Valorization of ucuúba (Virola surinamensis) press cake using green extraction methods

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Research Article

Keywords: bioactive compounds, pressurized liquid extraction, microwave-assisted extraction, antioxidant potential

Posted Date: September 29th, 2025

DOI: https://doi.org/10.21203/rs.3.rs-7508240/v1

Additional Declarations: No competing interests reported.

Abstract

Ucuúba seeds have a high fat content, which is highly valued by the cosmetics industry due to their richness in saturated fatty acids (SFAs). The extraction of fat from the seeds results in a press cake co-product, rich in bioactive components such as antioxidants. The aim of this work was to characterize the press cake (C1) resulting from the hydraulic pressing of shelled ucuúba seeds, and to evaluate the influence of residual fat on the recovery of compounds with antioxidant potential, by defatting pretreatment to obtain the degreased cake (C2). Following the biorefinery concept, C1 and C2 were subjected to extractions using ethanol and hydroethanolic solutions through three different methods: Soxhlet (SOX), Microwave-Assisted Extraction (MAE), and Pressurized Liquid Extraction (PLE). The extracts were analyzed for total phenolic content (TPC), total flavonoid content (TFC), total carotenoid content (TCC), and antioxidant potential by ABTS and DPPH assays. The most abundant macronutrients from C1 were total carbohydrates content (43.32 g 100 g^{-1}), lipids (33.24 g 100 g^{-1}), and protein (15.09 g 100 g^{-1}). The highest extraction yields were obtained from C1 by MAE and PLE with 100% ethanol. Extract by MAE with ethanol 70% from C2 showed the highest values of TPC (126.78 mg GAE g^{-1}), TCC (18.74 mg BCE g^{-1}), and antioxidant activity by DPPH assay (791.71 µmol TE g^{-1}), while SOX with ethanol 50% provided the highest TFC (13.10 mg QE g^{-1}), and with 100% ethanol showed the highest ABTS (1286.69 µmol TE g^{-1}). The main phenolic compounds found in samples extracted with ethanol 70% were ferulic acid (12788.94–14885.64 µg g^{-1}), vanillic acid (1388.36–3021.41 µg g^{-1}), and vanillin (809.75–1778.66 µg g^{-1}). Therefore, extracts from the degreased cake C2 showed higher concentrations of bioactive compounds with antioxidant potential, evidencing the positive effect of the defatting pretreatment. Additionally, the optimization of the alternative methods such as MAE and PLE may contribute to the valorization of this underutilized biomass.

1. Introduction

The increasing demand for natural and sustainable products and processes has driven research focused on the valorization of agro-industrial byproducts [1], such as oilseed cakes resulted from the oil or fat recovery from the seeds, which are potential sources of high-value bioactive compounds [2, 3]. Although this byproduct is often underutilized or discarded, it can be used in the development of food, cosmetic, and pharmaceutical products, contributing to sustainability and reducing waste in the production chain [4].

The Amazon bioeconomy converges between biodiversity, innovation, and sustainable development, while pursuit a comprehensive use of the Amazonian abundant natural resources to generate economic opportunities and foster environmental conservation [5]. Besides, the unique flora of the region arouse the creation of exclusive phytotherapeutics and phytocosmetics, which are emerging in innovative business models [6].

Ucuúba (*Virola surinamensis*), an Amazonian native specie, is primarily recognized for the seed's fat, which is commonly used in the cosmetics industry [7]. However, this specie is currently endangered due

to overexploitation of its wood, underscoring the urgent need for sustainable approaches centered on non-timber products to relieve the pressure on the ucuúba [8], for instance, assessing the richessness in biocomponents associated to the Virola genus [9]. Besides, the underutilized co-product from the pressing of ucuúba seeds, the residual cake (press cake), presents bioactive compounds such as phenolic compounds, flavonoids, and other phytochemicals, which present antioxidant properties [10, 11] and could be valued by the recovery of these bioactive or functional ingredients, increasing the processing chain value, while promoting more sustainable resource management practices.

To maximize the potential of ucuúba press cake, advancements in extraction technologies are fundamental. Methods such as microwave-assisted extraction (MAE) and pressurized liquid extraction (PLE) have proven effectiviness in recovering bioactive compounds from various plant sources, and should be optimized for each application in order to increase the process yield, while maintaining the extract quality [12–15]. Moreover, combining advanced and environmentally friendly techniques compared to traditional methods, the biorefinery approach enhances the use of the raw materials, producing sequentially more than one high-quality products and/or byproducts [16, 17].

Therefore, the biorefinery of the ucuúba press cake promotes a more efficient use of the natural resources, while fosters sustainable practices, and has not been previously reported in the literature. This study explores the application of alternative extraction techniques (PLE and MAE), in a biorefinery strategy to recover different valuable fractions from the ucuúba press cake. The extractions efficiency was assessed by evaluating the influence of the press cake defatting, previous to the extractions (PLE and MAE), followed by the characterization of the recovered fractions, in an innovative approach to increase the productivity of the ucuúba processing chain.

2. Material and methods

2.1 Samples

Ucuúba seeds (5,000 g) were gathered in Breves, Ilha do Marajó, Brazil (1°40'57" S, 50°28'51" W) and generously supplied by the company 100% Amazonia (Belém, PA, Brazil). The seeds were manually shelled, then heated for 120 minutes at 60°C in a forced-air circulation oven (Nova Ética, São Paulo, Brazil), and subsequently subjected to two extraction cycles using a Tecnal® TE-098 hydraulic press (Piracicaba, Brazil) for the seed fat recovery. The resulting press cake was used for all subsequent analyses. Initially, the press cake was submitted to particle size reduction using a knife mill (De Leo, Porto Alegre/RS, Brazil). The press cake resulting from the pressing of shelled seeds (C1) was used for the sequential extractions. Additionally, to evaluate the influence on biocomponents recovery of the residual fat content of the cake, a defatting treatment was carried out previous to the extractions. The cake samples were pretreated by Soxhlet with hexane as the solvent's boiling temperature, following the AOAC [18] and described by method 920.39C, and comprising approximately 10 cycles per hour and constant drip rate of 8 drops per second. The resulting sample is the defatted press cake (C2), with was then submitted to the extractions, analogous to C1.

Three ethanol concentrations (50%, 70%, and 100%) were evaluated as solvents for the untreated cake (C1), using PLE and MAE processes (sections 2.3.2 and 2.3.3, respectively). The Soxhlet method (SOX) also was used for comparative purpose, using solvent concentration based on the recovery of phenolic compounds (TPC) (Section 3.2.3 - Table 2), and represented by ethanol 70%. For the defatted press cake (C2), a SOX screening procedure with different solvents was conducted, and the selection criteria for solvent was the resulting TPC value, as conducted for C1. Again, ethanol 70% was selected, and then used as solvent for the alternative extraction methods, MAE and PLE, conducted at constant 50°C. Figure 1 illustrates the experimental assays conducted to obtain the press cakes and their respective extracts.

2.2 Proximal composition of pressing cake (C1)

Proximal composition analyses were performed according to the Association of Official Analytical Chemists [18]. Moisture (935.40) was determined in a forced-air oven at 105°C until constant weight. Ash (932.03) was determined in a muffle furnace at 550°C. Protein (920.87) was determined by the micro-Kjeldahl method (N × 5.30). Lipids (920.39) were determined by Soxhlet extraction using hexane at its boiling point (~ 69°C). Total carbohydrates were obtained by difference, and the energy value was calculated by summing the crude protein and carbohydrate contents multiplied by 4 and the total lipid content multiplied by 9. Minor carbohydrates (monosaccharides and disaccharides) were quantified by the phenol–sulfuric acid method [19], as adapted by Masuko et al. [20], and expressed as "carbohydrates". Analyses were performed in duplicate, and results were expressed in g 100 g^{-1} on a wet basis (mean ± standard deviation).

