



Ciência e Tecnologia de Alimentos

ISSN: 0101-2061

revista@sbcta.org.br

Sociedade Brasileira de Ciência e
Tecnologia de Alimentos
Brasil

Cosme RIBEIRO, Milton; de Barros VILAS BOAS, Eduardo Valério; RIUL, Tania Regina;
PANTOJA, Lílian; Albuquerque MARINHO, Helyde; Soares dos SANTOS, Alexandre
Influence of the extraction method and storage time on the physicochemical properties
and carotenoid levels of pequi (*Caryocar brasiliense* Camb.) oil
Ciência e Tecnologia de Alimentos, vol. 32, núm. 2, abril-junio, 2012, pp. 386-392
Sociedade Brasileira de Ciência e Tecnologia de Alimentos
Campinas, Brasil

Available in: <http://www.redalyc.org/articulo.oa?id=395940335026>

- How to cite
- Complete issue
- More information about this article
- Journal's homepage in redalyc.org

redalyc.org

Scientific Information System

Network of Scientific Journals from Latin America, the Caribbean, Spain and Portugal

Non-profit academic project, developed under the open access initiative

Influence of the extraction method and storage time on the physicochemical properties and carotenoid levels of pequi (*Caryocar brasiliense* Camb.) oil

Influência do método de extração e do tempo de armazenamento sobre as propriedades físico-químicas e o teor de carotenóides do óleo de pequi (Caryocar brasiliense Camb.)

Milton Cosme RIBEIRO^{1*}, Eduardo Valério de Barros VILAS BOAS¹, Tania Regina RIUL², Lílían PANTOJA³, Helyde Albuquerque MARINHO⁴, Alexandre Soares dos SANTOS⁵

Abstract

The objective of this study was to analyze the physicochemical properties and carotenoid levels of pequi oil obtained by different extraction methods and to evaluate the preservation of these properties and pigments during storage time. The pequi oil was obtained by solvent extraction, mechanical extraction, and hot water flotation. It was stored for over 180 days in an amber bottle at ambient conditions. Analyses for the determination of the acidity, peroxide, saponification and iodine values, coloration, total carotenoids, and β -carotene levels were conducted. The oil extraction with solvents produced the best yield and carotenoid levels. The oil obtained by mechanical extraction presented higher acidity (5.44 mg KOH.g⁻¹) and peroxide values (1.07 mEq.kg⁻¹). During the storage of pequi oil, there was an increase in the acidity and the peroxide values, darkening of the oil coloration, and a reduction of the carotenoid levels. Mechanical extraction is the less advantageous method for the conservation of the physicochemical properties and carotenoid levels in pequi oil.

Keywords: *Caryocar brasiliense*; β -carotene; vegetal oil.

Resumo

Este estudo objetivou analisar as propriedades físico-químicas e o teor de carotenóides totais do óleo de pequi (OP) obtido por diferentes métodos de extração, bem como avaliar a conservação dessas propriedades e dos pigmentos durante o armazenamento. O OP foi obtido por extração com solventes, extração mecânica e flotação com água quente, e armazenado por 180 dias em frascos âmbar sob condições ambientes. Foram realizadas análises para determinação dos índices de acidez, peróxido, saponificação e iodo, da coloração e do teor de carotenóides totais e de β -carotenos. Verificou-se que a extração com solventes promoveu um maior rendimento em óleo e o maior valor de carotenóides totais. A extração mecânica resultou em um óleo com acidez (5,44 mg KOH.g⁻¹) e índice de peróxido (1,07 mEq.kg⁻¹) elevados. Ao longo do armazenamento do OP, houve aumento da acidez e do índice de peróxido, escurecimento do óleo e redução do teor de carotenóides. A extração mecânica foi o método menos vantajoso para a conservação das propriedades físico-químicas e do teor de carotenóides no óleo de pequi.

Palavras-chave: *Caryocar brasiliense*; β -caroteno, óleo vegetal.

1 Introduction

Vegetal oils represent one of the main products extracted from plants currently utilized as food (REDA; CARNEIRO, 2007). In Brazil, due to the great biodiversity of oleaginous fruits, various studies have been carried out with the objective of analyzing the characteristics of those edible oils.

Among the vegetal oil produced from fruits found in the Brazilian Savanna region, those obtained from the pulp of pequi (*Caryocar brasiliense* Camb.) stand out, and they have currently been used in the preparation of rice, ground meat and chicken dishes, as well as in the production of sweets, preserves and liqueurs (POZO, 1997; LIMA, 2006).

Both the pequi fruit and its oil are products of great importance for the employment generation and income,

especially in the Brazilian Midwestern states (CARVALHO; SAWYER, 2007).

In popular medicine, pequi oil is considered a tonic, and it can be used in the treatment of bronchitis, influenza, colds, and manifestations related to vitamin A deficiency (BRANDÃO; LACA-BUENDÍA; MACEDO, 2002; RIBEIRO, 1996).

Recent studies have recognized the great potential of the pequi pulp oil for the production of cosmetics (PIANOVSKI et al., 2008). Furthermore, due to the low level of polyunsaturated fatty acids and high concentration of monounsaturated acid, this oil can be used in processes that involve frying and cooking (LOLOS; OREOPOULOU; TZIA, 1999; SEGALL et al., 2006).

