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Article

Fatty Acid Content and Physicalchemical Properties of Cagaite Seed Oil (*Eugenia dysenterica* DC) Obtained by Different Extraction Methods

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Cagaiteira, the fruit popularly known as cagaite, belongs to the Myrtaceae family. Seed oil cagaite (*Eugenia dysenterica* DC) was obtained by three methods of extraction, extraction using an ultrasound (OCU), extraction by mechanical pressing (OCP) and extraction with Soxhlet extractor (OCS) in 3 different times (3, 6 and 9 h) and the content of fatty acids and physicalchemical properties were compared. The rate of saturated fatty acids ranged from 19.46% (OCP03) to 31.18% (OCS09), while the amount of unsaturated fatty acids ranged from 54.72% (OCP03) to 67.64% (OCS09). Linoleic and linolenic acids, important in nutrition food, varied between 32.22-38.11 and 5.55-8.78%, respectively. For oxidative stability, OCUAq (heated ultrasound extraction) samples showed the highest induction periods, showing a positive correlation with antioxidant activity and phenol content, demonstrating the efficiency of the use of ultrasonic extraction to obtain quality oils from cagaite seeds.

Keywords: extraction, oils, seeds, *Eugenia dysenterica* DC, cagaite, ultrasonic extraction, optimization

Introduction

The cagaiteira (*Eugenia dysenterica* DC) belonging to the family Myrtaceae, is a fruit tree of the Cerrado biome, which reaches up to 10 meters in height, and is widely found in this biome and in the states of Goiás, Minas Gerais and Bahia and can be found in large groupings. The fruit, popularly known as cagaite, is spherical in shape with a diameter of 3 to 5 cm and a light-yellow color, with a weight varying from 14 to 20 g and the seeds are cream color, oval in shape, with a diameter of 0.8 to 2.0 cm.[1-3]

Some studies on the content of fatty acids of cagaite are available in the literature. de Almeida[1] studied the nutritional composition of cagaite and verified its high water content, high content of essential polyunsaturated fatty acids and that the linoleic acid presented greater quantity than olive oil and palm oil. Also, the linolenic acid found exceeds the oil of corn, sunflower, peanut, soybean, oil palm and olive. Martinotto *et al.*[4] studied cagaite fruits and detected the fatty acids linoleic acid (10.5%) and linolenic acid (11.86%). Jorge *et al.*[5] evaluated the centesimal composition, antioxidant activity, fatty acids and tocopherols in cagaite seed oils obtained from ethyl alcohol extraction for 30 min and found a large amount of carbohydrates, considerable antioxidant activity and high content of phenolic compounds. The fatty acids in greater quantity were oleic acid and linoleic acid. Camilo *et al.*[6] carried out an investigation of the variation of the fatty acid components of the cagaite seed oil, gathering 440 samples of different plant seedlings and obtaining oils with 27% of saturated fatty acids and 73% of unsaturated acids for the samples analyzed.

Thus, considering the high levels of fatty acids present in cagaite, it is interesting to produce oils with the highest quality. However, the extraction process is a step that requires caution and care. There are several techniques of extraction of vegetable oils and the most used are by mechanical pressing or extraction using solvents.[7] Extraction using organic solvents presents higher yields,

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but they are unfavorable to the environment since they emit gases in the atmosphere, and operationally, steps are needed to remove this solvent, increasing operating costs and reducing its quality, because when subjected to high temperatures, some properties may be lost.[8,9]

The extraction of oils by mechanical pressing is a safer, lower cost method and better quality oil is obtained.[10] However, low yields are obtained by this method.[11] Thus, other forms of extraction of the oils are necessary. Extraction using ultrasound is an alternative. In this method, ultrasound waves cause physical and chemical changes in the samples due to pressure changes, which leads to cavitation.[12,13] Cavitation allows greater contact of the solvent with the intracellular product.[14] In the ultrasound, a more efficient contact between sample-solvent is allowed thanks to the stirring of the solvent during the extraction, increasing the penetration of the solvent in the sample.[15] Thus, the use of ultrasound increases the yields during extraction and presents potential for applications in the extraction of oils.[15] Some studies have been conducted for the production of oils using ultrasonic extraction from pomegranate,[16,17] winter melon,[18] papaya,[19] orange peel,[20] grape,[21] papaya[22] and canola.[23]

Until the present moment, there were no reports in which the fatty acid profile was compared, physicalchemical properties of cagaita seeds were compared using the three extraction techniques. Therefore, this work investigated and compared the results with the purpose of obtaining an oil of the seeds of cagaite of better quality.

Experimental

Collection of cagaite fruits and obtaining the seed powder for extraction

10.5 kg of cagaite (*Eugenia dysenterica* DC) fruits were collected in the botanical reserve of the Instituto Federal de Mato Grosso do Sul, Nova Andradina campus, MS, Brazil (22º 04' 50'' S 53º 27'15" W) in October 2017, and placed for drying in an oven at 50 °C for 24 h. The fruits were opened and the seeds removed. The seeds were then ground in a knife mill (WILLY MACRO-SV-1, Belo Horizonte, Brazil), obtaining at the end of this stage, 2.3 kg of cagaite seed flour that was used for oil extraction.

Extraction of the oil of the seeds of the cagaite using Soxhlet

In the extraction of the oil using the Soxhlet extractor (MyLabor, São Paulo, Brazil), 100 g of the fine powder of the seeds of the cagaite was used with the same volume of 250 mL of hexane (Dinâmica, MS, Brazil), maintaining the heating at 70 ºC. The extraction time was 3 h (OCS03), 6 h (OCS06), or 9 h (OCS09). After the extraction was complete, the extractor was allowed to come to room temperature and then the solvent was evaporated using the rotary evaporator.