2.3 Extractions

The cake obtained directly from the hydraulic pressing process (C1) and the defatted cake obtained after Soxhlet extraction with hexane (C2) were submitted to further extractions to using ethanol or hydroethanolic solutions as solvents by three methods: SOX, MAE, and PLE, as presented in Fig. 1, and described in sections 2.3.1, 2.3.2, and 2.3.3.

2.3.1 Soxhlet extraction (SOX)

Soxhlet extraction was performed following the procedure by the Association of Official Analytical Chemists [18] (method 920.39C) with modifications to optimize the quality and efficiency of the process. The samples used consisted of 5 g of dried and ground seed cake, wrapped in filter paper cartridges. Various ethanol concentrations were tested: 100 % ehanol, and ethanol/water mixtures (50/50, 70/30 v/v). For each extraction, 200 mL of the selected solvent was added to a distillation flask. The extraction was carried out at the boiling temperature of each solvent. The total extraction process lasted 6 hours with approximately 10 cycles per hour, maintaining a constant drip rate of 8 drops per second. After extraction, the solvent-solute mixture was concentrated using a rotary evaporator, as described in Section 2.4.

2.3.2 Pressurized Liquid Extraction (PLE)

The PLE unit was employed to carry out the high-pressure extractions following the methodology by Andrade, Trivellin and Ferreira [21] with modifications. The optimal extraction time was determined by an extraction kinetics study, conducted as follows: the extraction bed was loaded with 3 g of C1 or C2, mixed with 33 g of glass beads, and placed inside the extractor. The solvent was pumped at a volumetric flow rate of 4 mL min^{-1} using an HPLC pump (Waters, model 515, Milford, Massachusetts, USA, with a maximum capacity of 10 mL min^{-1}) until the system reached the required operating pressure. The solvent was then heated to 50°C through a heating bath (Microquímica, Brazil). Once the desired operating conditions (100 bar, 50°C, 4 mL min^{-1}) were established, the outlet valve was opened to collect samples in amber test tubes. These pressure and temperature conditions were selected based on previous studies on agro-industrial residues, which reported promising results [22–24]. Extract samples were collected every 2 minutes during the first 10 minutes, then every 5 minutes up to 30 minutes, and finally every 10 minutes until reaching a total extraction time of 60 minutes. This procedure enabled the generation of the extraction kinetics curve for ethanol 70%, selected as the intermediate concentration among the three solvent concentrations evaluated. After evaporating the solvent using a rotary evaporator (section 2.4), the accumulated mass of each fraction time was calculated. A piecewise linear function was then fitted to the experimental data using Origin Pro 2021 software (OriginLab, Northampton, USA). The piecewise model was used, adjusting three linear segments corresponding to different steps of the extraction process: constant extraction rate (CER), falling extraction rate (FER), and diffusion-controlled (DC) periods, according to described by Weinhold et al. [25]. Breakpoints were defined to distinguish between these phases, with the corresponding mathematical equations outlined by Equations 1, 2, and 3.

For $t \leq t_{CER}$ (CER step);

$$y = a_1 + k_1 t$$

1

For $t_{CER} \leq t \leq t_{FER}$ (FER step);

$$y = a_1 + k_1 t_{CER} + t_2 \left(t - t_{CER}\right)$$

2

For $t \geq t_{FER}$ (DC step).

$$y = a_1 + k_1 t_{CER} + k_2 \left(t_{FER} - t_{CER}\right) + k_3 \left(t - t_{FER}\right)$$

3

where: y represent the accumulated mass and t the time, a_1 is the linear coefficient in the phase CER; k_1, k_2, k_3 are the angular coefficients of the lines in the CER, FER and DC phases, respectively; t_{CER} is the time of the end of CER period, with the beginning of FER period, which ends at time t_{FER} and starts

the DC period. After applying the described model, the t_{FER} value was obtained, and extractions for each ethanol concentration were carried out according to the detailed procedure until t_{FER} value was reached. All extractions were performed in duplicate, and the results were expressed as mean ± standard deviation.

2.3.3 Microwave assisted extraction (MAE)

Microwave-assisted extraction (MAE) was carried out following the methodology of Mazzutti et al. [26], with modifications. The extraction was performed using the equipment (Monowave™ 300, Anton Paar, Graz, Austria) with 850 W of power, and a maximum pressure of 2 MPa. For the procedure, 1 g of C1 or C2 and 20 mL of ethanol 100 %, r ethanol/water solutions (50%, 70% v/v) were placed in borosilicate glass vials and inserted into the microwave reactor at 50°C. The extraction process was performed in three stages: 1) heating to the desired temperature (50°C), 2) reaction for calculated time at 1000 rpm, and 3) cooling with compressed air to 35°C. The extracts were filtered using qualitative filter paper (Whatman®, Grade 42) to remove solids. The filtered solutions were stored in a conventional freezer at -18°C until analysis. Solvent removal was conducted as described in section 2.4. Assays were performed in duplicate for C1 and C2 samples, with yield results expressed as mean ± standard deviation.

The processing time for the MAE process was determined based on extraction kinetics performed on ucuúba seed cakes (C1 or C2) using 1g of sample and ethanol 70% (v/v) (intermediate concentration). For both cakes, collection times were 1, 3, 4, 5, 7, 10. 13, 15, 20, 30, and 45 minutes. The resulting extraction experimental curves were fitted using the Gompertz model with Origin Pro 2021 software, applying Eq. 4.

$$y = ae^{-e(-k(t-t_c))}$$

4

Where: y represents the yield of the extraction process as a function of time (%); ae represents the theoretical maximum yield expected at the end of the process (%); t_c is the time when the inflection point occurs (min); k is the growth rate constant.

2.4 Removal of solvents from extracts

The extracts obtained from the extraction processes (sections 2.3.1, 2.3.2 and 2.3.3) were subjected to rotary evaporation to remove the more volatile solvent (ethanol) using a rotary evaporator (Fisatom 802, São Paulo, Brazil) at approximately 65°C until complete evaporation. For the hydroethanolic solutions, the residual water was removed using a lyophilizer (Liotop L 101, Liotop, São Carlos, Brazil). The dried extracts were then inerted with nitrogen for 1 minute and stored in amber vials at -18°C.

2.5 Extraction yield

The extraction yield (X_0) was calculated as a percentage (%) using Eq. 5, where m_{ext} is the mass of the dry extract obtained, and m_{rm} is the total amount of raw material used for the extraction.

$$X_0\,(\%) = \frac{m_{ext}}{m_{rm}} \times 100$$

5

2.6 Analysis of the extracts

2.6.1 Phytochemicals

The total phenolics content (TPC) was determined using the method described by Koşar, Dorman and Hiltunen [27] with some modifications. A standard curve of gallic acid was constructed for quantification. Extracts obtained by SOX, MAE, and PLE were dissolved in hydroalcoholic solutions at 10 mg mL⁻¹ and diluted as needed. Then, 10 µL of each sample were mixed with 50 µL of Folin's reagent, followed by 150 µL of 20% sodium carbonate and water to the final volume. After 2 h incubation at room temperature in darkness, absorbance was measured at 760 nm using a spectrophotometer (Agilent, Epoch-BioTek, USA). TPC was expressed as mg of gallic acid equivalents per gram of extract (mg GAE g⁻¹).

