

INNOVATIVE APPLICATION OF *PASSIFLORA CINCINNATA* SEED OIL FOR THE COSMETICS INDUSTRY

Lília Calheiros de Oliveira Barretto – profaliliabarretto@gmail.com

Núcleo de Graduação em Agroindústria – Universidade Federal de Sergipe

Eliziane Monteiro Santos – monteirolh@outlook.com

Núcleo de Graduação em Agroindústria – Universidade Federal de Sergipe

Carla Crislan de Souza Bery – crisberys@gmail.com

Programa de Pós-Graduação em Engenharia Química – Universidade Federal de Sergipe

Roberta Menezes Santos – menezesroberta84@gmail.com

Programa de Pós-Graduação em Química – Universidade Federal de Sergipe

Gabriel Francisco da Silva – gabriel@ufs.br

Programa de Pós-Graduação em Ciência da Propriedade Intelectual – Universidade Federal de Sergipe

Abstract— *Passiflora cincinnata* represents an alternative crop for small farmers in rainfed conditions. Its seeds oil contains high levels of polyunsaturated fatty acids as oleic, linoleic, palmitic and stearic acids. The aim of this study was to obtain and characterize the lipid fraction of passion fruit seeds cultivated in rural properties, as a proposal for development of an innovative solid fragrance. The seeds were submitted to direct solar drying process to remove moisture for 240 minutes at an average temperature of 37°C. Lipid fraction was extracted according to a cold process method using methanol, chloroform and water as solvents. Fatty acid ethyl esters were identified and quantified through gas chromatography analysis. Oxidative stability was evaluated using a Rancimat equipment and TG curves were obtained in a thermobalance analyzer. The solid perfume was produced by mixing beeswax (50%), passion fruit seed oil (31%) and Protium heptaphyllum essence (19%). The Page mathematical model presented satisfactory fitting to the experimental data, expressing R² above 98%. The extraction yield of *Passiflora cincinnata* Mast. seed oil was 13.5 %. The oleic (75.5%), linoleic (11.2%), palmitic (9.8%) and stearic acid (3.4%) were the most found fatty acids in the extracted oil. The oxidative stability period obtained in this work was 6.49 h. The TG and DTG curves of passion fruit oil showed a slight decomposition similar to other *Passiflora* oils. During storage time, no variations in the visual aspect were observed. Fragrance was apparently preserved, which indicates that the ingredients proportion applied was adequate.

Keywords — Agroindustry; oleic acid; seeds; solid perfume.

1 INTRODUCTION

Fruit farming is one of the main activities within agricultural sector at Brazilian Northeast (SOUZA et al., 2018). The yellow passion fruit production chain (*Passiflora edulis* f. *flavicarpa*), for example, is a very significant task and contributes to the socioeconomic development of that region, serving as an alternative crop mainly for family-based agriculture (DIAS et al., 2012; FALEIRO et al., 2019). Nowadays *Passiflora* commercial cultivars are well systematized and serve as reference for other species, like *P. cincinnata* Mast. (FALEIRO; JUNQUEIRA; BRAGA, 2005).

Popularly known in Brazil as ‘maracujá do mato’, ‘maracujá da caatinga’, ‘maracujá mochila’ or ‘maracujá tubarão’, this species is widely distributed in the States of Goiás, Minas Gerais and Bahia (SANTOS et al., 2017a). Since it is perennial and drought resistant, *P. cincinnata* represents an alternative crop for small farmers in rainfed conditions (SANTOS et al., 2016). This passion fruit has been used for production of juices and jellies. Also, some types are grown for ornamental or medicinal purposes (QUEIROZ, 2011). In addition, the Brazilian Agricultural Research Corporation (Embrapa) released the first *P. cincinnata* commercial variety which is adapted to semi-arid areas (BIROLO, 2016).

In juice industry, passion fruit seeds are generally disposed after being crushed, however this by-product is a great source of oil which is highlighted by considering the quantity of industrially processed fruits (MALACRIDA; JORGE, 2012; REGIS; RESENDE; ANTONIASSI, 2015). Passion fruit seed oil is composed predominantly of unsaturated fatty acids, mainly oleic and linoleic, considered of high nutritional and technological value (FERRARI; COLUSSI; AYUB, 2004; LOPES et al., 2010; MALACRIDA; JORGE, 2012). Thus, this oil can be used for many industrial purposes, being applied in cosmetics, food products, paints, soaps and drugs (DOMINGUES et al., 2014).

