence of specific bands in the spectra of microcapsules MC1 and MC2, such as 887, 1450, and 1644 cm^{-1}, indicated the encapsulation of the EO. The similarity in peaks among MC1, MC2, and their encapsulating matrices suggested that encapsulation was successful. The gelatin spectrum presented characteristic peaks at 3340, 1653, and 1456 cm^{-1}, whereas the gum Arabic spectrum presented peaks at 2930, 1420, 1609, and 3421 cm^{-1}. Interactions between the EO and wall materials were observed in MC1 and MC2, as some peaks present in the control microcapsules were absent in the encapsulated samples.

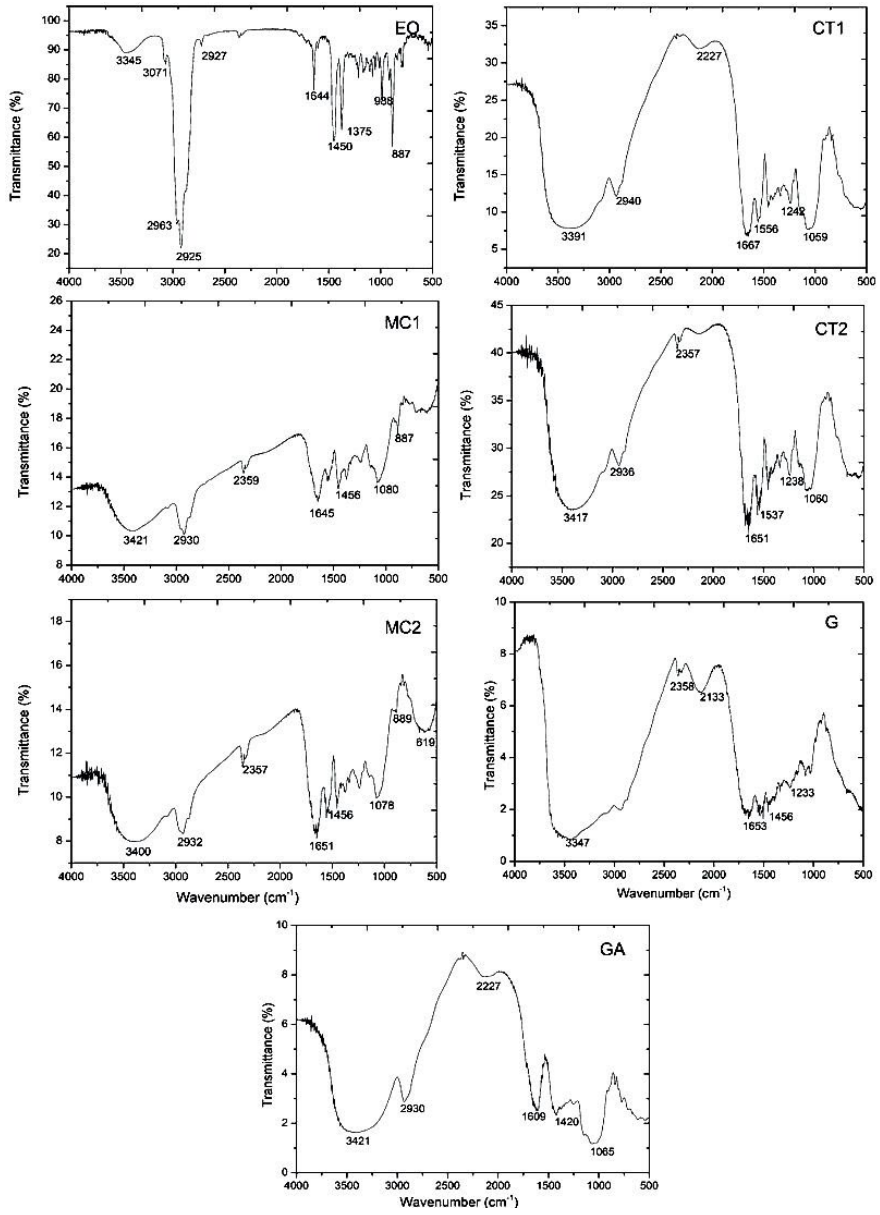

Figure 3: FTIR spectra of free EO, microcapsule G:GA 1:2 (MC1), microcapsule G:GA 2:1 (MC2), control microcapsule G:GA 1:2 (CT1), control microcapsule G:GA 2:1 (CT2), gelatin (G), and gum arabic (GA).

