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Processing

Pequi kernel oil extraction by hydraulic pressing and its characterization

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Abstract - The seed by-products of pequi pulp processing have a kernel in its core which is not used due to the difficulty of its extraction from the spinous endocarp. However, this kernel has high quality oil which can be used for human consumption. Thus, the kernel and the oil composition as well as the conditions to obtain the kernel oil by hydraulic pressing were evaluated in this study. The kernel showed high lipid content (55.76%), therefore being a good source for obtaining oil. The oil extraction by hydraulic pressing presented a higher yield at 5.5 tons to 6.0 tons of force and 9% to 10% moisture. Oil recovery was 75%. The pequi kernel oil showed low acid (0.17 mg KOH/g) and peroxide (1.22 mEq O_2/kg) values. The kernel oil also presented high levels of oleic acid (42.47%). The results indicate that the kernel oil extraction is an alternative form for using seeds to increase the producer/processor income and to decrease residue volumes in the pequi processing industry.

Index terms: *Caryocar coriaceum,* Lipid, Oil recovery, Oil yield.

Extração de óleo de amêndoa do Pequi por prensagem hidráulica e sua caracterização

Resumo- Os caroços, subprodutos do processamento da polpa de pequi, têm em seu núcleo uma amêndoa que não é utilizada devido à dificuldade de sua extração a partir do endocarpo espinhoso. No entanto, essa amêndoa possui óleo de alta qualidade que pode ser usado para consumo humano. Neste trabalho, avaliaram-se a composição da amêndoa e do óleo, bem como as condições de obtenção do óleo da amêndoa por prensagem hidráulica. A amêndoa apresentou alto teor lipídico (55,76%), sendo boa fonte de obtenção de óleo. A extração de óleo por prensagem hidráulica apresentou maior rendimento nas faixas de 5,5 ton a 6,0 ton de força e 9% a 10% de umidade. A recuperação de óleo foi de 75%. O óleo de amêndoa de pequi apresentou baixos valores de índice de acidez (0,17 mg KOH / g) e peróxidos (1,22 mEq O2 / kg). Além disso, o óleo de amêndoa apresentou altos níveis de ácido oleico (42,47%). Esses resultados indicam que a extração de óleo de amêndoa é uma forma alternativa de utilização de caroços para aumentar a renda do produtor / processador e para diminuir o volume de resíduos no processamento de pequi.

Termos para indexação: *Caryocar coriaceum*, lipídeo, recuperação de óleo, rendimento em óleo.

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Introduction

The pequi tree belongs to the Caryocaraceae family and the *C. coriaceum* species mainly occurs in the Mid-North of Brazil (Maranhão and Piauí States) and in the States of Ceará, Bahia, Pernambuco and Goiás (LORENZI, 1992; RAMOS; SOUZA, 2011). However, the *C. coriaceum* species has not received due attention from researchers. Few studies are available in the literature involving the production and chemical-nutritional characterization of fruits of this species (SILVA; MEDEIROS FILHO, 2006; OLIVEIRA et al., 2010a, OLIVEIRA et al. 2010b, RAMOS; SOUZA, 2011).

The edible pequi kernel is considered a by-product mainly due to the difficulty in extracting it from the spiny endocarp. The kernel can be used in oil extraction, as an ingredient in flour and sweets, or as a snack in salty or sweet form (DAMIANI et al., 2013; ALVES et al., 2014).

Ramos and Souza (2011) reported that pequi fruit seeds (*Caryocar coriaceum*) from the Maranhão and Piauí regions have an average weight of 25.9 g, and its kernels are 1.8 g. Araújo et al. (2018) reported proximate composition of pequi kernels (*Caryocar coriaceum*) as 50.0% lipids, 5.8% ash, 33.3% protein, 5.0% fiber and 5.7% carbohydrates.

Lima et al. (2007) found four major components in pequi kernels (*Caryocar brasiliense*): lipids (51.5%), proteins (25.3%), carbohydrates (8.3%) and dietary fiber (2.2 %), with a low moisture content (8.7%) and a high mineral content represented by ashes (4.0%). According to these authors, the main fatty acids of the pequi kernel are palmitic (43.76%) and oleic (43.59%).