According to Mariana et al. (2013), there are many methods for oil extraction from oilseeds. The purpose of those extraction methods is the optimization of the process by collecting the maximum quantity of the existing oil with minimal costs. Previous studies have reported the hexane solvent extraction as a very efficient technique to recover oil from *P. cincinnata* seeds (ANDRADE et al., 2013). Due to the relation of hexane to several repercussions such as air pollution, toxicity and harmfulness, alternative options could be a promising approach to replace solvent extraction (KUMAR et al., 2017).

Passion fruit seeds from BRS Sertão Forte cultivar, developed by Embrapa, presented an lipid content of 17.2% (LIMA; ARAÚJO, BRITTO, 2018). Araújo et al. (2010) reported a 24.0% of fat content in the seeds. Those levels are relevant for the passion fruit oil industry. *P. cincinnata* oil showed a high level of linoleic acid (74.3%), also known as ω -6, that is a polyunsaturated fatty acid (PUFA) (LOPES et al., 2010; INNES; CALDER, 2018).

Cosmetics industry is an extremely important activity within the economy of many countries, including Brazil, which contributes to employment growth and reduces regional inequalities through the sustainable exploitation of different biomes, especially in the Amazon forest. Consumer market has demanded the adoption of clean, economically and environmentally sound production technologies, which require a huge and enthusiastic effort by researches and companies, in order to provide natural ingredients, besides competitive and innovative formulations (GALEMBECK; CSORDAS, 2010).

Solid perfumes are combinations of waxes, oils and fragrances concentrated in compact packaging that can simply be carried safely and with great convenience. United States Patent Office reports an invention of a solid perfume dated of 1942 (US2300769A patent number) that referred to a wax consistency stick impregnated with a fragrant essence employed for personal use. More recent data (US7723284B2 patent number; 2010) describes a solid perfumed preparation with high persistency in the form of microbeads which can be used in cosmetics and in household product industries.

Despite being a product already known by ancient history, solid perfumes are still considered as innovative products for many people. Thereby, familiar agroindustry could apply this technology to produce different fragrances produced from tropical Brazilian biodiversity.

In order to provide more support for scientific research about *Passiflora cincinnata* Mast. species, the aim of this study was to obtain and characterize the lipid fraction of passion fruit seeds cultivated in rural properties in the State of Sergipe, as a proposal for development of an innovative solid fragrance.

2 MATERIAL AND METHODS

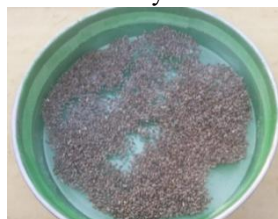
2.1 PLANT MATERIAL AND SAMPLE PREPARATION

Mature passion fruits (*Passiflora cincinnata* Mast.) were acquired from a local farm in Feira Nova, Sergipe, Brazil in February/2019. The fruits were washed and sanitized in a chlorinated water solution (100 mg.kg⁻¹ active chlorine) during 10 minutes. The seeds of passion fruits were obtained by manual pulp extraction. The seeds were placed in a sieve and washed through light friction to eliminate the arils. The batch was prepared one day before drying experiment. Beeswax was acquired from a local bee producers association. *Protium heptaphyllum* essence was purchased from Peter Paiva Company.

2.2 SOLAR DRYING EXPERIMENT

The seeds were placed in homogeneous layers on specific trays as shown in Figure 1. The seeds were submitted to a direct solar drying process to remove moisture for 240 minutes at an average temperature of 37°C and relative humidity of 50%. A digital thermo-hygrometer (ThermoPro TP50, USA) was used to measure every thirty-minute interval the ambient air temperature and humidity. Initial moisture content of the samples was determined according to AOAC official method 934.06.

Figure 1. Passion fruit seeds over tray submitted to solar drying process.