Extraction of oil from the seeds of the cagaite using mechanical pressing

In the method of mechanical pressing, 100 g of seeds were weighed and placed in a stainless steel cylinder and pressed under pressure of 8 tons for 3 h (OCP03), 6 h (OCP06), or 9 h (OCP09). At the end of the extraction time, the oil obtained was weighed and stored under refrigeration.

Extraction of oil from the seeds of the cagaite using ultrasound

For the extraction of the oil, the ultrasonic equipment L100-Schuster (Santa Maria, Brazil) with ultrasonic frequency of 42 kHz was used with thermostated bath. To study also the influence of temperature, the extractions were first made with the temperature of the bath at room temperature (25 °C). Thus, 100 g of the seed powder of the cagaite was placed in an Erlenmeyer flask and the volume of 250 mL of hexane was added. The extractor vial was capped with a layer of plastic film. Thereafter, each sample was left at extraction time of 3 h (OCUAm03), 6 h (OCUAm06), or 9 h (OCUAm09). Then, other extractions were performed with the bath temperature at 70 °C, and the procedures described above were carried out, thus obtaining OCUAq03, OCUAq06 and OCUAq09 after 3, 6 and 9 h of extraction, respectively. After extraction, the flask was removed and allowed to cool to room temperature for 1 h. Thereafter, the oil was separated from the remaining plant material and evaporated from the solvent in the rotary evaporator.

Extraction yield (O%)

The yield of the different extractions in terms of the percentage of the oil was calculated according to equation 1, where Moil is the mass of the oil obtained and Mscg is the mass of the seeds initially used for the extraction.

$$O(\%) = \left(\frac{Moil}{Mscg}\right) \times 100 \quad (1)$$

Determination of the composition of fatty acids

To identify and quantify the fatty acids present in the samples, the methyl esters were prepared according

to ISO 5509:2000[24] using gas chromatography coupled with flame ionization detector (GC-FID). An aliquot of 250 mg of each esterified sample was mixed with 5 mL of 10 mg mL^{-1} methyl heptadecanoate solution, used as the internal standard, and then injected into a Varian CP-3800 gas chromatograph (Walnut Creek, USA) with automatic injector and ionization detector in flame (FID). The column used was a BPX 70 (SGE Analytical Science, Pflugerville, USA) measuring 30 m in length, 0.25 mm internal diameter and 0.25 μm film. Chromatographic parameters are shown in Table 1. A previous injection with chromatographic standards was performed to identify the peaks, define the quantification interval and to identify the retention time of the internal standard (C17) (Sigma-Aldrich, St. Louis, USA).

Table 1. Chromatographic parameters of GC-FID analysis of the samples of the oils of the cagaite seeds

Injector and detector parameters	
Injection volume / μL	1
Temperature of the injector / °C	200
Detector	FID
Injection mode	split
Split reason	1:100
Temperature of the detector / °C	250
Parameters of oven compartment	
Heating rate / (°C min^{-1})	4
Isotherm / min	10
Total running time / min	52
Temperature / °C	80
Drag gas	helium
Flow / (mL min^{-1})	1

FID: flame ionization detector.

Physical chemical properties of cagaite seed oils

The official methods of the American Oil Chemists' Society (AOAC) were used to measure the following parameters: acidity index (AI), peroxide value (PI), iodine value (IV) and refractive index (RI) of the oil samples obtained by the different extraction methods to compare their values.

Antioxidant activity and phenolic content of the oils of the seeds of cagaite

The free radical DPPH (2,2-diphenyl-1-picryhydrazyl) (Sigma-Aldrich, St. Louis, USA) method was used to evaluate the antioxidant activity of the oils obtained in each extraction, according to the methodology described by Brand-Williams *et al.*[25] 0.1 mL aliquot of each sample was diluted and added to 3.9 mL of a methanolic solution of DPPH (0.001 M) and after 30 min, the absorbance measurement was performed at the wavelength of 517 nm. A control measure of DPPH was made without any oil sample. The test was performed in triplicate. The calculation of the antioxidant activity was determined by equation 2:

$$\text{DPPH scavenging activity}(\%) = \left(\text{Acontrol} - \frac{\text{Asample}}{\text{Acontrol}}\right) \times 100 \quad (2)$$

Acontrol is the absorbance of the DPPH solution and Asample is the absorbance of the sample.

The content of phenolic compounds was also measured using the Folin-Ciocalteu method described by Liu *et al.*[26] The amount of 2.50 g of oil from each oil was solubilized in 5 mL of hexane and extracted using a 80:20 (v/v) methanol:water solution (Dinâmica, MS, Brazil). Upon separation of the aqueous phase, it was collected, centrifuged and dried at room temperature. In a 50 mL volumetric flask, the dried sample was transferred using 5 mL of methanol and 2.5 mL of the Folin-Cicateou reagent (Sigma-Aldrich, St. Louis, USA) and 10 mL of sodium carbonate (Sigma-Aldrich, St. Louis, USA) were added, adjusting the volume thereafter using deionized water. After 30 min, absorbance readings were taken at 765 nm. This measure was performed in triplicate. The results are expressed as mg equivalents of gallic acid *per* 100 g of oil (mg GAE kg^{-1}).