Total flavonoids content (TFC) was determined based on the method used by Woisky and Salatino [28], with some modifications. Initially, 10 mg of the dried extracts were dissolved in 1 mL of the extraction solvent, followed by an ultrasonic bath (Eco-Sonics, Ultronique Q3.0/37 A, Brazil) to ensure complete solubilization. The analysis solution was prepared by mixing 0.15 mL of the extract solution with 0.15 mL of a 2% AlCl3 solution and 0.7 mL of ethanol (p.a.). After 30 minutes of rest, absorbance was measured at 415 nm using a spectrophotometer (Agilent, Epoch-BioTek, USA). A control solution prepared following the same procedure but without the addition of $AlCl_3$ was used as the blank. The presence of flavonoids was indicated by the appearance of a green coloration. For quantification, a calibration curve was constructed using quercetin at concentrations of 0.075 to 0.25 mg mL^{-1}. The total flavonoid content was expressed as quercetin equivalents per gram of extract (mg EQ g^{-1}).

The total carotenoids content (TCC) was quantified using a modified version of the method described in Kuhnen et al. [29]. Dried extracts were diluted in a solvent mixture of methanol, hexane, and acetone in a 2:1:1 ratio (v/v/v), then subjected to an ultrasonic bath (Eco-Sonics, Ultronique Q3.0/37 A, Brazil) for 2 minutes. Following this, the samples were centrifuged for 15 minutes (Solab, SL-700, Brazil) at 3400 rpm. The supernatant was collected, and its absorbance was measured at 450 nm using a spectrophotometer (Agilent, Epoch-BioTek, USA). The TCC of the extracts was determined based on a standard curve using spectrophotometric standard β-carotene with concentrations ranging from 0 to 0.055 mg mL^{-1}. Results were expressed as milligrams of β-carotene equivalents per gram of dried extract (mg BCE g⁻¹) and standard deviation.

2.6.2 Antioxidant potential

The radical scavenging activity was determined using the $ABTS^+$ radical assay, following the method described by Re et al. [30]. The ABTS radical was generated by reacting 2.45 mM potassium persulfate with 7 mM of 2,2'-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), and the resulting radical

solution was stored in the dark at room temperature for 16 hours before use. The solution was then diluted to an absorbance of 0.7 (± 0.2) at 734 nm. Dried extracts were reconstituted in their respective extraction solvents to prepare the test solutions. Trolox was used as the standard, and a calibration curve, ranging from 0 to 300 μmol L^{-1}. The diluted extracts were mixed with the ABTS solution and allowed to react in the dark for 45 minutes. After the reaction, absorbance was measured at 734 nm using a spectrophotometer (Agilent, Epoch-BioTek, USA). The results are expressed as micromoles of Trolox equivalent per gram of dried extract (μmol TE g^{-1}).

The antioxidant capacity was also assessed using the DPPH (2,2-diphenyl-1-picrylhydrazyl) radical scavenging assay, following the protocol outlined by Mensor et al. [31]. Trolox was employed as the standard to generate the calibration curve, spanning concentrations from 0 to 300 μmol L^{-1}. Solutions of the dried extracts were prepared by reconstituting them in their respective extraction solvents. The DPPH radical solution was mixed with the diluted extracts and allowed to react for 30 minutes at ambient temperature, protected from light. Subsequently, the absorbance of the samples was measured at a wavelength of 517 nm using a spectrophotometer (Agilent, Epoch-BioTek, USA). The results are expressed in micromoles of Trolox equivalent per gram of dried extract (μmol TE g^{-1}) and standard deviation.

2.6.3 Phenolic profile determination by HPLC–ESI-MS/MS of ucuúba seed cake extracts

For the liquid chromatography analysis, the extract samples C2-SOX-70, C1-MAE-70, and C1-PLE-70 were selected as the most representative of each cake sample (C1 and C2). This sellection was based on the TPC results presented in section 3.2.3. The sellected extracts-maintained consistency in the ethanol concentration, as 70% ethanol yielded the most notable results in the majority of the analyzed extracts. Additionally, one extract from each extraction method was selected to facilitate comparisons between the techniques.

The dry extracts (0.1 g) were defatted twice with 25 mL of hexane in an ultrasonic bath (15 min, room temperature), followed by centrifugation at 3400 rpm for 15 min. After solvent removal, acid hydrolysis was performed by adding 5 mL of methanol and 5 mL of 6 mol L^{-1} HCl, incubating the mixture at 85°C for 30 min. The pH was adjusted to 2 with NaOH (6 and 1 mol L^{-1}), and the hydrolysates were extracted three times with 10 mL of diethyl ether, centrifuged (3400 rpm, 10 min), and the solvents evaporated at 40°C. Final residues were resuspended in 1 mL of HPLC-grade methanol, diluted 1:10 in methanol/water (30:70, v/v), and analyzed by LC-ESI-MS/MS. The phenolic profile (quantification) was determined according to Seraglio et al. [32], using an HPLC system (1200 Series, Agilent Technologies, Germany) coupled to a hybrid triple quadrupole/linear ion trap mass spectrometer (Q Trap 3200, Applied Biosystems/MDS Sciex, Canada) operating in negative electrospray ionization mode. Chromatographic separation was performed on a Synergi 4u Polar-RP column (150 × 2.0 mm, 4.0 μm, Phenomenex, USA) at 30°C, with a flow rate of 250 μL min^{-1} and injection volume of 10 μL. The mobile phases were 95% methanol/5% water (v/v) (A) and water with 0.1% formic acid (v/v) (B), using a segmented gradient.

Forty-eight phenolic compounds were identified and quantified by LC-MS/MS using calibration curves of standards and confirmed by 2–3 specific MRM transitions and retention times. Sample results were expressed in μg g^{-1}.

2.7 Statistical analysis

The data are presented as mean ± standard deviation. The analysis of graphs and results, which included the application of the t-test, Tukey test, and Pearson's correlation, was performed using OriginPro 2021 software (OriginLab, Northampton, USA), with a significance level of 5%.

3 Results and discussion

3.1 Proximate composition of cake

Table 1 shows the values of the proximate composition of the cake produced from the hydraulic pressing of ucuúba seeds (C1). It is important to note that the defatted cake (C2) is expected to present higher concentrations of the remaining components, as the residual lipid fraction has been removed a behavior also observed in the study by Polmann et al. [33] with Gurguéia nut cake.

The analyses from C1 revealed low moisture content, suggesting its favorable storage stability, thereby reducing microbial spoilage risks [34]. The total ash content was similar to obtained by Sobczak et al. [35] for various seeds press cakes, including avocado pit, sunflower, and flax (from 2.30 to 8.09 g 100 g^{-1}), indicating that the mineral composition of ucuúba cake is similar to that of other agro-industrial by-products. The protein content in C1 is closely aligned with that of *araticum* seed cake (15.36 g 100 g^{-1}), making it comparable to other oilseeds and suggesting that ucuúba cake could serve as a significant source of plant-based protein [36]. Additionally, the lipids content from various seeds press cakes is typically reported to range between 1.30 and 31.42 g 100 g^{-1}, where, according to Sobczak et al. [35] results, the most oily seeds are sunflower and pumpkin seed press cakes.

Table 1
Proximal composition (wet basis) and minor carbohydrates of ucuúba seed cake from hydraulic press.