Aguilar et al. (2011) studied the effect of a diet supplemented with pequi (*Caryocar brasiliense*) on blood lipid and glycemic levels and liver histology in mice. The authors observed that total cholesterol serum and HDL-cholesterol were significantly higher in mice fed diets containing pequi kernels at 33%. However, there was no change in blood levels of triglycerides, atherogenic fraction and glucose. Torres et al. (2016) reported that pequi kernel oil (*Caryocar brasiliense*) extracted by cold pressing attenuates liver damage induced in rats by carbon tetrachloride due to its antioxidant and anti-inflammatory properties. Oliveira et al. (2010b) also reported the topical anti-inflammatory action of pequi kernel oil (*Caryocar coriaceum*) and the accelerated recovery of skin lesions.

Therefore, the objective of this work was to evaluate the extraction of pequi (*Caryocar coriaceum*) kernel oil by hydraulic pressing and to characterize its contents. The oil obtained from the pequi kernel is an alternative use for the by-product generated in the pequi pulp industrial process.

Material and Methods

Pequi kernel

Pequi seeds (*Caryocar coriaceum*) were obtained in Chapada do Araripe, Ceará, Brazil as a residue from pulp extraction. They were washed in running water and sanitized in chlorinated solution (200 mg/L) for 15 min, followed by oven drying at 60 °C for 24 h. Kernels were obtained by cutting the seeds in halves with a guillotine and manually removing the skin. Kernels were characterized for moisture, ashes, total lipids, proteins (N x 6.25) and total carbohydrates (by difference) with the analysis determined in triplicate (INSTITUTO ADOLFO LUTZ, 2008).

Extraction of kernel oil by hydraulic pressing

As the moisture is one of the experimental design variable the pequi kernels were ground (Robot Coupe R502V.V, Vincennes, France) and dried at 110ºC for two hours in order to allow subsequent moisture adjustment. Moisture was determined (INSTITUTO ADOLFO LUTZ, 2008) in the ground kernels and water (m/m) was added to adjust the moisture according to the values of the experimental design detailed below. The samples with the added water were kept for 24h in sealed multilayer bags for moisture homogenization. For the pressing tests, 35g of samples were packed in TNT60 bags that remained in an oven for 15 min at 40 ºC for temperature stabilization. The bags were placed in a stainless steel cylinder (18 cm^2 of base area) and pressed by a piston until the test force, remaining at this force for 3 min (hydraulic press Skay, Apiguana, Fortaleza, Brazil). The yield was calculated as the ratio between the amount of oil obtained and the amount of kernels, expressed by percentage (%, m/m).

A central composite design was implemented for the tests with two independent variables (moisture and force) and three repetitions in the central point ($2^2 + 2(2) + 3$ CP), totaling 11 trials (Table 1). The yield (%, m/m) was used as the dependent variable (answer). Statistical analyses of the data were performed by ANOVA (analysis of variance) using Protimiza Software (2020) to determine the variables which had significant effects on the dependent variables at $p<0.10$.

Table 1. Values and levels used in the central composite design to evaluate pequi (*Caryocar coriaceum*) kernel oil extraction by hydraulic pressing.

Variables	Levels				
	-1.41	-1	0	+1	+1.41
Moisture (%)	2.0	3.2	6.0	8.8	10.0
Force (ton)	2.0	2.6	4.0	5.4	6.0

Pequi kernel oil analysis

Oil obtained in the best conditions determined in the experiment described previously was analyzed for acid and peroxide values (AOCS, 1988). Calculated iodine value was determined using the fatty acid composition of the oil and factors approved by AOCS (1988). The saponification value was estimated from the fatty acid composition by recalculating the average molecular weight of the fatty acids, considering that three moles of KOH are needed to saponify one mole of triacylglycerol. Fatty acid composition was determined through the methyl esters (FAMEs) obtained according to Hartmann and Lago (1973). FAMEs were determined by gas chromatography using a Shimadzu 2010 Plus device, equipped with a flame ionization detector (FID), a SP2560 column (100% bis-cyanopropyl polysiloxane; Supelco Bellefonte, USA) with dimensions of 100 m x 0.25 mm id x 0.20 µm df. The carrier gas (hydrogen) flow rate was 1.5 mL/min. The temperatures of the injection port and detector were 220 ºC and 230 ºC, respectively. The GC oven was programmed as follows: column initial temperature of 80 °C, increasing at the rate of 11.0 °C/min to 180 °C, then at 5.0 °C/min to 220 ºC and held for 9 minutes. FAMEs were identified by comparing the retention time of samples and the appropriate fatty acid methyl ester standards purchased from Supelco (Bellefonte, USA). Each fatty acid was expressed in percentages of relative area, obtained by area normalization (fatty acid peak area relative to the total area of the chromatogram).