Oxidative stability of the cagaite seeds oils

The oxidative stability of the oils was measured using the METROHM equipment (Model Rancimat 873, Newark, USA) using the methodology described by Tabee *et al.*[27] Thus, 2.5 g of oil samples were analyzed in heating of 110 °C and constant air flow of 20 L h^{-1}. The temperature correction factor (ΔT) was set at 0.9 °C. The products formed by the decomposition were carried by a flow of air to a conductivity measuring cell that dissolves the volatile acids of the oil in deionized water. The induction time (h) is measured when there is an abrupt increase in electrical conductivity and this property is automatically calculated by the equipment's software.

Statistical analysis

All extractions and measurements of the oil properties obtained from the seeds of the cagaite by the different extraction methods used in this work were in triplicate. After obtaining all the data, we performed the analysis of variance (ANOVA) followed by the Duncan multiple range

procedure, at a level of 95% significance ($p > 0.05$) using Minitab software version 17.1.[28]

Results and Discussion

Extraction yield and physicalchemical quality of cagaite seeds oil

Table 2 shows the yield and quality of the oils obtained by the different types and times of extraction using the fine powder of cagaite seeds oil.

As the extraction time increased, there was an increase in the oil yield in all extraction methods tested, and Soxhlet extraction (OCS) proved to be more efficient in relation to the oil mass obtained from ultrasound extraction. The extraction by pressing was the least efficient of the three methods used, when compared with yields obtained with the same extraction time but different extraction methods. The extractions using ultrasound showed the best yield with heating, reaching 68.23% yield after 9 h of extraction (OCUAq09) and was lower in extractions performed in ultrasound without heating, compared to the same extraction time (OCUAm09-66.71%). In turn, the OCUAq yields were higher than the values of the OCP samples, which shows the increase in the yield and efficiency of extractions using ultrasound. Jorge *et al.*[5] carried out a study using dehydrated and crushed seeds of cagaite with ethyl alcohol at 40 °C as the extracting solvent, obtaining a yield extraction of 3.75%. Ixtaina *et al.*[29] investigated the yield of raw chia seed oils using pressing and extraction with hexane, achieving approximately 30% more oil in this method than pressing. Bhuiya *et al.*[30] studied the extraction of Australian Native Beauty Leaf Seed oil and obtained the best yields using chemical extraction with hexane. Chielle *et al.*[31] optimized the oil yield of papaya seeds under different conditions, obtaining seed oil with 19.23% yield.

The physicalchemical quality of the cagaite seeds oils obtained in these three different extraction methods was evaluated through parameters such as AI, PV, IV and RI. The AI provides a relevant information on the state of conservation of the oil and demonstrates its decomposition evaluated by the amount of free fatty acids. PV measures the amount of oxygen in the peroxide form in the sample and is the result of oxidation.[32] The Agência de Vigilância Sanitária (ANVISA) stipulates that the values for AI and PV do not exceed the maximum limit of 4.0 mg KOH *per* g and 15 meq O_2 *per* kg oil, respectively.[33] There were significant differences in the AI and PV in the oils obtained by the different extraction methods ($p > 0.05$) and all presented AI and PV below the limit established by ANVISA. The AI and PV were higher for OCS samples (OCS09-0.60 mg KOH *per* g and 0.67 meq O_2 *per* kg oil) and the samples obtained by ultrasound showed the lowest acidity values (OCUAm03-0.29 mg KOH *per* g) and the sample that presented lower PV was obtained by pressing (OCP03-0.19 meq O_2 *per* kg oil), indicating the low presence of free fatty acids and reactive radicals in the oils of the cagaite seeds.

The IV evaluates the amount of unsaturations of fatty acids present in samples of oils and RI is associated with saturations of the bonds and is affected by several factors such as amounts of free fatty acids, oxidation and heat treatment.[31] IR is widely used to evaluate the quality of vegetable oils.[34] The peroxide and refractive index are important parameters, since they show the oxidation of oils and fats and the presence of some flavors and odors in these samples demonstrate their

Table 2. Extraction yield and physicalchemical properties of cagaite seeds oil obtained by different extraction methods

Extraction	Extraction yield / %	Acidity index / (mg KOH *per* g oil)	Peroxide value / (meq O_2 *per* kg oil)	Iodine value / (g I *per* 100 g oil)	Refractive index (25 °C)
OCS03	64.84 ± 0.44	0.46 ± 0.18	0.43 ± 0.02	141.57 ± 0.21	1.4643 ± 0.0002
OCS06	70.12 ± 0.23	0.55 ± 0.14	0.54 ± 0.02	143.09 ± 0.29	1.4644 ± 0.0005
OCS09	73.11 ± 0.19	0.60 ± 0.11	0.67 ±0.04	145.18 ± 0.41	1.4646 ± 0.0006
OCP03	49.01 ± 0.22	0.32 ± 0.19	0.19 ± 0.01	138.01 ± 0.19	1.4622 ± 0.0002
OCP06	56.37 ± 0.38	0.48 ± 0.17	0.21 ± 0.02	140.37 ± 0.27	1.4634 ± 0.0003
OCP09	62.58 ± 0.51	0.53 ± 0.16	0.33 ± 0.04	142.74 ± 0.35	1.4642 ± 0.0003
OCUAm03	53.27 ± 0.37	0.29 ± 0.11	0.24 ± 0.03	118.13 ± 0.12	1.4617 ± 0.0002
OCUAm06	60.29 ± 0.31	0.37 ± 0.08	0.31 ± 0.02	121.09 ± 0.18	1.4621 ± 0.0003
OCUAm09	66.71 ± 0.39	0.42 ± 0.08	0.35 ± 0.02	126.17 ± 0.21	1.4623 ± 0.0003
OCUAq03	58.44 ± 0.35	0.38 ± 0.12	0.37 ± 0.02	122.13 ± 0.28	1.4618 ± 0.0003
OCUAq06	64.21 ± 0.28	0.41 ± 0.09	0.41 ± 0.02	126.42 ± 0.32	1.4624 ± 0.0004
OCUAq09	68.23 ± 0.31	0.44 ± 0.06	0.53 ± 0.03	129.25 ± 0.35	1.4631 ± 0.0006