Thermogravimetric analysis (TGA)

The TGA and thermogravimetric derivative (DTG) curves for the free EO, microcapsules (MC1, MC2, CT1, and CT2), gelatin, and gum arabic are shown in Figure 4, illustrating the mass loss (%) of the samples with an increase in temperature. The analysis assessed the thermal stability of the microcapsules compared to that of the free EO.

The initial mass loss of the wall materials and microcapsules occurred due to the water content. The TG and DTG curves of the EO showed progressive mass loss as the temperature increased, peaking at about 90 °C because of its volatile nature, with complete mass loss at 250 °C. The control microcapsules without EO (CT1 and CT2) presented a mass loss peak starting at 250 °C, indicating wall material degradation at this temperature. For the MC1 and MC2 microcapsules, three regions of mass loss were observed: 1) water loss (50 °C), 2) EO degradation (200 °C), and 3) wall material degradation (250 °C). This behavior suggested enhanced protection of the EO in encapsulation systems using gelatin and gum arabic, which matched the findings of previous studies (Araújo et al., 2020; Wang et al., 2016). MC1, with a higher gum arabic content than gelatin, was expected to offer greater thermal protection because of its thicker layer formation, but this was not confirmed in the TGA. The G:GA ratios of 1:2 and 2:1 resulted in similar EO protection up to about 200 °C, enhancing the thermal stability of the EO.

Antioxidant capacity

The results of the antioxidant capacity of free EO, MEO, and synthetic standards of ascorbic acid and BHT are shown in Table 3, as assessed using the iron ion reduction power (FRAP method) and the $ABTS^{\bullet+}$ free radical scavenging methods.