Received 12/10/2011

Accepted 04/01/2012 (005096)

¹ Department of Food Science, Universidade Federal de Lavras – UFLA, CP 3037, CEP 37200-000, Lavras, MG, Brasil, e-mail: miltoncristeiri@gmail.com

² Department of Nutrition, Universidade Federal dos Vales do Jequitinhonha e Mucuri – UFVJM, Rua da Glória, 187, Centro, CEP 39100-000, Diamantina, MG, Brasil

³ Institute of Science and Technology, Universidade Federal dos Vales do Jequitinhonha e Mucuri – UFVJM, Rua da Glória, 187, Centro, CEP 39100-000, Diamantina, MG, Brasil

⁴ Health Sciences Research Coordination, Instituto Nacional de Pesquisa da Amazônia – INPA, Av. André Araújo, 2936, Petrópolis, CP 478, CEP 69083-000, Manaus, AM, Brasil

⁵ Department of Basic Sciences, Universidade Federal dos Vales do Jequitinhonha e Mucuri – UFVJM, Rua da Glória, 187, Centro, CEP 39100-000, Diamantina, MG, Brasil

*Corresponding author

<http://dx.doi.org/10.1590/S0101-20612012005000053>

The pequi oil is composed predominantly of oleic and palmitic acid (FACIOLI; GONÇALVES, 1998; SEGALL et al., 2006; GARCIA et al., 2007) and has, in smaller amounts, stearic, linoleic, and palmitoleic acids (FASCIOLI; GONÇALVES, 1998; AQUINO et al., 2011). Oils rich in saturated fatty acids with more than ten carbon atoms, such as the palmitic acid, and with lower levels of linoleic acid are more stable in the auto-oxidative rancidity degradation process (FETT; MORETTO, 1998).

Among the triacylglycerols present in the pequi oil are, especially, the glycerol trioleate, dioleoyl palmitoyl glycerol and the dipalmitoyl oleoyl glycerol (SEGALL et al., 2006).

Several studies have identified the pequi pulp as a source of carotenoids (RAMOS et al., 2001; AZEVEDO-MELEIRO; RODRIGUEZ-AMAYA, 2004; OLIVEIRA et al., 2006; LIMA et al., 2007) and, depending on the extraction process, some authors argue that high levels of carotenoids can also be found in the pequi oil (AQUINO et al., 2009).

The extraction method can cause changes in the physicochemical properties of the oils (REDA; CARNEIRO, 2007), which can lead to an inappropriate oil for human consumption according to oil quality standards (AGÊNCIA..., 2005). High temperatures, long thermal exposure, time of treatment, irradiation, and high oxygen concentration lead to lipidic oxidation and affect the physicochemical properties of the oils (RAMESH, 1995).

Due to the economical and nutritional importance of the pequi oil, studies on better oil extraction yields and preservation of its physicochemical properties have been conducted (AQUINO et al., 2011).

Given the above reasons, the present study aimed at determining the physicochemical properties and the total carotenoids, especially the β -carotene level, of pequi oil extracted by different methods, as well as analyzing the changes in these parameters due to storage time.

2 Material and methods

The pequi fruits used in the experiment were acquired ripe from a market in the city of Diamantina/MG in the month of January of 2009. Later, they were selected by size, uniformity, and absence of damage. After selection, they were washed, immersed in a 50 ppm sodium hypochlorite solution for 15 minutes, and stored in a freezer at $-18\text{ }^{\circ}\text{C}$ until extraction.

2.1 Extraction of pequi oil

The pequi oil was obtained by three different methods: solvent extraction, mechanical extraction, and hot water flotation.

During solvent extraction, the fruits were depulped with a knife, and the pulp was oven-dried at $60\text{ }^{\circ}\text{C}$ for 24 hours. The dry material was ground using a blender (METVISA model LQ-6), into a fine flour. 250 g of pequi flour were placed in two flat-bottomed balloon flasks containing one liter of acetone or ethyl ether. The flasks were wrapped with aluminum foil to protect them from light for about 4 hours. The extracted material

was then filtered through N° 1 Whatman filter paper and taken for solvent recovery in a Soxhlet apparatus. The extraction was repeated twice. The resulting oil was placed in an amber flask.

Mechanical extraction was based on the methodology described by Ribeiro et al. (2008). The pequi fruit was cut in half for kernel removal. Afterwards, they were taken to dry at $60\text{ }^{\circ}\text{C}$ for 24 hours. Next, they were put in an expeller press (Mini Prensa Ecirtec MPE-40) with the original configuration. The oil obtained was initially filtered in a stainless steel thin sieve at room temperature ($28\text{ }^{\circ}\text{C}$) and later filtered overnight through N° 1 Whatman filter paper at $60\text{ }^{\circ}\text{C}$.