deterioration.[35] The IV showed higher values in the OCS samples, especially in the sample with the longest extraction time (OCS09-145.18 g I *per* 100 g oil) and the lowest values were obtained by extraction using the ultrasound at room temperature with lower extraction time (OCUAm03-118.13 g I *per* 100 g oil). The OCUAq samples presented lower IV than the OCP samples, when comparing the same extraction times, which shows that the ultrasound extraction with heating was more effective in the extraction of saturated fatty acids. The VI values obtained in this work are similar with some values found for palm, soybean, sunflower and corn oils.[36,37] The values of IR were not influenced by the different extraction methods ($p > 0.05$), since the values varied between 1.4673 and 1.4682 and are values found for oils of other seeds available in the literature.[38]

Composition of the fatty acids of the oils of cagaite seeds

Initially, a research was done in the literature to find previous works in which the content of fatty acids present in the oils of the cagaite seeds was investigated. Martinotto *et al.*[4] studied cagaite fruits and found the contents of 10.5 and 11.86% for linoleic acid and linolenic acid, respectively. Jorge *et al*.[5] studied the fatty acid profile of dehydrated and crushed seeds of cagaite using ethyl alcohol as an extractor, obtaining 37.66% of saturated fatty acids and 62.34% of unsaturated fatty acids. Camilo *et al*.[6] carried out an investigation of the variation of the fatty acid components of the cagaite seed oil, gathering 440 samples of different plant seedlings and obtained oils with 27% of saturated fatty acids and 73% of unsaturated acids, with no significant variation between the analyzed samples.

In this work, three different oil extraction methods were performed and in each method, the extraction time was further varied. The fatty acid profile obtained and its proportions in the samples were investigated and are presented in Table 3.

The samples obtained by the three extraction methods had a significant change in the amount of acid present in each oil. The lowest values of fatty acids were obtained by pressing, being 74.18% for OCP03 and 80.09 and 82.37% for samples OCP06 and OCP09, respectively. The extraction using Soxhlet had the highest levels of fatty acids with 98.62% in the OCS09 sample.

The three fatty acids present in higher quantities, C18:2 (linoleic acid), C18:1 (oleic acid) and C16:0 (palmitic acid), were also found by Jorge *et al*.[5] and Camilo *et al*.,[6] but the percentages were different, and factors such as genotype, growth condition, extraction method, among others may be the causes for these diferences.[39] The rate of saturated fatty acids varied from 19.46% (OCP03) to 31.18% (OCS09) while the amount of unsaturated fatty acids ranged from 54.72% (OCP03) to 67.64% (OCS09). The levels of linoleic and linolenic acid, important in the diet, varied between 32.22-38.11% and 5.55-8.78%, respectively.

The OCUAq samples presented smaller amounts of fatty acids than those found in the OCS samples, but still, samples with good amounts of fatty acids were obtained. Comparing the samples obtained at room temperature, OCUAm samples (OCUAm03-79.05 ± 0.24%) had a higher yield of fatty acids than the samples obtained by cold pressing

Table 3. Composition of fatty acids of cagaite seed oils extracted by different extraction methods