Components (g $100g^{-1}$)[1]	C1*
Moisture	5.16 ± 0.13
Total ash	3.19 ± 0.23
Proteins[2]	15.09 ± 0.57
Lipids	33.24 ± 2.71
Total carbohydrates content[3]	43.32 ± 0.65
Energetic value (kcal $100g^{-1}$)[4]	532.80
Carbohydrates (mg $100g^{-1}$)[5]	5.93 ± 0.59

* C1: cake resulted from the hydraulic press of the seeds, without pretreatment. [1] Constituents in mean values ± standard deviation of two replicates, expressed in wet basis. [2] Protein conversion factor of 5.30. [3] Total carbohydrates content calculated by diference of the macronutrients. [4] Calculated considering crude protein, and total carbohydrate contents, multiplied by 4, and also the total lipids content multiplied by 9. [5] Minor carbohydrates determined by spectrophotometric method.

The lipids content from C1 showed slightly higher content compared to results from Sobczak et al. [35] for different oily seeds, confirming the commom name of ucuúba, also called “fat tree”, and attributed to the naturally high lipids content from ucuúba seeds. It is work mention that the pressing method used may have contributed to the lipid retention in the cake due to low pressing efficiency, which affected the C1 lipid content. The high value of total carbohydrate content from ucuúba seed cake (43.32 g 100 g^{-1}) represents all carbohydrate fraction including crude fibers, which according to Rodrigues et al. [37], ucuúba seeds cake contains approximatelly (33.91 g of fiber per 100 g^{-1}). Additionally, the minor carbohydrate content was (5.93 g 100 g^{-1}), indicating that most of the carbohydrates in the seed cake (C1) are present in complex forms, such as dietary fibers. The energetic value of the C1 cake was (532 kcal 100 g^{-1}), reflecting its high caloric density primarily attributed to its lipid and protein contents. These results highlight the potential of ucuúba cake as a promising source of valuable compounds, reinforcing its applicability in various industrial sectors and aligning with a biorefinery strategy aimed at the integral utilization of by-products.

3.2 Ucuúba cake extracts obtained by SOX, MAE, and PLE

3.2.1 Kinetics extraction curves

Kinetics assays were conducted for MAE and PLE methods to aid the definition of the processing extraction time through the kinetics curves, as presented by Weinhold et al. [38], and applied by Rudke et al. [39]. Figure 2 shows the kinetics extraction curves for samples C1 and C2 submited to MAE (Figs. 2(a) and 2(b) respectively) and to PLE (Figs. 2(c) and 2(d), respectively). The kinetics curves for MAE were fit with Gompertz model (Figs. 2(a) and 2(b)), and the kinetics parameters generated were: ae = 18.1347, k = 0.1698, and R^2 = 0.9933 for C1, and ae = 15.4272, k = 0.1579, and R^2 = 0.9911 for C2, indicating the good adjust of the model. The extraction time was defined later than the constant extraction rate (CER) period, and after the beginning of the falling extraction rate (FER) period, as discussed in the literature [40]. Therefore, 20 min was defined as the adequate period for C1 and C2 submitted to MAE.

Analogously, the kinetics curves for PLE, represented by Figs. 2(c) and 2(d) for C1 and C2 respectively were adjusted by Piecewise model. The kinetics parameters for C1 were defined as: a_1 = 0.0569 g, k_1 = 0.0489 g min-^{1}, t_{CER} = 8.3822 min, k_2 = 0.0099 g min^{-1}, t_{FER} = 26.7556 min, k_3 = 2.29 x 10^{-4} g min^{-1}, and R^2 = 0.9787. These results suggest the definition of the end of the CER as 9 min and the end of the FER as 27 min (adjusting the modeliung values of t_{CER} and t_{FER}). During the first 9 min, corresponding to the CER period, the high extraction rate is typicaly due to the high availability of solutes on the surface of the particles. At this period the mass transfer is controled by convection mechanism due to the significant amount of lipids on the press cake (C1), facilitating its solubilization by the solvent. At the FER period, from 9 to 27 min, the easily accessible solute on the surface of the particles begins to deplete, and mass transfer by diffusion from the interior of the particles starts and is combined with the convection, slowing the mass transfer, compared to the CER period [40]. The extraction was completed at the beginning of the third period, from 27 to 45 min, where the mass transfer is diffusion controlled (DC period). This stage is primarily governed by the diffusion of solutes from the interior of the particles, occurring very slowly due to their low availability. Therefore, the total extraction time was determined to be 30 min, consistent with the time indicated by the model (t_{FER} = 26.76 min).

Figure 2(d) illustrates the extraction kinetics of the C2 sample subjected to the PLE process. The kinetic parameters derived from the Piecewise model were as follows: a_1 = 0.0567 g, k_1 = 0.0592 g min^{-1}, t_{CER} = 8.03 min, k_2 = 0.0089 g min^{-1}, t_{FER} = 26.34 min, k_3 = 0.0017 g min^{-1}, and R^2 = 0.9803. From these results, the end of CER period was als considered at 9 min, and the FER period up to 27 min (adapting from the t_{CER} and t_{FER} values), when the DC period starts. Based on these parameters, the total extraction time was selected as 30 min.

3.2.2 Extraction yield

Figure 3 shows the extraction yields from ucuúba seed cakes obtained by SOX, MAE, and PLE with ethanol 100% or with hydroethanolic solutions at 50% and 70% (v/v), with results statistically different. The highest yields were observed for sample C1, which contained 33.24% residual lipid fraction (Table 1). The extracts C1-SOX-70, C1-MAE-100, and C1-PLE-100 reached yields close to 30%, with no significant differences between them ($p > 0.05$). In contrast, the extracts obtained with 50% ethanol

produced significantly lower yields, around 15%, suggesting lower ethanol concentrations do not effectively solubilize the liposoluble compounds present in the matrix.

This behavior is due to the high solubility of the residual fat in ethanol, then, hydroethanolic mixtures with high ethanol concentration contribute to an efficient extraction [41]. On the other hand, the best yield values for the extracts from the degreased cake (C2) were statistically lower compared to the highest yield values obtained from C1. Specifically for C2, the extracts C2-SOX-50, C2-SOX-70 and C2-PLE-70 achieved the highest yield values, without significant differences. High yields for C2 were obtained using hidroethanol mixtures due to the ability of these solvents to extract a broader range of compounds, including phenolic and other polar compounds, which are better solubilized in water. Therefore, to obtain high extractions yields it is important to select properly the solvent and the extraction method.

3.2.3 Quality profile of the extracts from ucuúba seed cake extracts

Quality attributes of the extracts recovered from C1 and C2 were evaluated in terms of total phenolics, flavonoids and carotenoids content (TFC, TFC and TCC), respectively, and also related to the antioxidant potential evaluated by ABTS and DPPH assays. These quality attributes are presented in Table 2 for extract from C1 and C2 obtained by SOX, MAE, and PLE techniques and different solvents. Overall, a significant increase in the recovery of phytochemicals and the antioxidant potential was observed for the extracts from C2, compared to the ones from C1.

This behavior is due to the reduction in the residual lipid fraction from the cake by the pretreatment applied, i.e., the lower lipid content from the cake probably improves the recovery of the antioxidant compounds due to higher interaction with the solvent, and reduced competition of the solutes for dissolution.