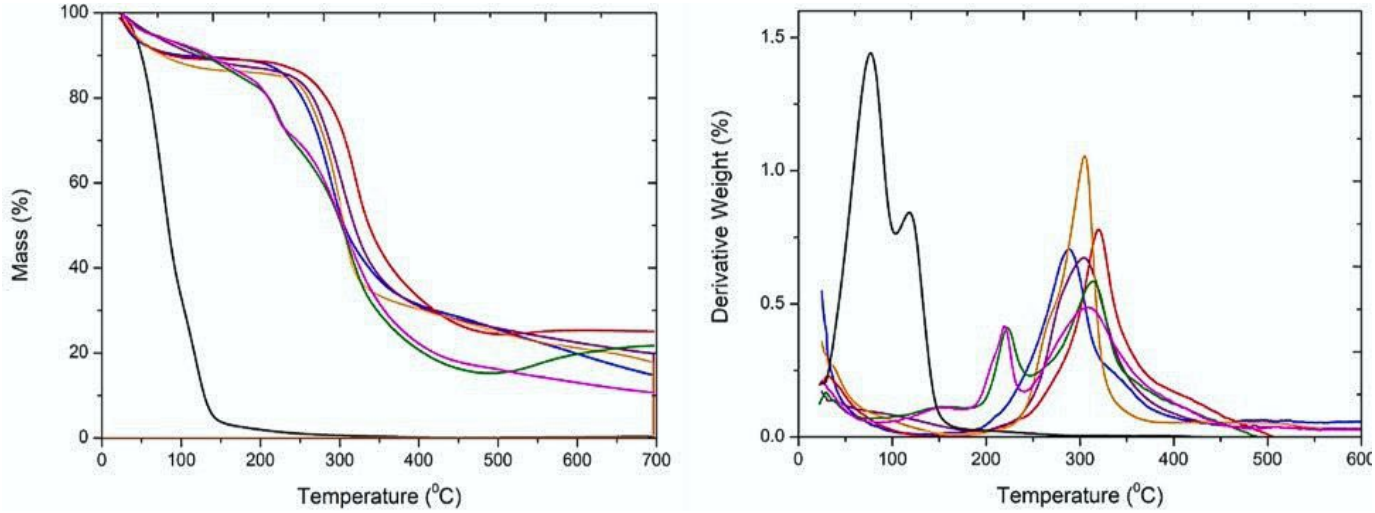

Figure 4: Thermogravimetric and thermogravimetric derivative curves of the free EOs, wall materials, and microcapsules. Color representation of the curves: black (free EO), green (MC1), pink (MC2), blue (CT1), red (CT2), purple (gelatin), and orange (gum arabic).

Table 3: Antioxidant capacity of free and microencapsulated EOs from the fruit residues of *C. adamantium* and the synthetic antioxidant standards ascorbic acid and BHT

	FRAP (mmol $FeSO_4$/g)	$ABTS^{\bullet+}$ Inhibition (%I)[1]	$ABTS^{\bullet+}$ (IC_{50}) (µg/mL)
EO	0.183 ± 0.03[b]	59.37 ± 2.9[a]	946.06 ± 89.3[b]
MEO (MC1)	0.181 ± 0.08[b]	55.27 ± 1.7[a]	955.99 ± 33.7[b]
MEO (MC2)	0.173 ± 0.01[b]	44.01 ± 1.9[b]	1192.10 ± 46.6[a]
AA	15.353 ± 1.51[a]	n.a.	2.55 ± 0.2[c]
BHT	12.085 ± 0.56[a]	n.a.	5.76 ± 0.4[c]

The values represent the means and standard deviations of the means of three independent assays. Different letters in the columns represent statistically significant differences ($p < 0.05$). $ABTS^{\bullet+}$ radical inhibition (% I) by MEO at a concentration of 1000 µg/mL. AA: ascorbic acid; $ABTS^{\bullet+}$ = $ABTS^{\bullet+}$ free radical; BHT = butylhydroxytoluene; EO: essential oil; FRAP = Ferric Reducing Antioxidant Power; MEO = microencapsulated essential oil. n.a: not applicable at this concentration.

Initially, the antioxidant capacity of free EO was measured, with a Fe^{3+} ion reduction capacity of 0.183 mmol $FeSO_4$/g. The amount of reduced Fe^{3+} is directly related to the antioxidant capacity of a sample. A study by Silva et al. (2018) reported a $FeSO_4$/g value of 0.193 mmol for the EO of *Myrcia sylvatica* (Myrtaceae) leaves. A relatively high antioxidant capacity of 3.83 mmol $FeSO_4$/g was recorded in the EO of *Campomanesia xanthocarpa* leaves (Sugauara et al., 2020). In the $ABTS^{\bullet+}$ radical inhibition method (%I), free EO inhibited the radical by 59.37% at a concentration of 1000 µg/mL, corresponding to an IC_{50} of 946.06 µg/mL. In the same genus, Lorençoni et al. (2020) reported no antioxidant activity in the EO of *Campomanesia phaea* leaves using the ABTS method. The EO from *Psidium guineense* (Myrtaceae) inhibited 50% of the $ABTS^{\bullet+}$ radical at a concentration of 780.13 µg/mL (Nascimento et al., 2018); this finding was similar to the results of our study. The antioxidant capacity of a sample is greater when it shows a higher percentage of inhibition (%I) or a lower sample concentration required to inhibit 50% of the free radical activity.