Fatty acids	Composition of fatty acids / %											
	OCS03	OCS06	OCS09	OCP03	OCP06	OCP09	OCUAm03	OCUAm06	OCUAm09	OCUAq03	OCUAq06	OCUAq09
C10:0	2.89 ± 0.02	3.02 ± 0.01	3.09 ± 0.04	2.13 ± 0.02	2.38 ± 0.01	2.43 ± 0.03	2.47 ± 0.01	2.71 ± 0.03	2.97 ± 0.02	2.54 ± 0.02	2.82 ± 0.02	3.01 ± 0.01
C16:0	16.01 ± 0.01	17.89 ± 0.02	18.93 ± 0.02	11.67 ± 0.02	12.79 ± 0.05	13.33 ± 0.04	12.36 ± 0.01	14.65 ± 0.03	15.03 ± 0.06	14.14 ± 0.03	15.58 ± 0.02	16.38 ± 0.05
C16:1	0.98 ± 0.01	1.06 ± 0.01	1.13 ± 0.02	0.91 ± 0.01	0.98 ± 0.02	1.11 ± 0.02	0.86 ± 0.02	0.88 ± 0.02	0.95 ± 0.02	0.89 ± 0.02	0.96 ± 0.02	1.02 ± 0.03
C18:0	1.76 ± 0.02	1.92 ± 0.01	2.06 ± 0.01	1.02 ± 0.02	1.26 ± 0.04	1.33 ± 0.02	1.18 ± 0.02	1.27 ± 0.04	1.52 ± 0.03	1.25 ± 0.01	1.36 ± 0.04	1.58 ± 0.02
C18:1	18.98 ± 0.01	19.54 ± 0.02	19.62 ± 0.01	16.04 ± 0.02	17.76 ± 0.01	18.37 ± 0.04	15.68 ± 0.04	16.21 ± 0.03	18.19 ± 0.02	1.89 ± 0.04	17.88 ± 0.04	18.42 ± 0.02
C18:2	36.16 ± 0.02	38.01 ± 0.05	38.11 ± 0.02	32.22 ± 0.01	33.93 ± 0.02	34.21 ± 0.06	34.51 ± 0.02	34.66 ± 0.01	35.08 ± 0.04	34.76 ± 0.01	35.34 ± 0.02	36.02 ± 0.01
C18:3	8.64 ± 0.01	8.72 ± 0.02	8.78 ± 0.02	5.55 ± 0.02	5.68 ± 0.03	5.91 ± 0.01	7.15 ± 0.04	7.42 ± 0.05	7.75 ± 0.03	7.43 ± 0.02	7.65 ± 0.01	7.88 ± 0.01
C20:0	1.52 ± 0.02	1.73 ± 0.03	1.78 ± 0.01	1.08 ± 0.01	1.25 ± 0.03	1.36 ± 0.02	1.09 ± 0.02	1.17 ± 0.02	1.25 ± 0.02	1.13 ± 0.04	1.26 ± 0.03	1.38 ± 0.01
C24:0	2.38 ± 0.02	2.54 ± 0.01	2.63 ± 0.01	1.53 ± 0.02	1.88 ± 0.01	2.09 ± 0.02	1.74 ± 0.04	2.02 ± 0.05	2.43 ± 0.03	1.98 ± 0.05	2.22 ± 0.03	2.51 ± 0.03
C22:0	2.48 ± 0.03	2.66 ± 0.03	2.69 ± 0.01	2.03 ± 0.04	2.19 ± 0.03	2.23 ± 0.03	2.01 ± 0.02	2.11 ± 0.03	2.33 ± 0.04	2.03 ± 0.02	2.28 ± 0.02	2.44 ± 0.02
SFA	27.04 ± 0.12	29.76 ± 0.11	31.18 ± 0.10	19.46 ± 0.13	21.75 ± 0.17	22.77 ± 0.16	20.85 ± 0.12	23.93 ± 0.20	25.53 ± 0.20	23.07± 0.17	25.52 ± 0.16	27.30 ± 0.14
MFA	19.96 ± 0.02	20.60 ± 0.03	20.75 ± 0.03	16.95 ± 0.03	18.74 ± 0.03	19.48 ± 0.06	16.54 ± 0.06	17.09 ± 0.05	19.14 ± 0.04	17.78 ± 0.06	18.84 ± 0.06	19.44 ± 0.05
PFA	44.80 ± 0.03	46.73 ± 0.07	46.89 ± 0.04	37.77 ± 0.03	39.61 ± 0.05	40.12 ± 0.07	41.66 ± 0.06	42.08 ± 0.06	42.83 ± 0.07	42.19 ± 0.03	42.99 ± 0.03	43.90 ± 0.02
UFA	64.76 ± 0.05	67.33 ± 0.10	67.64 ± 0.07	54.72 ± 0.06	58.34 ± 0.08	59.60 ± 0.13	58.20 ± 0.12	59.17 ± 0.11	61.97 ± 0.11	59.97 ± 0.09	61.83 ± 0.09	63.34 ± 0.07
TFA	91.80 ± 0.17	97.09 ± 0.21	98.62 ± 0.17	74.18 ± 0.19	80.09 ± 0.25	82.37 ± 0.29	79.05 ± 0.24	83.10 ± 0.31	87.50 ± 0.31	83.04 ± 0.26	87.35 ± 0.25	90.64 ± 0.21

SFA: saturated fatty acids; MFA: monounsaturated fatty acids; PFA: polyunsaturated fatty acids; UFA: unsaturated fatty acids, TFA: total fatty acids.

(OCP03-74.18 ± 0.19%). García-Ayuso *et al.*[40] evaluated the extraction of soybean, rapeseed and sunflower oil using a microwave-assisted Soxhlet extractor and compared the results with those obtained through conventional Soxhet extraction. Szentmihályi *et al.*[41] studied various extraction methods (traditional solvent extraction with ultrasonic, microwave, sub and supercritical extraction) of oil from rose hip seeds and all extraction methods proved to be more efficient (higher yield) when compared to the traditional Soxhlet extraction. Cravotto *et al.*[42] evaluated different unconventional techniques for obtaining kiwi seed oil and all unconventional techniques evaluated (power ultrasound (US), microwaves (MWs; closed vessels) and MW-integrated Soxhlet) proved to be fast, effective and safe. Tambunan *et al.*[43] studied the effects of mechanical extraction on the physicalchemical properties of *Jatropha curcas* oil and concluded that crushing the seed, higher temperatures and preheating help to increase the extraction yield when compared to conventional extraction using Soxhlet. Samaram *et al.*[44] investigated the yield, antioxidant activity and oxidative stability of papaya seed oil obtained by ultrasound-assisted extraction (UAE) and obtained a higher recovery of papaya seed oil with the most desirable antioxidant activity and stability. Some studies[45-48] comparing different extraction methods are available in the literature with other seed oils.

Antioxidant activity, phenolic content and oxidative stability of cagaite seed oils

The antioxidant activity and the phenol content present in cagaite seeds oil through the different extraction methods are presented in the Table 4.