The hot water flotation method is an old handmade practice of obtaining the pequi oil, as described by Pozo (1997). During the extraction by this method, the pequi fruits (putamen) were placed in a boiling water bath in an aluminum container for 40 minutes. Next, they were removed from the water bath and depulped using a spoon. Once cold, the pulp was boiled again in the same cooking water. As the oil drops started spreading out over the water surface, it was collected with a spoon, placed in a container, and heated until complete water evaporation.

At the end of each extraction process, the yield calculation was made by multiplying the mass of the recovered oil multiplied by one hundred grams and dividing by the dry matter weight in grams. The pequi oil extracted was maintained at room temperature in an amber flask, covered, and protected from light.

2.2 Physicochemical analyses

The fruit pulp moisture was determined according to Association of Official Analytical Chemists (ASSOCIATION..., 1990). For the characterization of pequi oil physicochemical properties, the analyses of the acidity were conducted according to the method of the Instituto Adolfo Lutz (INSTITUTO..., 2008). The peroxide and saponification values were determined according to the methodology described by the American Oil Chemists Society (AMERICAN..., 1990). The iodine value was determined according to Wijs method (AMERICAN..., 1984).

2.3 Determination of total carotenoids and β -carotene

The determination of the total carotenoids in pequi oil was conducted using hexane as solvent at 450 nm in a SP-22 Biospectro spectrophotometer. The calculations of the carotenoid levels, expressed in $\text{mg}\cdot 100\text{ g}^{-1}$, were carried out according to Higby (1962) using the following formula: total carotenoids = $(A_{450} \times 100 \times V) / (250 \times L \times W)$, where A_{450} is the absorbance value at 450 nm; V is the solvent volume; L is the length of light path expressed in centimeter; W is the sample mass (g); and ϵ (extinction coefficient) = $250\text{ L}\cdot\text{g}^{-1}\cdot\text{cm}^{-1}$.

The determination of the β -carotene was carried out as described by Rodriguez-Amaya and Kimura (1989). For this analysis, the samples were saponified with 10% KOH and separated in a dry-packed glass column (2 cm diameter \times 30 cm height) containing a mixture of Hyflo Super cel (Celite®) and MgO (2:1). Silica gel 60G coated aluminum plates

(20 × 20 × 0.25 cm) with a movable phase containing a solution of 3% methanol in benzene were used for the analytical control.

The extinction coefficient values for the calculation of the β -carotene level followed those reported by Davies (1976). All analysis steps, from obtaining the sample to the identification and quantification of the β -carotene, were conducted protected from light.

2.4 Evaluation of color

The color of the pequi oil was measured at three different graduation marks of a transparent glass flask. The coordinates were then determined by the CIE L a* b* method using a Minolta CR – 400 Colorimeter. The coordinate L indicates the coloration that goes from light (100) to dark (0); coordinate a* assumes values from –80 to +100, in which the extremes correspond to green and red, respectively, and b* assumes values that can vary from –50 (blue) to +70 (yellow).

2.5 Experimental design

The analyses of physicochemical properties, total carotenoids, and β -carotene level consisted of four treatments or extraction methods (hot water flotation, expeller press, and ethyl ether and acetone solvent extractions) and three repetitions and three times (0, 90 and 180 days of storage). However, color analysis was an exception; it was performed with ten repetitions for points corresponding to 0 and 180 days of storage.

The averages of the different treatments applied were compared by variance analysis (ANOVA) at a 5% level of significance, except for the color and carotenoid analyses. For the variables in which significant differences were detected, the Tukey test was used ($p < 0.05$).

3 Results and discussion

The pequi pulp used in this study presented 54.60% moisture, similar to the values found by Vera et al. (2007) and Aquino (2009), who identified 48.13 and 51.71% moisture in the pulps of the fruits from the states of Goiás and Minas Gerais, respectively. According to these authors, the variation in the pequi pulp moisture level can be related to climatic factors and the different varieties of those fruits found in each area.

According to the data shown in Table 1, the highest extraction yields of the pequi oil were obtained with the use of acetone (60.53%) and ethyl ether (58.47%) solvents. On the other hand, the hot water flotation method promoted the lowest yield of pequi oil extraction (19.37%) showing that this method does not remove most of the oil present in the pulp.

The results of the oil yield in the solvent extraction can be related to the moisture level because according to Aquino et al. (2009), the lower the moisture of the pequi pulp, the higher the extraction strength of non-polar solvents.

The result found in the extraction with acetone was close to that found by Aquino et al. (2011), who reported 61.07% using the same solvent. On the other hand, the oil yield obtained

in this study in the extraction with solvents was higher than those found by Segall et al. (2006), who obtained 48.73% of oil extracted from the pequi pulp with hexane, and the values found by Garcia et al. (2007), who evaluated the oil content of Brazilian Savanna fruits, *amburana* (21.5%), *baru* (37.6%), and pequi (51.9%) also extracted with hexane.

Mechanical extraction by expeller pressing did not produce oil at a high yield, probably due to the oil residue retained in the cake. In spite of that, such yield is similar to that observed in the mechanical extraction of soy and cotton oil (18 to 20%) (TURATTI, 2000).