2.3 MATHEMATICAL MODEL

Drying kinetics was obtained by weighing the seeds every 30 min up to a constant weigh was reached. The moisture ratio were calculated (Eq. 1) and the curves of moisture ratio as a function of drying time were plotted (CRANK, 1975; PARK; YADO; BROD, 2001).

$$Y = \frac{X - X_{eq}}{X_0 - X_{eq}} \quad Eq.1$$

where:

Y - moisture ratio of the product, dimensionless;

X - moisture content of the product, dry basis;

X₀ - initial moisture content of the product, dry basis; and;

X_{eq} - equilibrium moisture content of the product, dry basis.

Experimental data were adjusted to the mathematical models used in different works as described in Table I (SANTOS et al., 2018).

TABLE I
MATHEMATICAL MODELS USED TO PREDICT DRYING OF AGRICULTURAL SEEDS IN THIN LAYERS

Model	Name
$Y = a \exp(-k t)$	Henderson and Pabis (Henderson; Pabis, 1961)
$Y = a \exp(-k t) + b$	Logarithmic (Yagcioglu et al., 1999)
$Y = \exp(-k t)$	Newton (Lewis, 1921)
$Y = \exp(-k t^n)$	Page (Page, 1949)
$Y = \exp[-a - (a^2 + 4 b t)^{0.5} / (2 b)]$	Thompson (Thompson et al., 1968)
$Y = a \exp(-k t) + b \exp(-k_1 t)$	Two Terms (Henderson, 1974)
$Y = a \exp(-k t) + (1-a) \exp(-k a t)$	Two Terms Exponential (Sharaf-Eldeen et al., 1980)
$Y = a \exp(-k.t) + (1 - a) \exp(-b t)$	Verma (Verma et al., 1985)
$Y = 1 + (a t) + (b t^2)$	Wang and Singh (Wang e Singh, 1978)

In which: Y – moisture content ratio of the seeds, dimensionless; t - drying time, h; k - coefficient of drying; “a”, “b”, “c”, “d” and “m” - constants of the models.

2.4 OIL EXTRACTION

In order to extract the lipid fraction from dried passion fruit seeds, the Bligh & Dyer method (1959) was used. In this method, the sample is homogenized together with methanol, chloroform and water to obtain a

single phase. Subsequently, a new amount of chloroform is added, causing a separation of two phases, one more hydrophilic and the other, lipophilic. The lipids are dissolved in chloroform, which can be removed by evaporation (BRUM; ARRUDA; REGITANO-D'ARCE, 2009). This technique was adapted in the laboratory with the substitution for similar polarity solvents to those applied at the original method, as described below.

10 g of dried seeds were weighed into a 250 ml Erlenmeyer flask where 50 ml of cereal alcohol were added in place of methanol and 25 ml of hexane were added instead of chloroform. 10 mL of distilled water was added to the erlenmeyer flask, which was capped with cotton swab. The mixture was homogenized for 2 minutes and 10 mL of hexane was added. The mixture was further stirred for 1 minute and then filtered using quantitative filter paper. 10 mL of hexane was added to the tissue residue (filter) and stirred for 20 minutes. Then 25 mL of hexane and 25 mL of 1.5% anhydrous sulfate solution were added. The mixture was stirred for a further 2 minutes. The solution with the sample was transferred to a separatory funnel to obtain the lipid fraction

After separation, the hexane phase was subjected to rotaevaporation (SL-126 vacuum rotary evaporator, Solab) at a temperature of 70°C and pressure of 700 mmHg. The oil was stored in amber glass under refrigeration ($5 \pm 1^\circ\text{C}$) prior to physicochemical analyzes.

2.5 EXTRACTION YIELD

The yield of the seeds drying process was calculated by relating the percentage obtained in relation to the dry seed in relation to the wet total mass.

2.6 CHARACTERIZATION OF *P. CININNATA* SEED OIL

Gas chromatography analysis

Stock solutions (1 mg.mL^{-1}) of the five ethyl esters (ethyl palmitoleate, ethyl palmitate, ethyl oleate, ethyl stearate, ethyl linolate) from Sigma-Aldrich Co. (St. Louis, MO). The analysis of fatty acid ethyl esters (FAEE) was performed following the EN14103:2003 method in which methyl heptadecanoate was used as an internal standard. Sample and standard were diluted in *n*-heptane. The composition of fatty esters was calculated according to the area of the peaks. The identities of the fatty esters were determined based on the retention time of methyl heptadecanoate. The result for the fatty acid methyl ester content was expressed as a mass fraction in percent using methyl heptadecanoate (FERNANDES et al., 2012).