Results and Discussion

Physico-chemical characterization of pequi kernel

The proximate composition of the pequi kernel is shown in Table 2. For pequi kernels from *Caryocar coriaceum* Ramos and Souza (2011) reported 2.4% ash, 48.5% lipids, 27.1% proteins and 21.9% of carbohydrates (on a dry basis), while Araújo et al. (2018) reported 5.8% ash, 50.0% lipids, 33.3% protein, 5.0% fiber, and 5.7% carbohydrates. Despite the variations between the results, it can be observed that the kernel is characterized by its high lipid content, therefore being a potential raw material for obtaining edible oil.

Table 2. Proximate composition of dried pequi kernel (*Caryocar coriaceum*) (n=3).

Determination	% (± sd)
Moisture	0.65 (± 0.05)
Ashes	0.28 (± 0.03)
Proteins	29.24 (± 0.74)
Lipids	55.76 (± 0.65)
Carbohydrates	14.07 (± 0.80)

Pequi kernel oil extraction by hydraulic pressing

The values obtained in the central composite design in which the influence of force and moisture on the kernel oil yield was studied are shown in Table 3. The linear and quadratic regression coefficients for force and linear regression coefficients for moisture were significant ($p<0.10$) (Table 4). The analysis of variance showed that the variation percentage explained by the model was 93.5% (Table 5). The results showed that both force and moisture influenced the oil extraction yield, and observing the contour plot (Figure 1) enables us to conclude that the optimal range for extraction is 5.5 ton to 6.0 ton of force and 9% to 10% moisture. The values were at the end of the scales, however preliminary tests showed that greater forces broke the TNT bag, thereby invalidating the process and higher moisture produces a paste which cannot be pressed. The yield was approximately 42% in relation to the pequi kernel weight, which was 75% of the total kernel oil content. The results were higher than those found by Lima et al. (2018) who reported 29% to 32% yield in the extraction of cashew kernel oil, and Navarro and Rodrigues (2016) who reported 35% to 40% yield for macadamia oil, both by pressing.

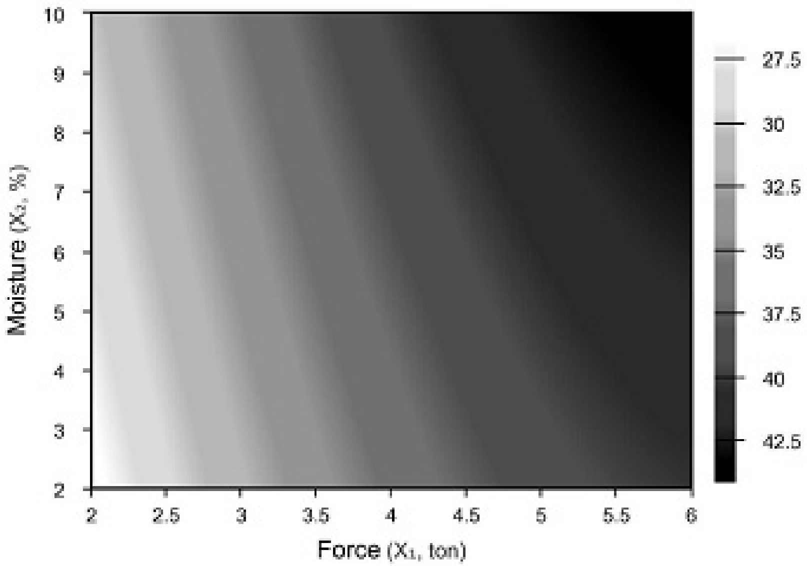

Figure 1. Contour plot for yield (%, m/m) in pequi (*Caryocar coriaceum*) kernel oil extraction by hydraulic pressing.

Table 3. Central composite design matrix and experimental results for pequi (*Caryocar coriaceum*) kernel oil extraction by hydraulic pressing.

Essay	Coded levels		Decoded levels		Yield
			Force (ton)	Moisture (%)	(%, m/m)
1	-1	-1	2.6	3.2	29.32
2	1	-1	5.4	3.2	41.24
3	-1	1	2.6	8.8	31.52
4	1	1	5.4	8.8	41.55
5	-1.41	0	2.0	6.0	29.66
6	1.41	0	6.0	6.0	42.03
7	0	-1.41	4.0	2.0	34.84
8	0	1.41	4.0	10.0	40.82
9	0	0	4.0	6.0	36.33
10	0	0	4.0	6.0	38.83
11	0	0	4.0	6.0	39.19

Table 4. Central composite design regression coefficients for yield (%, m/m) in pequi (*Caryocar coriaceum*) kernel oil extraction by hydraulic pressing.