The highest TPC values were found in extracts from C2, particularly C2-MAE-70 and C2-SOX-70, with contents of 126.78 and 109.80 mg GAE g^{-1} respectively. Notably, C2-MAE-70 extract showed significant differences ($p < 0.05$) compared to other extracts, highlighting the combined effectiveness of the microwave process applied to the pretreated sample (C2). These results reinforce the suitability of ethanol 70% as solvent due to its intermediate polarity, contributing to the extraction of hydrophilic and lipophilic compounds [42]. Besides, significantly low TPC values have been reported from extracts obtained with ethanol 100%. For instance, Terpinc et al. [43], reported TPC values of 4.5 mg GAE g^{-1} in camellia cake, 3.4 mg GAE g^{-1} in rapeseed cake, and 0.12 mg GAE g^{-1} in flaxseed cake. Additionally, Mannucci et al. [44] also evaluated flaxseed cake, without pretreatment, finding TPC content of 4.85 mg GAE g^{-1}.

Regarding total flavonoids content (TFC), the extracts with the highest concentrations were C2-SOX-50 (13.10 mg QE g^{-1}) and C1-PLE-50 (11.37 mg QE g^{-1}), with significant differences ($p < 0.05$) in comparison to the other extracts. These results highlight the effectiveness of ethanol 50% to recover flavonoids from C1 and C2. In contrast, ethanol 100% resulted in lower flavonoid concentrations, probably due to the

structure of myricitrin, the main flavonoid identified in the extracts (as presented in section 3.2.4). Myricitrin contains several hydroxyl groups and a rhamnose sugar, which confers hydrophilic character, while its aromatic core contributes a less polar region [45]. Therefore, solvents with intermediate polarity, such as ethanol 50%, are more adequate in dissolving flavonoids with these characteristics, as they can interact more effectively with the polar and non-polar regions of the molecule. When comparing the results of this study with those reported by Kaur et al. [46] for five other oilseed cakes, the TFC values observed were within the reported range, from 8.29 mg QE g^{-1} in mustard cake to 18.60 mg QE g^{-1} in nigella cake.

In the case of total carotenoids content (TCC), the highest levels were found at C2-MAE-70 (18.74 mg BCE g^{-1}) and C2-SOX-50 (18.54 mg BCE g^{-1}) extracts, with no significant differences ($p > 0.05$). Therefore, the carotenoids remain in the cake after pretreatment (removal of the lipid fraction). In a defatted cake (C2), the lower saturation of the solvent with heavy lipids allows higher solubility of the carotenoids. Although carotenoids are lipophilic, their affinity for the solvent is lower compared to other fatty components, which facilitates their extraction in a fat-free matrix. Furthermore, the TCC values obtained in this study are higher than those reported for buriti, a well known carotenoid rich Amazonian fruit, which presents concentrations of (10.06 mg BCE g^{-1}) from the pulp and (10.43 mg BCE g^{-1}) from the peel [47, 48].

Table 2
Total compounds of phenolics (TPC), flavonoids (TFC), and carotenoids (TCC), and antioxidant activity by DPPH and ABTS methods from extracts recovered with different solvents from cakes without pretreatment (C1) and with pretreatment (C2).

Cake	Extracts	TPC (mg GAE g^{-1})	TFC (mg QE g^{-1})	TCC (mg BCE g^{-1})	DPHH (μmol TE g^{-1})	ABTS (μmol TE g^{-1})
	C1-SOX-70	85.93 ± 3.56^{e}	2.74 ± 0.18^{h}	4.78 ± 0.91^{f}	537.03 ± 0.46^{i}	789.95 ± 1.47^{i}
	C1-MAE-50	108.06 ± 5.74^{bc}	7.85 ± 0.64^{de}	12.25 ± 0.08^{c}	602.68 ± 1.35^{g}	929.32 ± 1.03^{f}
	C1-MAE-70	99.41 ± 2.10^{cd}	6.59 ± 0.56^{fg}	10.27 ± 0.04^{d}	652.73 ± 1.69^{e}	1052.79 ± 2.61^{c}
C1	C1-MAE-100	15.66 ± 0.47^{g}	0.26 ± 0.17^{i}	3.49 ± 0.20^{fg}	105.45 ± 0.64^{k}	447.14 ± 1.32^{k}
	C1-PLE-50	65.46 ± 1.08^{f}	11.37 ± 0.23^{b}	12.17 ± 0.48^{c}	693.63 ± 0.64^{d}	975.02 ± 1.73^{e}
	C1-PLE-70	70.60 ± 2.66^{f}	5.86 ± 0.22^{g}	6.84 ± 0.48^{e}	625.09 ± 0.64^{f}	1004.89 ± 2.54^{d}
	C1-PLE-100	4.34 ± 0.36^{h}	2.72 ± 0.37^{h}	2.03 ± 0.09^{g}	182.47 ± 0.64^{j}	660.90 ± 1.27^{j}
	C2-SOX-50	98.49 ± 1.46^{d}	13.10 ± 0.25^{a}	18.54 ± 1.13^{ab}	656.96 ± 0.55^{e}	846.65 ± 1.47^{h}
	C2-SOX-70	109.80 ± 6.07^{b}	9.75 ± 0.33^{c}	16.81 ± 0.28^{b}	565.20 ± 0.55^{h}	869.50 ± 0.73^{g}
C2	C2-SOX-100	88.92 ± 3.47^{e}	8.05 ± 0.06^{de}	12.60 ± 1.36^{c}	755.92 ± 1.91^{c}	1286.69 ± 2.54^{a}
	C2-MAE-70	126.78 ± 0.47^{a}	8.40 ± 0.23^{d}	18.74 ± 0.18^{a}	791.71 ± 4.58^{a}	1257.91 ± 1.47^{b}
	C2-PLE-70	85.73 ± 1.17^{e}	7.19 ± 0.62^{ef}	11.28 ± 0.50^{cd}	779.82 ± 6.05^{b}	1049.74 ± 1.47^{c}

Values expressed as mean ± standard deviation (n = 3). Different letters within the same column indicate significant differences ($p < 0.05$) according to Tukey's test.

Lastly, the antioxidant activity of the C2-MAE-70 extract, obtained with ethanol 70%, showed a significantly higher DPPH value (791.71 μmol TE g^{-1}), significantly different from other samples ($p < 0.05$), and also achieved the second best ABTS result (1257.91 μmol TE g^{-1}). Meanwhile, the C2-SOX-100

extract showed the highest ABTS value (1286.69 μmol TE g^{-1}), with significant differences compared to other extracts ($p < 0.05$). These highlight the critical role of the deffating pretreatment (C2) to enhance the recovery of antioxidant compounds. Additionally, the results indicate that MAE samples recovered with ethano 70% showed high antioxidant potential, particularly from C2. The microwave energy from MAE promotes cellular disruption of the plant matrix, facilitating the release of compounds with antioxidant capacity [49]. Notably, the best ABTS results surpass those reported by de Souza Silva et al. [50] for hydroethanolic extracts of by-products from the pressing process of Amazonian açaí (820 μmol TE g^{-1}). Therefore, ethanol 70% and MAE maximizes the extraction of antioxidant compounds from C1 and C2.

The correlogram in Fig. 4 shows the Pearson correlation coefficients (r) between the phytochemical compounds (TPC, TFC, and TCC) and the antioxidant potential, evaluated by ABTS and DPPH assays. All variables exhibited positive correlations. Strong correlations ($r > 0.70$) were observed between TPC × TCC ($r = 0.82$), TPC × DPPH ($r = 0.84$), TPC × ABTS ($r = 0.71$), TFC × TCC ($r = 0.87$), TFC × DPPH ($r = 0.72$), TCC × DPPH ($r = 0.72$), and ABTS × DPPH ($r = 0.90$). These correlations indicate that phenolic compounds, flavonoids, and carotenoids significantly contribute to the antioxidant capacity of the samples.