The fatty ester composition of *P. cinnata* oil was determined by a gas chromatograph-mass spectrometer model QP2010 Plus (Shimadzu, Kyoto, Japan) with a ZB-5MS capillary column (60 m x 0.25 mm x 0.25 μm). Helium (1.0 mL.min^{-1}) was used as carrier gas. Split injection mode was used (1:50). The ion source temperature and interface temperature was 220 °C. The oven temperature was programmed as follows: 1 min at 130 °C, then 15 °C/min up to 220 °C, then 6 °C/min up to 240 °C, then 6 °C/min up to 260 °C. GC-MS spectra was also compared to commercial library data (Wiley and NIST).

Oxidative stability

The oxidative stability was evaluated using a Rancimat equipment model 873 (Metrohm, Herisau, Switzerland) with 3 g of sample under 10 L.h^{-1} air flow at 80 °C and 110 °C in accordance with EN 14112 method. The induction time was expressed in hours.

Thermal analysis

TG curves were obtained in a thermobalance analyzer model TGA-50 (Shimadzu, Kyoto, Japan), using aluminum crucibles. Measurement conditions were: average mass of the samples $10 \pm 0.5 \text{ mg}$, range of temperature from 25 to 800 °C, heating rate of $10 \text{ }^\circ\text{C.min}^{-1}$ and nitrogen flow of 50 mL.min^{-1} . The thermal stability of edible oil samples was measured as a function of initial decomposition temperature (T_{onset}) according to Santos et al. (2002).

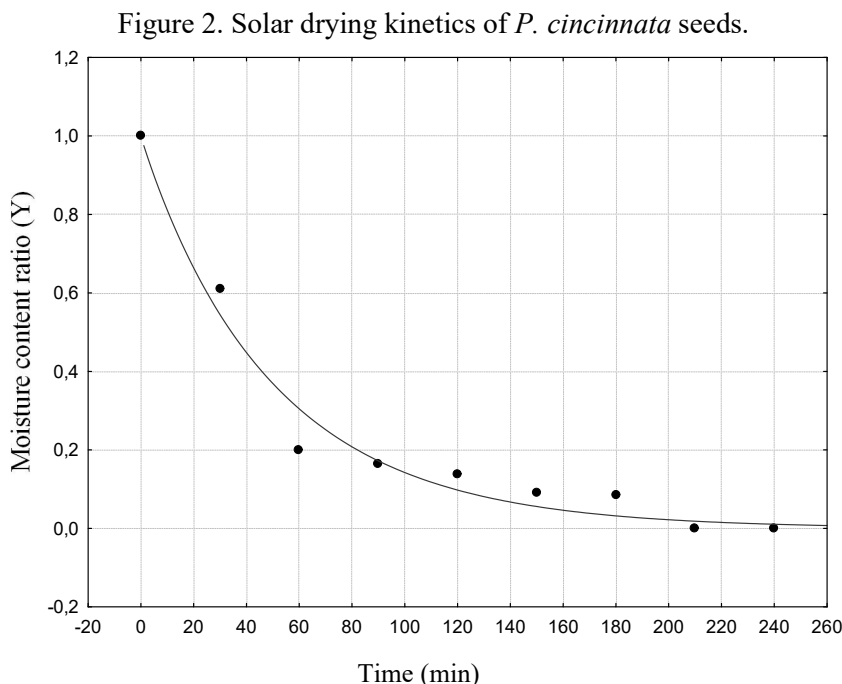
2.7 AGROINDUSTRIAL TECHNOLOGY APPLICATION OF *P. CINCINNATA* SEEDS OIL: SOLID PERFUME

Approximately 80 g of beeswax were weighed using a beaker and an analytical balance ME4002T (Mettler-Toledo, Switzerland). In an enameled pan, beeswax was added to 50 mL of *P. cinnannata* seed oil. The mixture was homogenized in a water bath at 70°C for 30 minutes. Subsequently, 30 mL of *Protium heptaphyllum* (Aubl.) essence were added to the mixture which was packed in small round styrene plastic containers (30 x 16 mm). The products were maintained at room temperature for three months when the its visual aspect was evaluated.