The values for antioxidant activity ranged from 51.78 to 72.11%. The oils obtained by pressing presented the highest percentage of antioxidant activity, as well as the highest total phenol content present in the sample, compared to the values obtained by the other extraction methods. The OCS samples showed the lowest antioxidant activity and the lowest total phenol content. Phenolic

Table 4. Antioxidant activity and phenol content present in the oils obtained from the cagaite seeds

Sample	Antioxidant activity (AA) / %	Phenolic content / (mg GAE kg^{-1})
OCS03	62.42	138.43
OCS06	60.89	135.54
OCS09	58.88	133.12
OCP03	56.32	101.67
OCP06	53.61	104.35
OCP09	51.78	100.08
OCUAm03	59.54	125.65
OCUAm06	54.33	113.18
OCUAm09	53.92	107.44
OCUAq03	72.11	140.42
OCUAq06	68.16	136.96
OCUAq09	65.22	133.50

GAE: equivalents of gallic acid.

substances, derived from the secondary metabolism of plants, constitute the most important and abundant group, occurring in plants contributing to the antioxidant and sensory properties of fruits, honey and vegetables.[49] The decrease in antioxidant activity and total phenol content in the OCUAq and OCS samples can be attributed to the extraction temperature, since phenolic compounds can be degraded when subjected to high temperatures and long extraction times, thus contributing to the significant loss of their activity antioxidant. In the OCUAm samples, the antioxidant activity and the phenol content increased as the extraction time was increased, the inverse behavior of the OCUAq samples, thus proving that there is a significant loss in the antioxidant properties of the oil samples when extracted using high temperature.

The antioxidant substances can eliminate the free radicals of the oils and prevent their oxidation.[44] Thus, to evaluate the oxidative stability, the samples were submitted to the Rancimat method in which the time in which the samples are able to withstand oxidation is evaluated, evaluating the so-called induction period (in hours). The values for the samples OCS, OCP, OCUAm and OCUAq in the three extraction times, are in the graph of Figure 1.

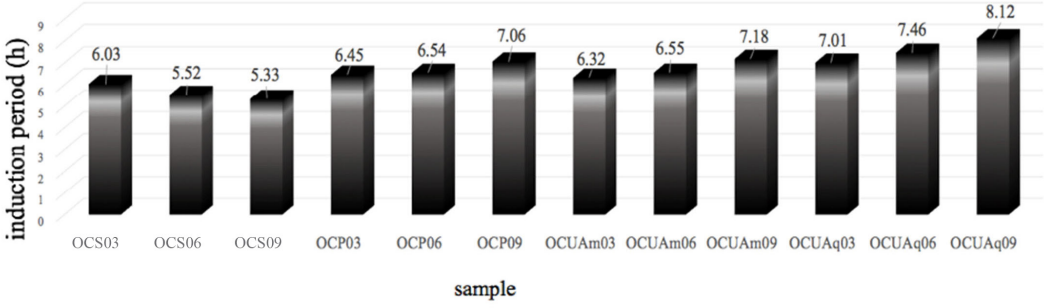

Figure 1. Induction periods (h) of the cagaite seed oils obtained by the Rancimat method.

The period of induction of the oils of the cagaite seeds submitted to the types of extraction in times of 3, 6 and 9 h varied from 5.33 h (OCS09) to 7.18 h (OCUAm09). The higher the amount of antioxidant and phenolic substances in the samples, the longer the induction period was found, thus demonstrating a positive correlation between the value of the antioxidant activity by the DPPH method, the results of the phenolic content and the Rancimat method.

Al Juhaimi *et al.*[50] obtained higher values for the antioxidant activity and the phenolic content of almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut in the cold pressing extraction when compared with the Soxhlet extraction. Some papers are available in the literature that demonstrate the resistance to oxidation of oils obtained by seeds extracted with ultrasound and other methods. Dias *et al.*[51] studied different extraction techniques (supercritical fluid extraction (SFE), Soxhlet and ultrasound-assisted extraction (UAE)) to obtain oil from umbu (*Spondias tuberosa*) seeds and evaluate yields, free fatty acid composition, total phenolic content and antioxidant activity. The highest yields were obtained by UAE with ethanol/water mixtures and by Soxhlet with ethanol and the antioxidant activity was higher in extracts obtained with polar solvents. Mohammadpour *et al.*[52] studied and compared UAE with the Soxhlet method of *Moringa peregrina* seed oil using the response surface methodology (RSM) to achieve the highest yield and evaluate the variables of the extraction process.

The OCS and OCUAq samples presented decreasing induction periods as the time of extraction of oils increased. This can be attributed to the fact that many antioxidant substances are decomposed with the action of high temperature over a period of time. In these samples, the oils obtained with longer extraction time had lower antioxidant activity and lower induction periods indicating the degradation of these antioxidant substances. The OCUAm samples showed an increase in the induction periods as the extraction time was increased, thus demonstrating that in the extraction using ultrasound with the bath at room temperature, the time contributed to the increase of antioxidant substances in the oils.

Conclusions

In this work, the fatty acid content and the physicalchemical properties of cagaite seed oil (*Eugenia dysenterica* DC) obtained by three extraction methods, Soxhlet (OCS), mechanical press extraction (OCP) and room temperature ultrasound extraction (OCUAm) and with heating ultrasound extraction (OCUAq) were studied. In all these methods, the extraction times were standardized at 3, 6 and 9 h. OCS samples showed the highest yield, followed by OCUAq. All samples showed good acidity values and the iodine value was within the limit stipulated by ANVISA. The rate of saturated fatty acids ranged from 19.46% (OCP03) to 31.18% (OCS09), while the amount of unsaturated fatty acids ranged from 54.72% (OCP03) to 67.64% (OCS09). Linoleic and linolenic acids, important in food nutrition, ranged from 32.22-38.11% and 5.55-8.78%. On the other hand, regarding the oxidative stability, the OCUAm samples presented the longest induction periods, showing a positive correlation with the antioxidant activity and the phenolic content and the efficiency of the method in the production of oil from cagaite seeds of considerable quality.