Total phenolics content (TPC) stands out, providing strong correlation with DPPH and ABTS values, highlighting its central role in antioxidant activity. Although flavonoids and carotenoids are present in smaller quantities, they also contribute significantly, particularly with DPPH results. Porrawatkul and Pimsen [51] also observed a strong correlation between the phenolic compounds and DPPH values from extracts with ethanol 95% from nutmeg seeds, confirming the importance of an efficient extraction process to recover these bioactive compounds to optimize the antioxidant capacity of the obtained extracts.

3.2.4 Phenolic compounds present in the extracts from ucuúba seed cake

The phenolic compounds identified in the C2-SOX-70, C1-MAE-70, and C1-PLE-70 extracts are summarized in Table 3. The most representative compounds were ferulic acid, vanillic acid, and vanillin respectively. The flavonoid with the highest concentration was Myricitrin, particularly abundant in extracts from cake without pretreatment (C1) obtained using PLE extraction, where its concentration exceeded 12.43 μg g^{-1}. This flavonoid was also detected in the C2-SOX-70 extract from degreased cake; however, quantification was not possible, possibly due to degradation caused by the high temperatures of the conventional SOX method. Although myricitrin is found throughout the plant kingdom, it is primarily produced by members of the Myricaceae family, such as the genus Virola [45]. Among the phenolic acids analyzed, ferulic acid and vanillic acid presented the highest concentrations in all the evaluated extracts, especially in the defatted cake (C2). Ferulic acid (4-hydroxy-3-methoxycinnamic acid) reached concentrations above 12000 μg g^{-1} in all the samples analyzed. This compound, widely recognized for its anti-inflammatory, antimicrobial, and anticancer properties, has also been reported in

other species within the Virola genus, such as in methanolic extracts of *Virola venosa* leaves [52]. Furthermore, ferulic acid has been found in the press cakes of various oilseeds, including *Hodgsonia heteroclita*, with a concentration of 8963.1 μg g^{-1}, as well as in sesame, flaxseed, white mustard, and rapeseed [43, 53, 54]. Widely recognized for its anti-inflammatory, antimicrobial, and anticancer properties, ferulic acid can also inhibit cell proliferation and induce apoptosis in osteosarcoma cells by blocking the PI3K/Akt pathway [55, 56]. Its low toxicity and high bioavailability enhance its potential for use in the food and cosmetic industries [57].

Vanillic acid also exhibited significant concentrations, exceeding 1300 μg g^{-1} in all evaluated extracts, with the highest levels found in the defatted cake (C2). A study reported that this compound accounted for 12.8% of the total phenolic compounds in peanut press cakes [58]. As a benzoic acid derivative, vanillic acid is recognized for its antimicrobial, anticancer, and wound healing properties [59]. It has demonstrated effectiveness against lung cancer cells, offers protection against toxicity in leukemia models, and promotes wound healing by regulating gene expression in fibroblasts [60]. However, it is essential to evaluate its environmental implications to ensure safe and sustainable use [61].

Table 3
Profile of phenolic compounds in extracts obtained with 70% ethanol from the non-defatted cake (C1) using Soxhlet extraction (SOX) and from the defatted cake (C2) using pressurized liquid extraction (PLE) and microwave-assisted extraction (MAE) (μg g^{-1}).

Phenolic compounds (μg g^{-1})	C2-SOX-70	C1-MAE-70	C1-PLE-70
Flavonoids			
Apigenin	0.43 ± 0.18	0.42 ± 0.16	0.60 ± 0.11
Eriodictyol	< LOQ	0.31 ± 0.09	0.58 ± 0.02
Fustin	0.73 ± 0.20	1.08 ± 0.67	0.58 ± 0.33
Hispidulin	2.96 ± 0.03	1.48 ± 0.00	1.54 ± 0.56
Quercetin	0.69 ± 0.24	0.61 ± 0.04	0.56 ± 0.04
Myricitrin	< LOQ	10.78 ± 0.67	12.43 ± 0.89
Taxifolin	< LOQ	6.96 ± 2.67	4.61 ± 1.11
Phenolic acids			
4-Aminobenzoic	591.70 ± 208.50	421.38 ± 14.89	478.65 ± 160.93
Protocatechuic	99.58 ± 9.11	79.51 ± 13.78	75.17 ± 7.11
p-Coumaric acid	263.62 ± 8.89	259.88 ± 25.56	198.64 ± 19.56
Vanillic acid	3021.41 ± 99.14	1388.36 ± 33.34	1562.64 ± 98.25
Caffeic acid	502.35 ± 1.78	401.39 ± 20.45	397.47 ± 54.68
Ferulic acid	14885.64 ± 23.34	13690.25 ± 124.70	12788.94 ± 95.80
Syringic acid	18.67 ± 1.33	9.43 ± 0.22	6.61 ± 6.00
Sinapic acid	32.90 ± 5.33	24.71 ± 0.44	21.45 ± 0.89
Phenolic aldehydes			
Vanillin	1778.66 ± 31.79	809.75 ± 108.03	1153.34 ± 207.38
Coniferaldehyde	209.38 ± 20.67	156.11 ± 3.33	157.95 ± 21.78
Syringaldehyde	104.03 ± 7.56	70.75 ± 10.00	29.46 ± 20.23
Sinapaldehyde	835.54 ± 204.72	557.95 ± 8.00	454.80 ± 2.22
Coumarins			
Umbelliferone	91.58 ± 9.11	71.20 ± 0.89	67.15 ± 6.45

The data are expressed as mean ± standard deviation (n = 2). < LOQ – identified but not quantified.

Vanillin was the phenolic aldehyde found in the highest concentration, particularly in the defatted cake (C2), with values exceeding 1700 μg g^{-1}. In comparison, this compound has been reported in much lower concentrations in the press cake of faveleira seeds, with a value of 2.09 μg g^{-1} [62]. Vanillin and its derivatives exhibit a broad range of pharmacological activities, including antidiabetic, anticancer, antioxidant, and antimicrobial effects [63]. Recognized as safe by regulatory bodies such as the FDA and FEMA, vanillin holds great potential for applications in functional foods and disease prevention [64]. In the biomedical field, it has been incorporated into hydrogel formulations for wound treatment, enhancing antibacterial properties and promoting cell viability [65].

4 Conclusion

This study presents novel and pioneering information on the sustainable use of ucuúba seed press cake, and representing the recovery of bioactive compounds from this underused byproduct by means of non-conventional extraction methods. The results demonstrate that this agri-food byproduct is rich in bioactive compounds with antioxidant activity. Sample C1 showed a significant content of lipids (33.24 g 100 g^{-1}), total carbohydrates content (43.32 g 100 g^{-1}), and proteins (15.09 g 100 g^{-1}). The highest yields were obtained for sample C1 using MAE and PLE methods with 100% ethanol as the solvent. The extract obtained using MAE with 70% ethanol showed the highest values of TPC (126.78 mg GAE g^{-1}), TCC (18.74 mg BCE g^{-1}), and antioxidant activity in the DPPH assay (791.71 μmol TE g^{-1}). On the other hand, the SOX method, using 50% ethanol, obtained the most representative values for TFC (13.10 mg QE g^{-1}), while the use of 100% ethanol showed the highest values in the ABTS assay (1286.69 μmol TE g^{-1}). A strong correlation was observed between phenolic compounds and antioxidant activities. The main phenolic compounds identified in the press cake extracts were ferulic acid (12788.94–14885.64 μg g^{-1}), vanillic acid (1388.36–3021.41 μg g^{-1}), and vanillin (809.75–1778.66 μg g^{-1}). These results suggest that the MAE method, combined with intermediate ethanol concentrations such as 70%, is the most effective for extracting and preserving the majority of bioactive compounds with antioxidant capacity from ucuúba press cake, outperforming the other techniques evaluated (SOX and PLE). Moreover, the degreasing pretreatment further improved the recovery of target analytes, emphasizing its relevance in optimizing the extraction process. Therefore, ucuúba press cake has high potential, promoting the application of its derived coproducts as ingredients in the food, cosmetic, and pharmaceutical industries. One possible cosmetic application of the obtained extracts is their use in skincare formulations, enriching their antioxidant value.