To compare the antioxidant capacity of free EO and MEO in the two microcapsule formulations and assess their applicability, the antioxidant capacity of MEO was calculated (Table 3), considering the insertion content (40.3%) and final retention of OE in the microcapsules (MC1: 76.99%; MC2: 78.53%). The results obtained from the FRAP method revealed no significant difference between MC1 and MC2 and free EO. In the ABTS method assessment, MEO from MC1 exhibited significantly different $ABTS^{\bullet+}$ radical inhibition rates than MC2 but did not significantly differ from the percentage of free EO that was inhibited. This suggested that microencapsulation with a gelatin/gum arabic ratio of 1:2 provided better protection than the formulation with a higher gelatin content (2:1). The IC_{50} values confirmed these results, with MC1 being 1.25 times more effective in neutralizing the $ABTS^{\bullet+}$ radical than MC2. MC1 retained 99% and 93% of the antioxidant capacity (FRAP and ABTS methods, respectively) of the free EO, whereas MC2 retained 94% and 74% (FRAP and ABTS, respectively). These results concerning the antioxidant capacity supported the advantages of the MC1 formulation reported in this study. The values of the synthetic standards (ascorbic acid and BHT) were similar to those reported in other studies (Araújo et al., 2023). The results suggested that microencapsulation effectively preserved the EO and its antioxidant activity under the tested conditions, indicating prolonged antioxidant capacity when applied in a product compared to free EO, as reported by Wang et al. (2021) for the encapsulation of ginger EO. Alves et al. (2014) reported 70% retention of β-caryophyllene, the main constituent of *Pterodon emarginatus* EO, in microcapsules after 45 days of storage at 25 °C.

The antioxidant capacity should be considered along with the chemical composition of the EO. The major constituents of the EO in this study (limonene, α-pinene, β-ocimene, and β-caryophyllene) were hydrocarbon-type monoterpenes and sesquiterpenes. The synergistic antioxidant effects of these constituents in free EO and MEO should be considered, as β-caryophyllene exhibits antioxidant activity *in vivo* (Gushiken et al., 2022) and *in vitro* (Nogueira Sobrinho et al., 2020). Additionally, limonene and α-pinene, which were predominant in the EO composition, exhibit antioxidant activity equivalent to that of Trolox and α-tocopherol in *Citrus* lemon EO, which has limonene (67%) and α-pinene (11%) as primary constituents (Himed et al., 2019). The authors reported that microencapsulated oil maintained the same antioxidant activity.

Incorporation of microcapsules and EOs in cosmetic formulations

The EO of *C. adamantium* has anti-inflammatory (Viscardi et al., 2017) and antiproliferative (Alves et al., 2020) properties, besides being highly aromatic. Moreover, its major constituents, limonene, and β-caryophyllene, have skin re-epithelialization properties, promote collagen synthesis, and enhance wound healing (Gushiken et al., 2022; Keskin et al., 2017). Considering these beneficial properties, it can be incorporated into cosmetic formulations, which can be further enhanced using encapsulated active ingredients. Some studies have also reported the application of EOs in the encapsulated form in cosmetic products: calendula and geranium EOs encapsulated in liposomes impart photoprotective and anti-aging effects to anti-aging facial creams (Lohani et al., 2021); nanoemulsified EOs of *Lippia origanoides* exhibited significant antifungal activity against *Candida* spp. in a prototype pharmaceutical cream (Benitez-Llano et al., 2023); and various applications of microencapsulated EOs in cosmetic products were presented by Carvalho, Estevinho and Santos (2016). In this study, incorporating microcapsules and EO into an anti-aging facial cosmetic cream helped maintain a stable emulsion with adequate organoleptic characteristics. The incorporation of free EO imparted the pleasant aroma of *C. adamantium* EO in the formulated cream.

Centrifugation test

A centrifugation test was conducted to predict potential instabilities in the cream, such as phase separation, creaming, flocculation, or coalescence (Rocco et al., 2023). The anti-aging cream remained a stable emulsion without phase separation when centrifuged at up to 3000 rpm. This result indicated that the product can maintain its initial characteristics during use, without separation of the oily phase or instability in the emulsion.