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Author Contributions

Rafael C. Rial was responsible for development of the experiments, implementation of methodologies, writing and interpretation of the results, writing original draft and writing-review and editing; Thais C. Merlo for development of the experiments, implementation of methodologies, writing and interpretation of the results; Piter H. M. Santos for development of experiments, implementation of methodologies, writing and interpretation of the results; Luiz Felipe D. Melo for development of experiments, implementation of methodologies, writing and interpretation of the results; Osmar N. de Freitas for development of experiments, implementation of methodologies, writing and interpretation of the results; Reginaldo Aparecido Barbosa for development of experiments, implementation of methodologies, writing and interpretation of the results; Carlos Eduardo D. Nazário for supervision and assistance in the interpretation of the collected data; Luiz Henrique Viana for supervision and assistance in the interpretation of the collected data.

References

1. de Almeida, S. P.; *Cerrrado Aproveitamento Alimentar*; EMBRAPA: Planaltina, 1998. [Link] accessed in November 2022
2. Naves, R. V.; de Almeida Neto, J. X.; da Rocha, M. R.; Borges, J. D.; Carvalho, G. C.; Chaves, L. J.; Silva, V. A.; *Pesqui. Agropecu. Trop.* **2007**, *25*, 107. [Link] accessed in November 2022

3. Nascimento, J. M.: *Atividade Anti-Inflamatória do Extrato Aquoso das Folhas da Eugenia dysenterica (cagaita) em Modelo Experimental de Inflamação Peritoneal em Camundongos BALB/c*; B.S. Degree, Universidade Federal de Uberlândia, Uberlândia, 2019. [Crossref] accessed in November 2022
4. Martinotto, C.; Paiva, R.; Soares, F. P.; Santos, B. R.; Nogueira, R. C.; *Cagaiteira (Eugenia dysenterica DC.*); UFLA: Lavras, 2008. [Link] accessed in November 2022
5. Jorge, N.; Luzia, D. M. M.; Bertanha, B. J.; *Rev. Chilena Nutricion* **2010**, *37*, 208. [Crossref]
6. Camilo, Y. M. V.; de Souza, E. R. B.; Naves, R. V.; Vera, R.; Vieira, M. D. C.; *Rev. Bras. Frutic*. **2016**, *38*, e-760. [Crossref]
7. Koubaa, M.; Mhemdi, H.; Vorobiev, E.; *J. Food Eng.* **2016**, *182*, 18. [Crossref]
8. Azadmard-Damirchi, S.; Habibi-Nodeh, F.; Hesari, J.; Nemati, M.; Achachlouei, B. F.; *Food Chem*. **2010**, *121*, 1211. [Crossref]
9. Yang, M.; Huang, F.; Liu, C.; Zheng, C.; Zhou, Q.; Wang, H.; *Food Bioprocess Technol*. **2013**, *6*, 3206. [Crossref]
10. Oyinlola, A.; Ojo, A.; Adekoya, L. O.; *J. Food Eng*. **2004**, *64*, 221. [Crossref]
11. Yang, G.; Zhou, X.; Li, C.; Nie, Y.; Zhang, X.; *Ind. Crops Prod*. **2013**, *50*, 190. [Crossref]
12. Martines, M. A. U.; Davolos, M. R.; Jafelicci Jr., M.; *Quim. Nova* **2000**, *23*, 251. [Crossref]
13. Adewuyi, Y. G.; *Ind. Eng. Chem. Res*. **2001**, *40*, 4681. [Crossref]
14. Wu, J.; Lin, L.; Chau, F. T.; *Ultrason. Sonochem*. **2001**, *8*, 347. [Crossref]
15. de Moraes, P. M.; Loureiro, V. R.; Padilha, P. M.; Neves, R. C. F.; Saleh, M. A. D.; dos Santos, F. A.; Silva, F. A.; *Quim. Nova* **2009**, *32*, 923. [Crossref]
16. Goula, A. M.; *J. Food Eng*. **2013**, *117*, 492. [Crossref]
17. Tian, Y.; Xu, Z.; Zheng, B.; Martin Lo, Y.; *Ultrason. Sonochem*. **2013**, *20*, 202. [Crossref]
18. Bimakr, M.; Rahman, R. A.; Taip, F. S.; Adzahan, N. M.; Islam Sarker, M. Z.; Ganjloo, A.; *Molecules* **2012**, *17*, 11748. [Crossref]
19. Samaram, S.; Mirhosseini, H.; Tan, C. P.; Ghazali, H. M.; *Molecules* **2013**, *18*, 12474. [Crossref]
20. Xhaxhiu, K.; Korpa, A.; Mele, A.; Kota, T.; *J. Essent. Oil-Bear. Plants* **2013**, *16*, 421. [Crossref]
21. da Porto, C.; Porretto, E.; Decorti, D.; *Ultrason. Sonochem*. **2013**, *20*, 1076. [Crossref]
22. Samaram, S.; Mirhosseini, H.; Tan, C. P.; Ghazali, H. M.; *Ind. Crops Prod.* **2014**, *52*, 702. [Crossref]
23. Jalili, F.; Jafari, S. M.; Emam-Djomeh, Z.; Malekjani, N.; Farzaneh, V.; *Food Anal. Methods* **2018**, *11*, 598. [Crossref]
24. ISO 5509: *Animal and Vegetable Fats And Oils-Preparation of Methyl Esters of Fatty Acids*, International Standardization Organization, Geneva, 2000.
25. Brand-Williams, W.; Cuvelier, M. E.; Berset, C.; *LWT-Food Sci. Technol*. **1995**, *28*, 25. [Crossref]
26. Liu, C.; Yang, M.; Huang, F.; *J. Am. Oil Chem. Soc*. **2012**, *89*, 73. [Crossref]
27. Tabee, E.; Azadmard-Damirchi, S.; Jägerstad, M.; Dutta, P. C.; *J. Food Compos. Anal*. **2008**, *21*, 169. [Crossref]
28. *Minitab* statistical software, 17.1; Minitab, Inc., State College, PA, USA, 2010.
29. Ixtaina, V. Y.; Martínez, M. L.; Spotorno, V.; Mateo, C. M.; Maestri, D. M.; Diehl, B. W. K.; Nolasco, S. M.; Tomás, M. C.; *J. Food Compos. Anal*. **2011**, *24*, 166. [Crossref]
30. Bhuiya, M. M. K.; Rasul, M. G.; Khan, M. M. K.; Ashwath, N.; Azad, A. K.; Mofijur, M.; *Energy Procedia* **2015**, *75*, 56. [Crossref]
31. Chielle, D. P.; Bertuol, D. A.; Meili, L.; Tanabe, E. H.; Dotto, G. L.; *Ind. Crops Prod.* **2016**, *85*, 221. [Crossref]
32. *Métodos Físico-Químicos para Análise de Alimentos*; Zenebon, O.; Pascuet, N. S.; Tiglea, P., eds.; Instituto Adolfo Lutz: São Paulo, 2008. [Link] accessed in October 2022
33. Agência Nacional de Vigilância Sanitária (ANVISA); Resolução No. 270, de 22 de setembro de 2005, *Regulamento Técnico para Óleos Vegetais, Gorduras Vegetais e Creme Vegetal*, Diário Oficial da União (DOU), Brazil, 2005. [Link] accessed in October 2022
34. Cecchi, H. M.; *Fundamentos Teóricos e Práticos em Análise de Alimentos*; Editora da Unicamp: Campinas, Brazil, 2018. [Link] accessed in October 2022
35. Moretto, E.; Fett, R.; *Tecnologia de Óleos e Gorduras Vegetais a Indústria de Alimentos*; Varela: São Paulo, 1998.
36. Azlan, A.; Prasad, K. N.; Khoo, H. E.; Abdul-Aziz, N.; Mohamad, A.; Ismail, A.; Amom, Z.; *J. Food Compos. Anal*. **2010**, *23*, 772. [Crossref]
37. Chebet, J.; Kinyanjui, T.; Cheplogoi, P. K.; *J. Sci. Innov. Res*. **2016**, *5*, 193. [Crossref]
38. Rudan-Tasič, D.; Klofutar, C.; *Acta Chim. Slov*. **1999**, *4*, 511. [Crossref]
39. Petropoulos, S. A.; Karkanis, A.; Fernandes, Â.; Barros, L.; Ferreira, I. C. F. R.; Ntatsi, G.; Petrotos, K.; Lykas, C.; Khah, E.; *Plant Foods Hum. Nutr*. **2015**, *70*, 420. [Crossref]
40. García-Ayuso, L. E.; Velasco, J.; Dobarganes, M. C.; de Castro, M. D. L.; *Chromatographia* **2000**, *52*, 103. [Crossref]
41. Szentmihályi, K.; Vinkler, P.; Lakatos, B.; Illés, V.; Then, M.; *Bioresour. Technol*. **2002**, *82*, 195. [Crossref]
42. Cravotto, G.; Bicchi, C.; Mantegna, S.; Binello, A.; Tomao, V.; Chemat, F.; *Nat. Prod. Res*. **2011**, *25*, 974. [Crossref]
43. Tambunan, A. H.; Situmorang, J. P.; Silip, J. J.; Joelianingsih, A.; Araki, T.; *Biomass Bioenergy* **2012**, *43*, 12. [Crossref]
44. Samaram, S.; Mirhosseini, H.; Tan, C. P.; Ghazali, H. M.; Bordbar, S.; Serjouie, A.; *Food Chem*. **2015**, *172*, 7. [Crossref]
45. Hernández-Santos, B.; Rodríguez-Miranda, J.; Herman-Lara, E.; Torruco-Uco, J. G.; Carmona-García, R.; Juárez-Barrientos, J. M.; Martínez-Sánchez, C. E.; *Ultrason. Sonochem*. **2016**, *31*, 429. [Crossref]