3 RESULTS AND DISCUSSION

3.1 DRYING KINETICS STUDY

The initial moisture content of the seeds was 15.05 (d.b.), reaching up to 2.80 (d.b.) at the end of drying. Figure 2 shows moisture ratio versus time curve for thin layer solar drying of passion fruit seeds.



The Page mathematical model presented satisfactory fitting to the experimental data, expressing R^2 above 98%. For the adjustment, a non-linear regression analysis was performed using the Quasi-Newton method, using the statistical software Statistica 7.0® (USA).

These results agree with those reported by Spoladore (2014) in which the Page model presented R^2 from 0.982 to 0.998 for the drying of passion fruit peel in thin layer. Correlation coefficient squared (R^2) and standard error (S) are crucial criteria for the accuracy of the fit to evaluate drying data. Also, a value of mean deviation relative less than 10 is related to a good fit of the model (MOHAPATRA; RAO, 2005).

According to the proposed mathematical modeling, after adjusting the experimental data, it was possible to obtain a coefficient of determination (R^2) of 0.988, with the parameter values adjusted for the Page model, respectively: $k = 0.02254$; $n = 0.9685$. The equation that describes the model is presented in Eq. 2.

$$Y = \exp(-0.02254 \times t^{0.9685}) \quad \text{Eq. 2}$$

where:

Y - moisture ratio (dimensionless);

t - drying time (minutes).

Page model also provided a mean relative error below 10% ($P = 2.47\%$) and a low estimated mean error ($SE = 0.031$). As reported by Siqueira, Resende and Chaves (2012), the smaller the SE, the better the fit of the model to experimental data.

3.2 PERCENTAGE OF *P. CINCINNATA* OIL EXTRACTED

The extraction yield of *Passiflora cincinnata* Mast. seed oil was 13.5 %, which confirms the efficiency of the extraction method. A similar yield (16.02%) was reported by Domingues et al. (2014) who extracted oil from passion fruit seeds (*Passiflora edulis*) by mechanical press. The process resulted in a yellow oil with characteristic odor and color (Figure 3).

Figure 3. *Passiflora cincinnata* seed oil.



3.3 FATTY ACID COMPOSITION

The fatty acid composition of the *P. cincinnata* seed oil is presented in Table II. The oleic (C18:1), linoleic (C18:2), palmitic (C16:0) and stearic acid (C18:0) were the most found fatty acids in the extracted oil. These results agree with those reported by Nyanzi, Carstensen and Schwack (2005), however oleic acid was the most dominant fatty acid presented in *P. cincinnata* seed oil.

TABLE II
FATTY ACID COMPOSITION OF PASSIFLORA CINCINNATA SEED OIL

Fatty acid	Values ¹ (%)
Laurate (C12:0)	N/D
Palmitoleic (C16:1)	N/D
Palmitic (C16:0)	9.85 ± 0.02
Oleic (C18:1)	75.48 ± 0.06
Linoleic (C18:2)	11.25 ± 0.02
Stearic (C18:0)	3.42 ± 0.01

¹Mean value ± standard error (n = 3).

The amount and composition of unsaturated fatty acids is determinant for the quality of edible vegetable oils (MALACRIDA; JORGE, 2012). According to Vieira (2006), the fatty acids that make up the passion fruit seeds oil do not differ qualitatively among the evaluated species, but their relative proportion can be different. The relative proportion of the major fatty acids (linoleic, oleic and palmitic) were similar for *Passiflora edulis* (67.99 % linoleic; 14.54 % oleic and 15.30 % palmitic); and for *Passiflora alata* (63.16 % linoleic; 15.02 % oleic and 18.75 % palmitic) and divergent from *Passiflora nitida* (35.53 % linoleic; 28.35 % oleic and 28.97 % palmitic).