According to Table 2, the pequi oil extraction method that used the expeller pressing was the one that resulted in a higher value of acidity (5.44 mg KOH.g⁻¹). On the other hand, the hot water flotation method presented the lowest value of this parameter (1.44 mg KOH.g⁻¹), which suggests that the oil obtained by the former method has higher free fatty acid levels resulting from the triacylglycerol hydrolysis reactions caused by the high temperatures reached during pressing (observed, but not registered).

The peroxide value was also higher in the oil obtained mechanically (1.07 mEq.kg⁻¹) demonstrating a higher vulnerability of this oil to the lipidic oxidation during the extraction process (Table 3).

Table 1. Pequi oil yield extracted by different methods.

| Extraction method | Oil level (%) |
|---------------------|---------------|
| Ethyl ether | 58.47 |
| Acetone | 60.53 |
| Expeller press | 22.41 |
| Hot water flotation | 19.37 |

Table 2. Titratable acidity analysis of pequi oil extracted by different methods and stored for 180 days.

| Parameter | Extraction method | Storage time | | |
|-----------------------------------|---------------------|--------------------|--------------------|---------------------|
| | | 0 days | 90 days | 180 days |
| Acidity (mg KOH.g ⁻¹) | Ethyl ether | 3.24 ^{aC} | 3.15 ^{aC} | 3.50 ^{aB} |
| | Acetone | 2.71 ^{aB} | 2.76 ^{aB} | 3.09 ^{aAB} |
| | Expeller press | 5.44 ^{aD} | 5.39 ^{aD} | 6.06 ^{bC} |
| | Hot water flotation | 1.44 ^{aA} | 1.71 ^{aA} | 2.92 ^{bA} |

Averages followed by the same lowercase letter on the lines and uppercase in the columns do not present a significant difference by the Tukey test ($p < 0.05$).

Table 3. Peroxide value analysis of pequi oil extracted by different methods and stored for 180 days.

| Parameter | Extraction method | Storage time | | |
|--|---------------------|---------------------|---------------------|---------------------|
| | | 0 days | 90 days | 180 days |
| Peroxide value (mEq.kg ⁻¹) | Ethyl ether | 0.752 ^{aA} | 0.754 ^{aA} | 0.938 ^{bA} |
| | Acetone | 0.725 ^{aA} | 0.706 ^{aA} | 0.884 ^{aA} |
| | Expeller press | 1.070 ^{aB} | 2.078 ^{aB} | 2.877 ^{aB} |
| | Hot water flotation | 0.760 ^{aA} | 0.938 ^{aA} | 1.024 ^{aA} |

Averages followed by the same lowercase letter on the lines and uppercase in the columns do not present a significant difference by the Tukey test ($p < 0.05$).

Significant differences in the saponification values of the oils resulting from the different extractions were not found (Table 4). This result indicates the existence of a similar amount of low molecular weight fatty acids in the oils. The same occurred for the oil iodine value, which also did not differ among the analyzed oils, a sign that they present practically equal amounts of unsaturations in the fatty acid chains.

The values of the physicochemical parameters of the pequi oil extracted with acetone can be compared to those found by Aquino et al. (2011), who found, 2.45 mg KOH.g⁻¹ acidity, 197.2 mg KOH.g⁻¹ saponification value, and an iodine value of 50.15% using the same solvent.

With regard to the hot water flotation method, the acidity and peroxide values found were different from those of Facioli and Gonçalves (1998), who found acidity value equivalent to 0.75 mg KOH.g⁻¹ and a peroxide value of 1.38 mEq.kg⁻¹ in the analysis of the pequi oil. However, they were close in terms of iodine and saponification values, 50% and 200 mg KOH.g⁻¹, respectively.

Analyzing Table 2, it can be observed that the acidity of the pequi oil increased significantly during the storage period only for the oils obtained by mechanical extraction and hot water flotation, possibly due to the occurrence of triacylglycerol hydrolysis reactions during storage.

The high level of acidity of the oil obtained by the mechanical expeller method indicates that there was higher decomposition of the oil. According to Ramesh (1995) and Reda and Carneiro (2007), such decomposition can occur through oxidation and hydrolysis reactions of the fatty acids. A fact that probably occurred due to the high temperature reached at the during mechanical oil extraction. It is worth pointing out that acidity values above 4.0 mg KOH.g⁻¹ make the oil inappropriate for human consumption (AGÊNCIA..., 2005).

One way to reduce the decomposition of the pequi oil would be by neutralization, which is a step in vegetal oil refining process (FETT; MORETTO, 1998).

In addition, mechanical extraction was the method that resulted in a higher peroxide value (Table 3). In spite of that, the values are acceptable for human consumption according to the criterion of Agência Nacional de Vigilância Sanitária (2005), which establishes a maximum limit of 15 mEq.kg⁻¹ for vegetal oils.

According to Lima and Gonçalves (1994) and Ramesh (1995), the increase in the peroxide value demonstrates the increase of the thermal and lipidic oxidation reactions forming hydroperoxides that can affect the aroma, color, and flavor of the oils leading to oil rancification.

Oil acidity and peroxide values are the main parameters that reflect the quality of oil. Changes in those parameters verified in the pequi oil might have been caused not only by the extraction method, but also by the high amount of short chain fatty acids present in the pequi oil, which according to Garcia et al. (2007) provide lower stability to the oil.