Abbreviations

ABTS: 2,2'-Azinobis-(3-ethylbenzothiazoline-6-sulfonic acid); AOAC: Association of official analytical Chemists; BCE: β-Carotene equivalents; C1: Ucuúba press cake obtained by hydraulic pressing (without pretreatment); C2: Degreased ucuúba press cake (after defatting pretreatment); CER: Constant extraction rate; DC: Diffusion controlled; DPPH: 2,2-Diphenyl-1-picrylhydrazyl; EtOH: Ethanol; FER: Falling extraction rate; GAE: Gallic acid equivalents; HPLC: High performance liquid chromatography; LC-ESI-MS/MS: Liquid chromatography–electrospray ionization–tandem mass spectrometry; MAE: Microwave-

assisted extraction; PLE: Pressurized liquid extraction; p.a.: Pro analysis grade (pure for analytical use); QE: Quercetin equivalents; R^2: Coefficient of determination; SOX: Soxhlet extraction; TE: Trolox equivalents; TCC: Total carotenoid content; TFC: Total flavonoid content; TPC: Total phenolic content; UV-Vis: Ultraviolet–visible spectroscopy.

Declarations

Acknowledgements

S. R. S. Ferreira would like to thank the National Council for Scientific and Technological Development (CNPq/Brazil) for the financial support (project 402304/2021-7). The authors also acknowledge Coordination for the Improvement of Higher Education Personnel (CAPES/Brazil), project PROEX 1513/2023, and Fundação de Amparo à Pesquisa e Inovação de Santa Catarina (FAPESC/Brazil), project 2022 TR 2176.

Declaration of competing interest

Ethical Approval

Not applicable.

Competing interests

The authors declare no conflict of interest in submitting the article.

Authors' contributions

Juan David Marmolejo Tascón: Conceptualization, Methodology, Validation, Visualization, Formal analysis, Data Curation, Investigation, Writing - Original Draft. **Jonas da Silva:** Formal analysis, Writing - Review & Editing. **Cristiano José de Andrade:** Supervision, Conceptualization, Resources, Funding acquisition, Writing - Review & Editing, Project administration. **Sandra Regina Salvador Ferreira:** Supervision, Conceptualization, Resources, Funding acquisition, Writing - Review & Editing, Project administration.

Funding

This work was partially funded by the Coordination for the Improvement of Higher Education Personnel (CAPES/Brazil), project PROEX 1513/2023, National Council for Scientific and Technological Development (CNPq), project 402304/2021-7, and Fundação de Amparo à Pesquisa e Inovação de Santa Catarina (FAPESC/Brazil), project 2022 TR 2176.

Availability of data and materials

The article presents all data relevant to this study.

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Figures

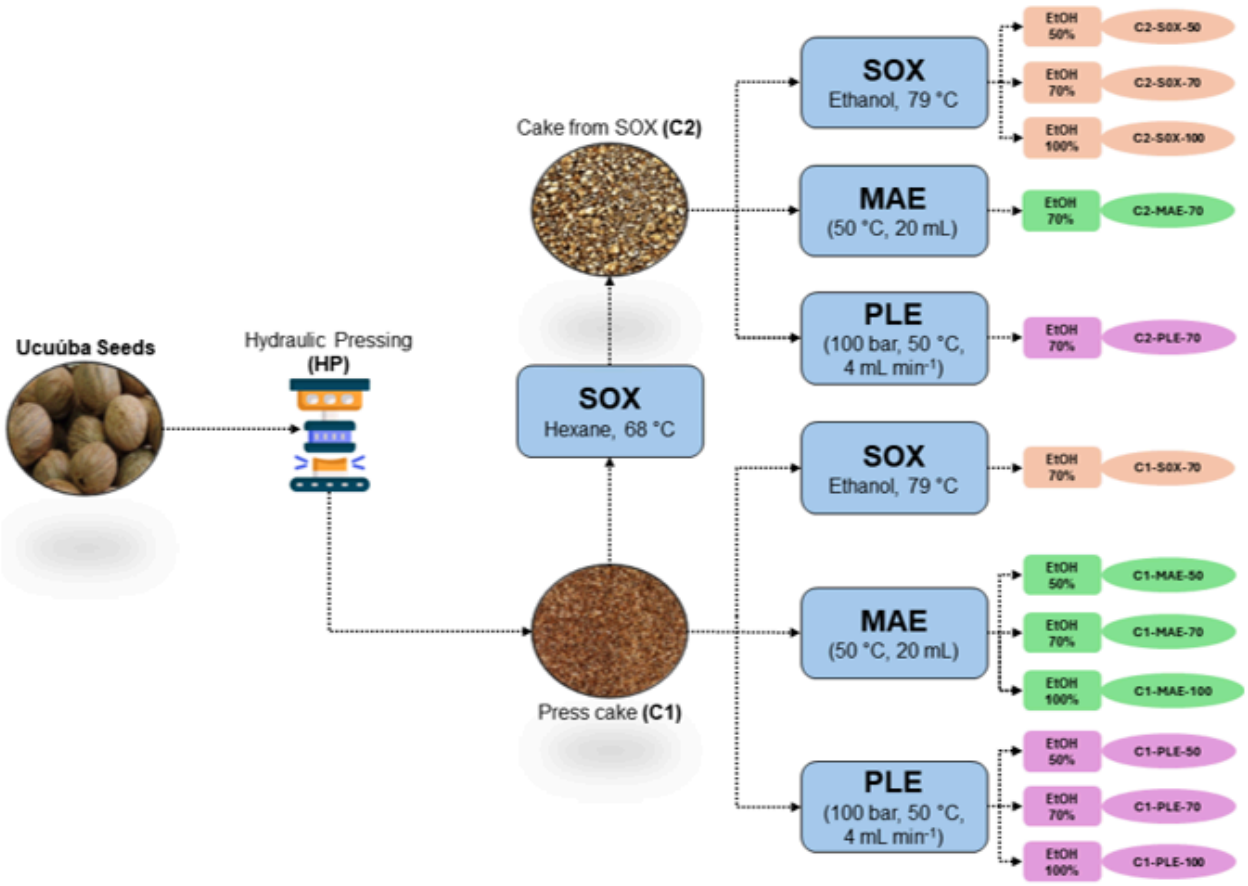

Figure 1

Extractions flowchart for obtaining extracts from ucuúba seed cake samples (C1 and C2) by using Soxhlet extraction (SOX), microwave-assisted extraction (MAE), and pressurized liquid extraction (PLE). The solvents used were ethanol (100%) and hydroethanolic mixtures (EtOH 50%, EtOH 70%).