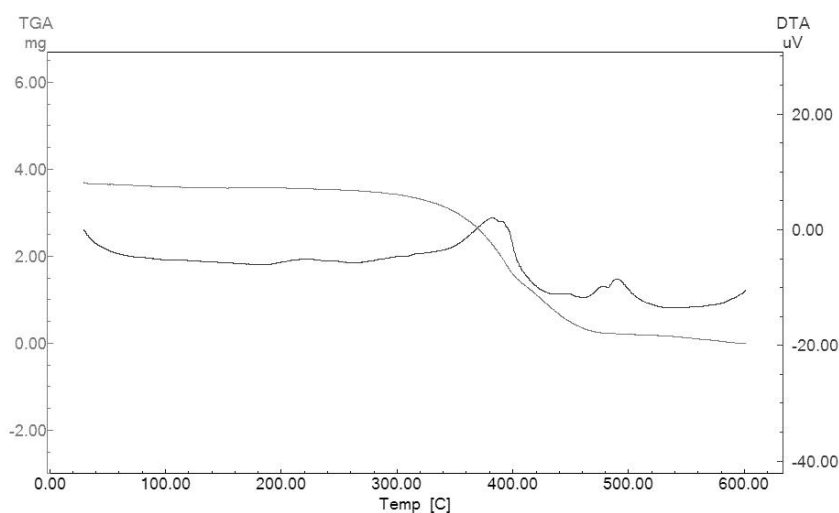
3.4 OXIDATIVE STABILITY

Rancimat procedure generates data corresponding to the initial part of the oxidation reaction (FOCKE; WESTHUIZEN; OOSTHUYSEN, 2016). The oxidative stability obtained in this work (6.49 h) was relatively similar to values reported by Malacrida and Jorge (2012) in *Passiflora edulis* oil (7.89 h). The *P. cincinnata* oil stability could be related to its chemical composition, primarily by the presence of oleic acid (75.48 %), which may have minimized the oxidation reaction. Lower stability periods were reported as 4.17 h (FERRARI; HECK; SILVA, 2006) and 5.29 h (CONCEIÇÃO; COUCEIRO; CHAAR, 2015) for *P. edulis* seed oil

3.5 THERMAL STABILITY

The thermogravimetric curve (Figure 4) shows the decomposition profile of the passion fruit oil. TG and DTG analyzes were used to investigate the thermal stability of passion fruit oil under ambient atmosphere. The TG and DTG curves of passion fruit oil showed a slight decomposition around 200°C and two distinct decomposition regions. The first one between 300 and 376°C probably corresponds to the oxidation of unsaturated fatty acids present in the oil. The second degradation region occurred with a higher decomposition rate than the first, with some unfolding, in a temperature range of 402 to 449°C of the main reaction near 425°C. This step may be associated with the decomposition of the fatty acid carbon chains. The mass loss of edible oils submitted to high temperatures is usually attributed to the thermal decomposition of components, carbonization and organic matter oxidation (FELSNER et al., 2004).

Figure 4. TG/DTG curves of *Passiflora cincinnata* seed oil.



3.6 SOLID PERFUME

The solid perfume production represents an economic and innovative alternative for Brazilian agroindustry. The formula suggested in this work resulted in a pasty and opaque product (Figure 5), which presented a characteristic aroma that could variate according to the essence applied. During storage time, no variations in the visual aspect were observed. Fragrance was apparently preserved, which indicates that the ingredients proportion applied was adequate.

Although few studies on solid perfume processing have been reported in the scientific literature, 80 patents were found at Derwent database and most of them are recent dated from the 2000s, which suggests that this type of product is still a matter of interest to the chemical industry.

Figure 5. Solid perfume produced with *Passiflora cincinnata* seed oil.



Whereas that cosmetics are high added value products, the use of regional raw materials as beeswax and passion fruit seed oil shows up as an important way of structuring the family agriculture. Also, the high quality of *P. cincinnata* seed oil contributed for maintain the product attributes, especially its aroma. In this context, future studies to qualify the product are suggested.

4 CONCLUSION

Passiflora cincinnata seed oil is presented in its composition unsaturated chain fatty acids, especially oleic acid. It is therefore suggested that it can be used in various agro-industrial segments and has potential for the development of value-added products. The oil extraction yield was 13.5%. Although low when compared to other passion fruit varieties, the quality of its chemical composition is emphasized.

The new product development process is a strong foundation for the local agribusiness growth. The use of passion fruit seed oil in a solid perfume as a way to reuse agro-industrial residues was timely, since the oil showed high thermal and oxidative stability, suggesting that the sensory characteristics of the perfume will be maintained during its lifetime.

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