Table 4. Saponification value analysis of pequi oil extracted by different methods and stored for 180 days.

| Parameter | Extraction method | Storage time | | |
|--|---------------------|----------------------|-----------------------|-----------------------|
| | | 0 days | 90 days | 180 days |
| Saponification value (mg KOH.g ⁻¹) | Ethyl ether | 218.66 ^{aA} | 223.50 ^{aBC} | 212.40 ^{aAB} |
| | Acetone | 217.31 ^{aA} | 214.15 ^{aAB} | 214.14 ^{aAB} |
| | Expeller press | 225.09 ^{bA} | 227.44 ^{bC} | 217.30 ^{aB} |
| | Hot water flotation | 214.36 ^{aA} | 205.42 ^{aA} | 205.64 ^{aA} |

Averages followed by the same lowercase letter on the lines and uppercase in the columns do not present a significant difference by the Tukey test ($p < 0.05$).

Table 5. Chromaticity coordinates (lightness coefficient L and coordinates a* and b*) measured during the storage of pequi oil extracted by different methods.

| Extraction method | Chromaticity values (storage time/days) | | | | | |
|---------------------|---|--------------------|--------------------|--------------------|--------------------|--------------------|
| | L (0) | L (180) | a* (0) | a* (180) | b* (0) | b* (180) |
| Ethyl Ether | 51.8 ^{ba} | 46.0 ^{aA} | 10.9 ^{bb} | 8.9 ^{aB} | 38.6 ^{ba} | 20.9 ^{aA} |
| Acetone | 55.5 ^{ba} | 47.1 ^{aA} | 10.3 ^{bb} | 9.1 ^{aB} | 43.5 ^{bb} | 23.2 ^{aB} |
| Expeller press | 53.7 ^{ba} | 44.4 ^{aA} | 8.9 ^{ba} | 7.3 ^{aA} | 42.4 ^{bb} | 20.0 ^{aA} |
| Hot water flotation | 53.6 ^{ba} | 46.9 ^{aA} | 14.1 ^{bc} | 10.7 ^{aB} | 35.4 ^{ba} | 21.6 ^{aA} |

L = value of 0 (black) to 100 (white); a* = value of -80 (green) to 100 (red); b* = value of -50 (blue) to +70 (yellow). Averages followed by the same lowercase letter on the lines and uppercase in the columns do not present a significant difference by the Tukey test ($p < 0.05$).

Among the analyzed oils, only that obtained by mechanical extraction underwent significant changes in the saponification value during the storage period (Table 4). According to Fett and Moretto (1998), this value corresponds to the amount of sodium hydroxide necessary to saponify the fatty acids resulting from the hydrolysis of 1 gram of sample. Thus, the result demonstrates that after 90 days of storage there was a reduction in the concentration of intact triacylglycerols in the oil obtained by mechanical extraction with a consequent free fatty acids increase.

The decrease in the saponification value and increase in the levels of acidity and peroxide value of the pequi oil obtained by mechanical extraction indicates the occurrence of fatty acid hydrolysis reactions during storage.

The pequi oil iodine value obtained with ethyl ether, acetone, expeller pressing, and hot water flotation did not undergo significant changes during the storage period. Since the iodine value is related to the amount of double bonds present in the sample (LIMA; GONÇALVES, 1994), those changes did not occur in the number of unsaturations of the pequi oil after 180 days of storage.

There was a reduction in the values of L, a*, and b* in the color evaluation of the pequi oil during storage no matter what extraction method was used (Table 5). Such reduction suggests darkening of the oil and loss of red coloration intensity.

The darkening of the pequi oil can be related to the fatty acids decomposition rate and the carotenoid degradation

during storage. According to Reda and Carneiro (2007), lipid decomposition can occur by hydrolysis, oxidation, and polymerization processes. However, carotenoid degradation can occur by oxidation processes or action of enzymes, such as lipoxygenase, which significantly catalyze the oxidation of the carotenoids and release of fatty acids promoting isomerizations (GODOY; RODRIGUEZ-AMAYA et al., 1998; AMAYA-FARFAN; KIMURA; RODRIGUES-AMAYA, 2008).

As observed in Figure 1 and Table 6, the pequi oil has high total carotenoid levels, and it is a predominant source of β -carotene. It can be seen in Figure 1 that the extractions that use solvents promote higher preservation of the total carotenoids. However, the levels of β -carotene found in the extractions with acetone (25.41 mg.100 g⁻¹) and ethyl ether (24.02 mg.100 g⁻¹) were very similar to those obtained by hot water flotation (25.47 mg.100 g⁻¹) and expeller pressing methods (24.02 mg.100 g⁻¹).

The higher total carotenoid levels obtained in the oils extracted with solvents, as far as everything indicates, are related to the exposure of the oil to lower temperatures in relation to the other extraction process methods. According to Godoy and Rodriguez-Amaya (1998), high temperature is one of the factors that contributed to the degradation of the carotenoids present in foods.