Accelerated stability evaluation

After 24 h of preparing the cream (day 1), the cosmetic exhibited a consistency classified as "1: normal, no change", appearing "uniform, homogeneous, and of medium consistency". The color and odor were also classified as "1: normal, no

change", with the odor described as "characteristic of guavira EO and pleasant". The odor may be attributed to the presence of limonene, the main constituent of *C. adamantium* EO. Limonene is a natural aromatic compound used in the cosmetic industry to replace artificial fragrances that may cause skin complications and health issues (Sharmeen et al., 2021).

The cream did not show visual changes in any organoleptic parameters during 90 days of storage at 25 °C, maintaining a classification of "1: normal, no change".

At 4 °C, a change in appearance was recorded after seven days, resulting in a firmer consistency than that on the first day, as expected because the formulation's components became more "solid" at lower temperatures. Although the consistency changed, there was no apparent precipitation or crystallization in the cream, suggesting stability even in cold climates. The color and odor remained unchanged at this temperature for up to 90 days.

At 45 °C, the cream exhibited phase separation, with the consistency classified as "2: slightly separated" at 30 days and "2: slightly precipitated" at 60 and 90 days. The color slightly darkened over time and was classified as "2: slightly modified", and the odor was also slightly altered. High temperatures accelerate physical-chemical and chemical reactions, leading to changes in the biological action, viscosity, appearance, color, and odor of the product. The cream was unstable at high temperatures, indicating the need for storage in cool conditions at 30 °C.

The pH of the cream remained between 4.4 and 3.97 under all storage conditions (4 °C, 25 °C, and 45 °C) for 90 days. The pH of the cream matched the slightly acidic pH range of the skin's surface (4.0–5.5) (Pavlačková et al., 2020), ensuring compatibility. Similar pH values were reported in a cosmetic cream formulated with propolis, which remained stable for 90 days (Bugnotto et al., 2006).

The spreadability results of the cream are shown in Figure 5. This parameter is crucial in pharmaceutical formulae designed for topical application, as it indicates how well the product will spread on the skin. The cream, which was prepared on the first day, had a spreadability of 4616.7 mm^2, maintaining this value even after adding 1.03 kg of mass. The cream stored at 4 °C and 25 °C retained 81.5% and 76.0% of its spreadability after 90 days, respectively, with the same mass addition, indicating suitable spreadability under these temperature conditions. However, the cream stored at 45 °C exhibited only 36.9% initial spreadability when 1.03 kg was added. This decrease might be attributed to moisture loss at higher temperatures, leading to an increase in the consistency of the cream. Insufficient spreadability can result in uneven application, affecting the dosage, absorption, and efficacy of active ingredients. Spreadability should increase with an increase in mass addition (Rocco et al., 2023). The results for the cream stored at 4 °C and 25 °C were satisfactory, matching the organoleptic properties of the cream and meeting the desired parameters for a cosmetic product, ensuring good spreadability, absorption, nongreasy appearance, pleasant odor, and overall appearance.

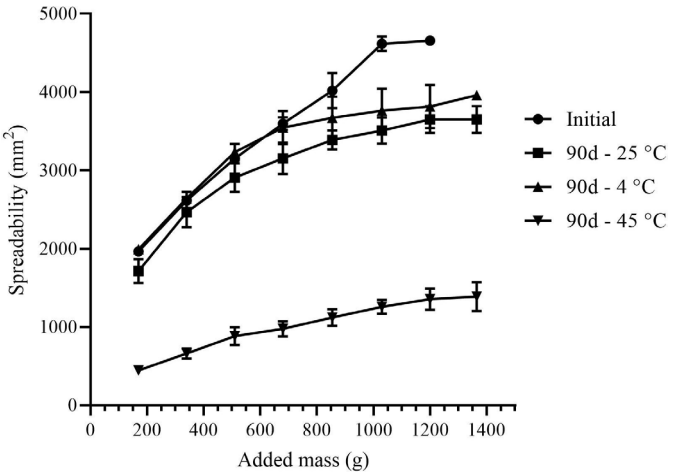

Figure 5: Spreadability of the cream formulation on day 1 and after 90 days of storage at different temperatures.