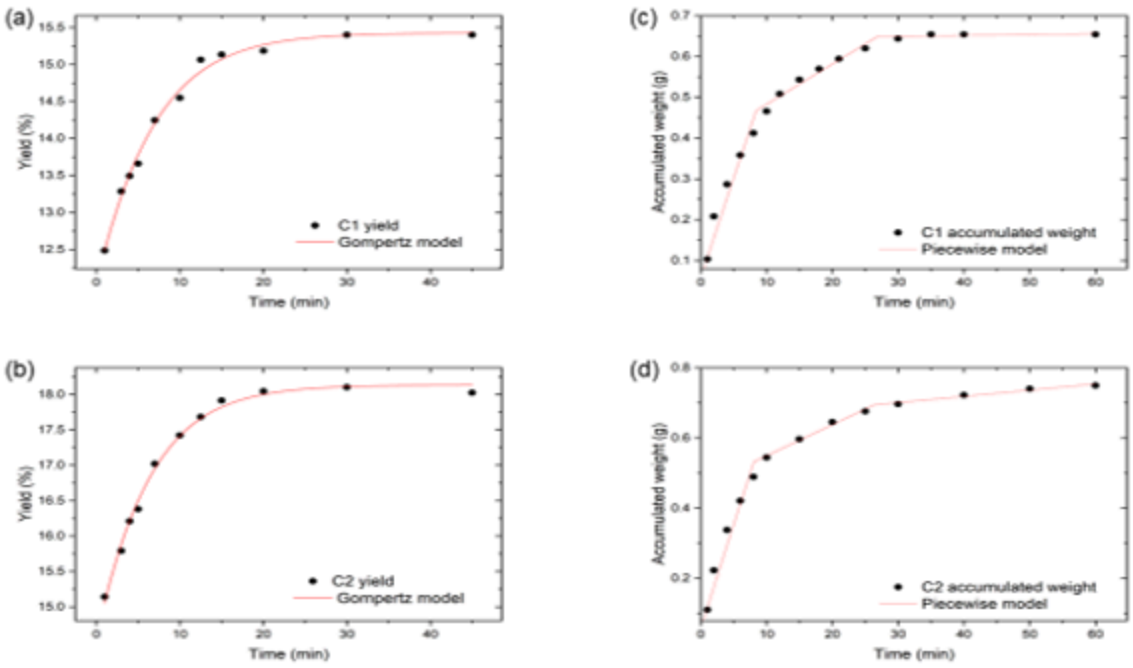

Figure 2

Kinetics extraction curves of ucuúba seed cakes obtained by microwave-assisted extraction (MAE), fitted to the Gompertz model: (a) C1 and (b) C2; and by pressurized liquid extraction (PLE), fitted to the Piecewise model: (c) C1 and (d) C2.

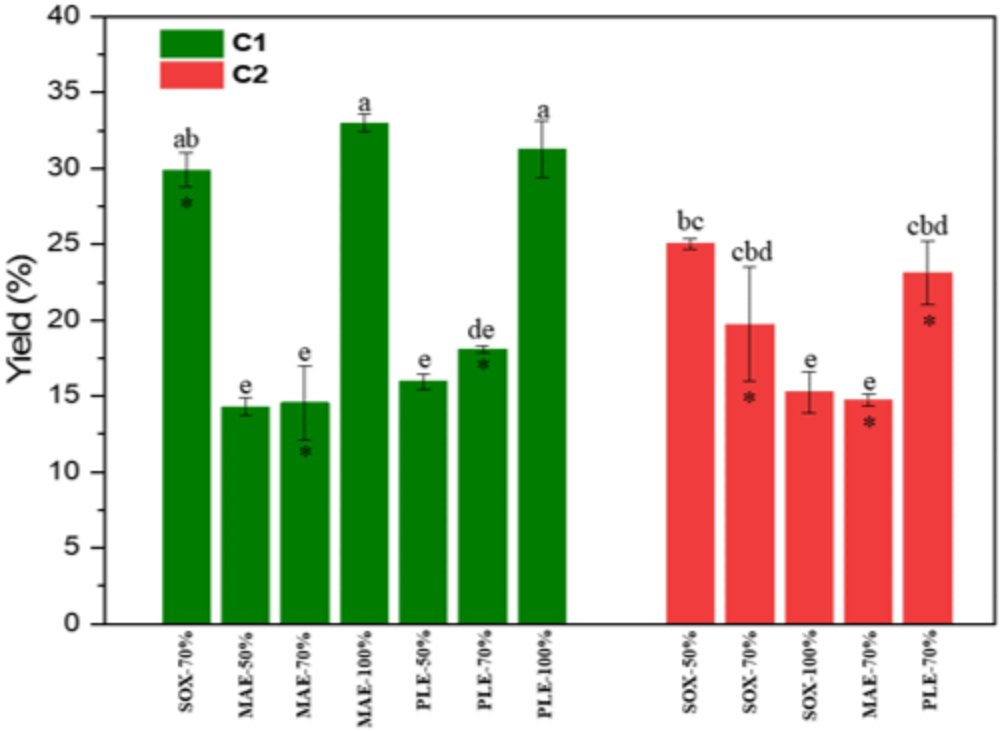

Figure 3

Extraction yields from ucuúba seed cakes (C1 and C2) obtained by SOX, MAE, and PLE with ethanol and with hydroethanolic solutions at 50% and 70% (v/v). * Extracts obtained using ethanol 70%.

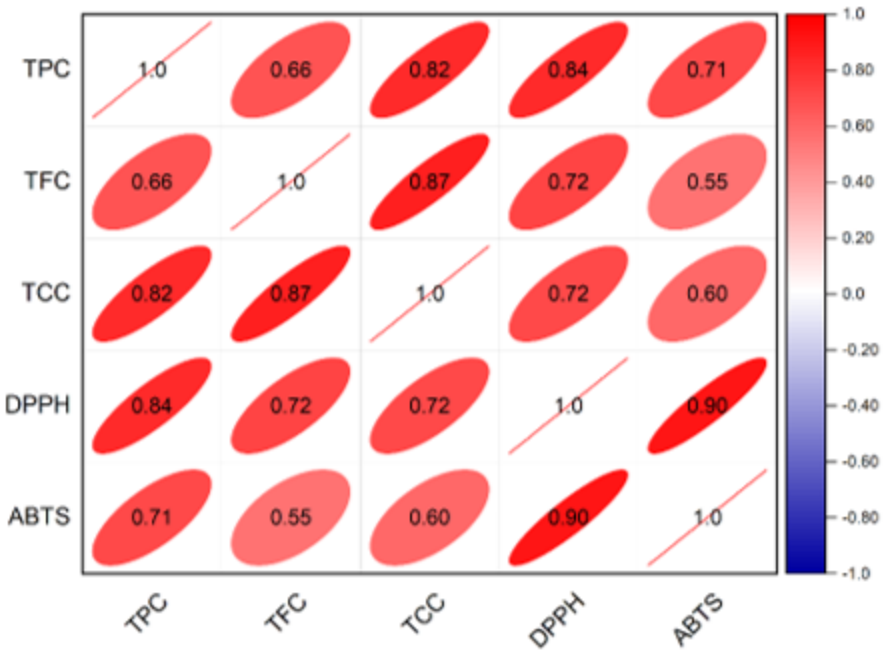

Figure 4

Correlogram showing the relationship between total phenolic compounds (TPC), total flavonoid compounds (TFC), total carotenoid compounds (TCC), (ABTS) radical scavenging, and scavenging activity of DPPH radical of extracts of ucuúba seed cakes from Pearson's coefficient (r).