The carotenoid levels of the oils extracted with use of solvents in this study were higher than those found by Aquino et al. (2011), who extracted pequi oil with acetone (30.0 mg.100 g⁻¹), hexane (19.9 mg.100 g⁻¹), and ethyl alcohol (29.6 mg.100 g⁻¹).

Regardless of the extraction method used, the pequi oil total carotenoids level were higher than those found in the pulp of the fruit in the studies of Lima et al. (2007), who found the equivalent to 7.25 mg.100 g⁻¹ of carotenoids, and that of Ramos et al. (2001), who found levels of 23.11 and 15.41 mg.100 g⁻¹ of total carotenoids in the raw and cooked pulp, respectively. These carotenoid values demonstrate that the pequi oil, as well as the fruit, is an excellent carotenoid source.

The β -carotene showed to be the main carotenoid present in the pequi oil reaching more than 50% of the total of carotenoids in the oil obtained by the use of solvents and more than 90% in the oil extracted by the boiling water and expeller pressing methods. The latter finding is consistent with the results of Oliveira et al. (2006), in which β -carotene also represents 90% of the level of total carotenoids present in the pulp of the fruit.

The results of this study can be correlated with those obtained by Ramos et al. (2001), who found that β -carotene is one of the main carotenoid of both raw and cooked pequi pulp (0.93 mg.100 g⁻¹) (1.22 mg.100 g⁻¹). However, they contradict those of Azevedo-Meleiro and Rodriguez Amaya (2004), who indicate violaxanthin, lutein, and zeaxanthin as the main carotenoids of the pequi pulp demonstrating the need for further research on the identity and amount of the carotenoids present in the oil and pulp of this fruit.

When comparing the data published by Rodriguez-Amaya, Kimura and Amaya-Farfan (2008), it can be seen

Table 6. Analysis of total carotenoids of pequi oil extracted by different methods and stored for 180 days.

| Parameter | Extraction method | Storage time | | |
|---|---------------------|----------------------|---------------------|---------------------|
| | | 0 days | 90 days | 180 days |
| Total carotenoids (mg.100 g ⁻¹) | Ethyl ether | 42.66 ^{bC} | 26.78 ^{aB} | 26.63 ^{aB} |
| | Acetone | 38.95 ^{bBC} | 27.73 ^{aB} | 24.50 ^{aB} |
| | Expeller press | 24.96 ^{cA} | 17.00 ^{bA} | 8.00 ^{aA} |
| | Hot water flotation | 26.87 ^{aA} | 25.04 ^{aB} | 22.24 ^{aB} |

Averages followed by the same lowercase letter on the lines and uppercase in the columns do not present a significant difference by the Tukey test ($p < 0.05$).

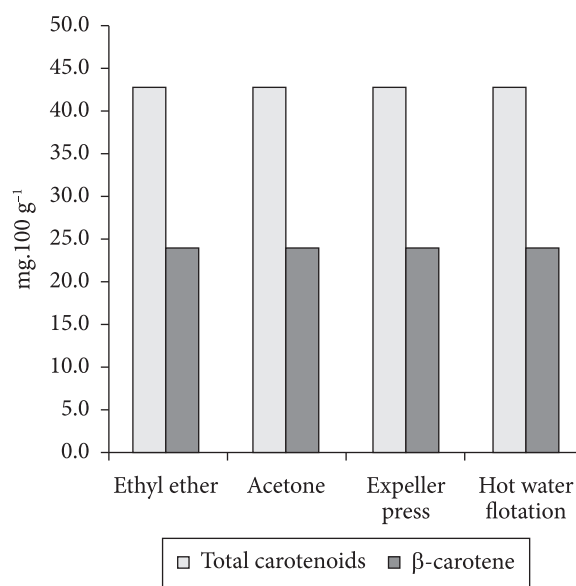


Figure 1. Results found in the analysis of total carotenoids and β -carotene of the pequi oil extracted by different methods.

that the pequi oil has higher content of β -carotene than that of mango (*Mangifera indica*) (1.80 mg.100 g⁻¹) and close to that of pupunha (*Bactris gasipaes*) (26.4 mg.100 g⁻¹). On the other hand, this content is lower than that of acerola (*Malpighia glabra*) (31.2 mg.100 g⁻¹) and raw carrot (*Daucus carota*) (40.8 mg.100 g⁻¹).

All the oils presented a decrease in the total carotenoid levels during storage, with exception of the oil obtained by hot water flotation. This indicates that the carotenoids of the oil extracted by cooking demonstrate higher stability after 180 days of storage at room temperature although the oil extracted with solvents did maintain carotenoid stability between 90 and 180 days of storage.

The pequi oil obtained by mechanical extraction was that which presented a higher decrease in the carotenoid levels during the 180 days of storage, probably due to the higher degradation of the oil during the extraction and oxidation processes. According to Amaya-Farfan, Kimura and Rodrigues-Amaya (2008), cutting and grounding foods lead to the release of enzymes that catalyze the carotenoid oxidation and an increase in the exposure of the carotenoids to oxygen as well.

4 Conclusions

In view of the results, due to its highest acidity and peroxide values, expeller pressing is the most harmful method of extraction for the pequi oil quality because of the higher degradation of the oil during extraction.