46. Khoei, M.; Chekin, F.; *Food Chem*. **2016**, *194*, 503. [Crossref]
47. Chouaibi, M.; Rezig, L.; Hamdi, S.; Ferrari, G.; *Ind. Crops Prod*. **2019**, *128*, 363. [Crossref]
48. Özcan, M. M.; Al-Juhaimi, F. Y.; Ahmed, I. A. M.; Osman, M. A.; Gassem, M. A.; *Food Meas. Charact*. **2019**, *13*, 648. [Crossref]
49. Moure, A.; Cruz, J. M.; Franco, D.; Manuel Domínguez, J.; Sineiro, J.; Domínguez, H.; Núnez, M. J.; Carlos Parajó, J.; *Food Chem*. **2001**, *72*, 145. [Crossref]
50. Al Juhaimi, F.; Özcan, M. M.; Ghafoor, K.; Babiker, E. E.; Hussain, S.; *J. Food Sci. Technol*. **2018**, *55*, 3163. [Crossref]
51. Dias, J. L.; Mazzutti, S.; de Souza, J. A. L.; Ferreira, S. R. S.; Soares, L. A. L.; Stragevitch, L.; Danielski, L.; *J. Supercrit. Fluids* **2019**, *145*, 10. [Crossref]
52. Mohammadpour, H.; Sadrameli, S. M.; Eslami, F.; Asoodeh, A.; *Ind. Crops Prod.* **2019**, *131*, 106. [Crossref]

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