Microbiological control of anti-aging cream

The microbiological control results of the anti-aging cream assessing aerobic mesophilic bacteria, molds/yeasts, coliforms, *P. aeruginosa,* and *S. aureus* growth are presented in Table 4.

The results indicated that the growth of aerobic mesophilic bacteria, molds, and yeasts was within acceptable limits, whereas coliforms, *P. aeruginosa,* and *S. aureus* were absent, meeting the requirements of Resolution No. 752/2022 (ANVISA, 2022). According to ANVISA guidelines, the acceptable limits for facial cream are up to 5×10^3 CFU/g for aerobic mesophilic microorganisms, molds, and yeasts, with coliforms, *P. aeruginosa*, and *S. aureus* absent. Non-sterile topical products must undergo microbial contamination control (ANVISA, 2019), indicating that manipulation practices are appropriate.

The search for new sources of EOs has great economic importance because of their high market value. In this context, the industry has shown an increase in interest in the use of waste, matching the principles of circular economy. This approach includes, among other aspects, the transformation of waste into new products, the addition of value, and the mitigation of the environmental effects associated with inadequate disposal (Gonçalves & Maximo, 2023). In this study, the residue generated by the industrial pulping of *C. adamantium* fruits was used in a promising way, highlighting the application of its EO.

The proposal to develop an anti-aging product with EOs aligns with the significant growth in the Brazilian cosmetic market (ABIHPEC, 2024). The incorporation of active ingredients, such as hyaluronic acid and retinol, enhances the effectiveness of such products. Claims serve as attention-grabbing advertisements that emphasize the benefits of the product, adding value to it. Products containing EOs or botanical extracts exhibit biological activity, and when encapsulated, they promote the retention of bioactive substances, promoting the creation of innovative products.

Table 4: Microbiological control of the anti-aging cream formulated with EO and *C. adamantium* EO microcapsules.

Period (days)	Aerobic Mesophilic Bacteria (CFU/g)	Molds/ yeasts (CFU/g)	Coliforms (CFU/g)	*P. aeruginosa* (CFU/g)	*S. aureus* (CFU/g)	Concordance (*)
7	0.2×10^3	0.2×10^3	0	0	0	Yes
30	0.8×10^3	0.2×10^3	0	0	0	Yes
60	1.1×10^3	0.2×10^3	0	0	0	Yes
90	1.4×10^3	0.7×10^3	0	0	0	Yes

The results are presented as the means (n = 9). (*) ANVISA (2019, 2022).

Conclusions

Microcapsules containing EO from the fruit residue of *C. adamantium* exhibited excellent retention of constituents and antioxidant properties comparable to those of free EO. Increasing the proportion of gum arabic in the formulation resulted in microcapsules with lower hygroscopicity and thermal stability. When incorporated into a facial cream, these microcapsules maintained stable emulsion, spreadability, and organoleptic characteristics for 90 days. This study highlighted the use of MEO from guavira waste in cosmetic formulations and suggested further research on its applications.

Author contribution

Conceptual idea: Bin, M.C., Argandoña, E.J.S.; Methodology design: Bin, M.C., Argandoña, E.J.S.; Data collection: Bin, M.C., Assis, L.S., Gonçalves, D.A., Alonso, C.P., De Melo, A.M.M.F., Cardoso, C.A.L.; Data analysis and interpretation: Bin, M.C., Valladão, D.M.S., Argandoña, E.J.S.; Writing and editing: Bin, M.C., Argandoña, E.J.S., Valladão, D.M.S.

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