The oil obtained by mechanical extraction presents shorter shelf life, and therefore is inappropriate for human consumption due to high acidity after 180 days of storage.

The pequi oil presented darkening and reduction of the intensity of the red coloration after 180 days of storage. The loss of total carotenoids during this period is one of the reasons that led to the color change.

The use of ethyl ether and acetone in the pequi oil extraction guarantees a higher oil yield and the preservation of the carotenoids contents. However, the carotenoid content of the oil obtained by hot water flotation presented higher stability during storage.

The pequi oil is a food rich in carotenoids, especially β -carotene.

Acknowledgements

The authors are grateful for the financial support provided by the The National Council for Scientific and Technological Development (Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq), The State of Minas Gerais Research Foundation (Fundação de Amparo à Pesquisa do Estado de Minas Gerais – FAPEMIG), and Brazilian research supporting foundation (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – CAPES).

References

- AGÊNCIA NACIONAL DE VIGILÂNCIA SANITÁRIA - ANVISA. Resolução RDC nº 270, de 22 de setembro de 2005. Dispõe sobre o regulamento técnico para óleos vegetais, gorduras vegetais e creme vegetal. **Diário Oficial da República Federativa do Brasil**, Brasília, 23 set. 2005.
- AMAYA-FARFAN, J.; KIMURA, M.; RODRIGUES-AMAYA, D. B. **Fontes brasileiras de carotenóides**: tabela brasileira de composição de carotenóides em alimentos. Brasília: MMA/SBF, 2008.
- AMERICAN OIL CHEMISTS' SOCIETY - AOCS. **Official methods and recommended practices of the American Oil Chemists' Society**. 4. ed. Champaign: AOCS, 1990.
- AQUINO, L. P. et al. Influência da secagem do pequi (*Caryocar brasiliense* Camb) na qualidade do óleo extraído. **Ciência e Tecnologia de Alimentos**, v. 29, n. 2, p. 354-357, 2009. <http://dx.doi.org/10.1590/S0101-20612009000200018>
- AQUINO, L. P. et al. Extraction of oil from pequi fruit (*Caryocar brasiliense*, Camb.) using several solvents and their mixtures. **Grasas y Aceites**, v. 62, n. 3, p. 245-252, 2011. <http://dx.doi.org/10.3989/gya.091010>
- ASSOCIATION OF THE OFFICIAL ANALYTICAL CHEMISTS - AOAC. **Official methods of analysis of the Association of the Official Analytical Chemists**. Arlington: AOAC, 1984.
- ASSOCIATION OF THE OFFICIAL ANALYTICAL CHEMISTS - AOAC. **Official methods of analysis of the Association of the Official Analytical Chemists**. Arlington: AOAC, 1990.
- AZEVEDO-MELEIRO, C. H.; RODRIGUEZ-AMAYA, D. B. Confirmation of the identity of the carotenoids of tropical fruits by HPLC-DAD and HPLC-MS. **Journal of Food Composition and Analysis**, v. 17, p. 385-396, 2004. <http://dx.doi.org/10.1016/j.jfca.2004.02.004>
- BRANDÃO, M.; LACA-BUENDÍA, J. P.; MACEDO, J. F. **Árvores nativas e exóticas do Estado de Minas Gerais**. Belo Horizonte: EPAMIG, 2002.
- CARVALHO, I. S. H.; SAWYER, D. R. Potenciais e limitações do uso da biodiversidade do cerrado: um estudo de caso da Cooperativa Grande Sertão no Norte de Minas. **Revista Brasileira de Agroecologia**, v. 2, p. 1449-1452, 2007.
- DAVIES, B. H. Carotenoids. In: GOODWIN, T. W. **Chemistry and biochemistry of plant pigments**. 2nd ed. London: Academic, 1976. v. 2, p. 38-165.
- FACIOLI, N. L.; GONÇALVES, L. A. G. Modificação por via enzimática da composição triglicéridica do óleo de piqui (*Caryocar brasiliense* Camb.). **Química Nova**, v. 21, n. 1, p. 16-19, 1998.
- FETT, E.; MORETTO, E. **Tecnologia de óleos e gorduras vegetais na indústria de alimentos**. São Paulo: Varela, 1998.
- GARCIA, C. C. et al. Thermal stability studies of some cerrado plant oils. **Journal of Thermal Analysis and Calorimetry**, v. 87, n. 3, p. 645-648, 2007. <http://dx.doi.org/10.1007/s10973-006-7769-x>
- GODOY, H. T.; RODRIGUEZ-AMAYA, D. B. Carotenoid composition of Brazilian nectarine (*Prunus pérsica*). **Revista do Instituto Adolfo Lutz**, v. 1, n. 57, p. 73-79, 1998.
- HIGBY, W. K. A simplified method for determination of some the carotenoid distribution in natura and carotene: fortified orange juice. **Journal Food Science**, v. 27, p. 42-49, 1962.
- INSTITUTO ADOLFO LUTZ - IAL. **Métodos físico-químicos para análise de alimentos**. 4. ed. São Paulo: Instituto Adolfo Lutz, 2008.
- LIMA, A. et al. Composição química e compostos bioativos presentes na polpa e na amêndoa do pequi (*Caryocar brasiliense* Camb.). **Revista Brasileira de Fruticultura**, v. 29, n. 3, p. 695-698, 2007.
- LIMA, A. Ouro do cerrado: processamento industrial da polpa de pequi garante renda a agricultores na entressafra. **Revista Minas faz Ciência**, n. 27, 34-42, 2006.
- LIMA, J.; GONÇALVES, L. A. G. Parâmetros de avaliação da qualidade de óleo de soja utilizado para fritura. **Química Nova**, v. 17, n. 5, p. 392-296, 1994.
- LOLOS, M.; OREOPOULOU, V.; TZIA, C. Oxidative stability of potato chips: effect of frying oil type, temperature and antioxidants. **Journal of the Science of Food and Agriculture**, v. 79, n. 11, p. 1524-1528, 1999. [http://dx.doi.org/10.1002/\(SICI\)1097-0010\(199908\)79:11%3C1524::AID-JSFA401%3E3.0.CO;2-H](http://dx.doi.org/10.1002/(SICI)1097-0010(199908)79:11%3C1524::AID-JSFA401%3E3.0.CO;2-H)
- OLIVEIRA, M. N. S. et al. Estádio de maturação dos frutos e fatores relacionados aos aspectos nutritivos e de textura da polpa de pequi (*Caryocar brasiliense* Camb.). **Revista Brasileira de Fruticultura**, v. 28, n. 3, p. 380-386, 2006. <http://dx.doi.org/10.1590/S0100-29452006000300010>
- PIANOVSKI, A. R. et al. Uso do óleo de pequi (*Caryocar brasiliensis*) em emulsões cosméticas: desenvolvimento e avaliação da estabilidade física. **Revista Brasileira de Ciências Farmacêuticas**, v. 44, n. 2, p. 249-259, 2008. <http://dx.doi.org/10.1590/S1516-93322008000200010>

- POZO, O. V. C. **O pequi (*Caryocar brasiliense*): uma alternativa para o desenvolvimento sustentável do cerrado no Norte de Minas Gerais**. 1997. 100 f. Dissertação (Mestrado em Administração Rural)-Universidade Federal de Lavras, Lavras, 1997.
- RAMESH, M. Microwave treatment of groundnut (*Arachis hypogaea*): extractability and quality of oil and its relation to lipase and lipoxygenase activity. **Lebensmittel Wissenschaft und Technologie**, v. 28, n. 1, p. 96-99, 1995. [http://dx.doi.org/10.1016/S0023-6438\(95\)80018-2](http://dx.doi.org/10.1016/S0023-6438(95)80018-2)
- RAMOS, M. I. L. et al. Efeito do cozimento convencional sobre os carotenóides próvitamínicos "A" da polpa do piqui (*Caryocar brasiliense* Camb.). **Boletim CEPPA**, v. 19, n. 1, p. 23-32, 2001.
- REDA, S. Y.; CARNEIRO, P. B. Óleos e gorduras: aplicações e implicações. **Revista Analytica**, n. 27, p. 60-67, 2007.
- RIBEIRO, A. E. O espaço, o homem e o seu destino no norte de Minas. In: UNIVERSIDADE FEDERAL DE LAVRAS - UFLA. Departamento de Administração e Economia. **Manejo sustentado do cerrado para uso múltiplo**: subprojeto agroecologia e desenvolvimento. Lavras: UFLA, 1996.
- RIBEIRO, M. C. et al. Uso de miniprensa na extração do óleo de pequi e análise da composição centesimal da torta. In: CONGRESSO BRASILEIRO DE PLANTAS OLEAGINOSAS, ÓLEOS, GORDURAS E BIODIESEL, 5., 2008, Lavras. **Anais...** Lavras: UFLA, 2008.
- RODRIGUEZ-AMAYA, D. B.; KIMURA, M. Carotenóides e valor de vitamina A em cajá (*Spondias lutea*). **Ciência e Tecnologia de Alimentos**, v. 9, n. 2, p. 148-162, 1989.
- SEGALL, S. D. et al. Triacylglycerol analysis of pequi (*Caryocar brasiliensis* Camb.) oil electrospray and a tandem mass spectrometry. **Journal of the Science of Food and Agriculture**, v. 86, p.445-452, 2006. <http://dx.doi.org/10.1002/jsfa.2349>
- TURATTI, J. M. Óleos vegetais como fontes de alimentos funcionais. In: SIMPÓSIO SOBRE ALIMENTOS FUNCIONAIS PARA O NOVO MILÊNIO: QUALIDADE DE VIDA E SAÚDE, 2000, Campinas. **Anais...** Campinas: Unicamp, 2000. p. 12-14.
- VERA, R. et al. Caracterização física e química de frutos do pequi (*Caryocar brasiliense* Camb.) oriundos de duas regiões no Estado de Goiás, Brasil. **Revista Pesquisa Agropecuária Tropical**, v. 37, n. 2, p. 93-99, 2007.