	Regression coefficients	Standart error	calculated t	*p*-value
Mean	37.76	0.62	61.05	0.0000
x_1	4.93	0.52	9.47	0.0000
x_1^2	-1.26	0.59	-2.12	0.0713
x_2	1.37	0.52	2.63	0.0337

x_1 = Force (ton); x_2 = Moisture (%).

Table 5. ANOVA for yield (%, m/m) in pequi (*Caryocar coriaceum*) kernel oil extraction by hydraulic pressing (R^2 = 93.5%).

Variation source	Sum of squares	Degrees of freedom	Mean square	F_{calc}	*p*-valor
Regression	219.3	3	73.1	33.7	0.00016
Residuals	15.2	7	2.2		
Lack of fit	10.3	5	2.1	0.9	0.61838
Pure error	4.9	2	2.4		
Total	234.5	10			

Characteristics of the pequi kernel oil

The acid value (0.17 mg KOH/g) observed was smaller than the maximum limit allowed for cold-pressed and unrefined oils in Brazilian legislation and in Codex Alimentarius (4.0 mg KOH/g) (ANVISA, 2005; CODEX ALIMENTARIUS, 2003). Likewise, the peroxide value observed (1.22 mEq O_2/kg) was smaller than the limit in those legislations (15 mEq O_2/kg). Both results reflected good quality oil (Table 6). Calculated iodine value was lower than the values ranging from 74.2 to 113.8 g I_2/ 100g oil reported by Moodley et al. (2007) for other kernels oils (macadamia, pecan, brazil, walnut and almond), which reflects the high saturated fatty acids content of the pequi kernel oil. The saponification value was also out of the range of 182.5 to 193.7 mg KOH / g oil reported by those authors for common kernels oils.

Table 6. Chemical characteristics and fatty acid composition of pequi (*Caryocar coriaceum*) kernel oil extracted by hydraulic pressing.

Determination	mean ± sd
Acid value (mg KOH/g)	0.17 ± 0.02
Peroxide value (mEq/kg)	1.22 ± 0.02
Iodine value* (g I_2/100g)	54.13
Saponification value* (mg KOH/g)	209.65
Fatty acid (%)	
Miristic (C14:0)	0.22 ± 0.03
Palmitic (C16:0)	39.49 ± 3.03
Estearic (C18:0)	1.63 ± 0.13
Oleic (C18:1 cis)	42.47 ± 2.11
Linoleic (C18:2 cis, cis)	10.17 ± 0.55
Not identified	6.02
∑ Saturated	41.34
∑ Unsaturated	52.63

*calculated from the fatty acids average percentages

The major fatty acids in pequi kernel oil were oleic (42.47%) and palmitic (39.49%), followed by linoleic acid (10.17%) (Table 6), which agrees with the results reported by Lima et al. (2007) and Torres et al. (2016). In addition to the benefits of consuming oils with high monounsaturated fatty acids content to prevent cardiovascular diseases (ALVES et al., 2016), some authors have reported anti-inflammatory action related to the consumption of pequi kernel oil (TORRES et al., 2016), as well as its topical use (OLIVEIRA et al., 2010b).

Conclusions

Pequi kernel have 55.76% of lipids, therefore being a good source for edible oil. The optimum ranges for oil extraction by hydraulic pressing are 5.5 ton to 6.0 ton of force and 9% to 10% moisture, with oil yield of approximately 42% in relation to the pequi kernel weight, representing 75% of oil recovery. In addition, pequi kernel oil is an excellent source of monounsaturated fatty acids for human consumption. Thus, the use of this by-product from pequi pulp production can became a new income source for the producing regions.

References

AGUILAR, E.C.; QUEIROZ, M.G.M.N.; OLIVEIRA, D.A.; OLIVEIRA, N.J.F. Serum lipid profile and hepatic evaluation in mice fed diet containing pequi nut or pulp (*Caryocar brasiliense* Camb.). **Ciência e Tecnologia de Alimentos**, Campinas, v.31, n.4, p.879-883, 2011.

ALVES, A.M.; FERNANDES, D.C.; BORGES, J.F.; SOUSA, A.G.O.; NAVES, M.M.V. Oilseeds native to the Cerrado have fatty acid profile beneficial for cardiovascular health. **Revista de Nutrição**, Araraquara, v.29, n.6, p.859-866, 2016.

ALVES, A.M.; FERNANDES, D.C.; SOUSA, A.G.O.; NAVES, R.V.; NAVES, M.M.V. Características físicas e nutricionais de pequis oriundos dos estados de Tocantins, Goiás e Minas Gerais. **Brazilian Journal of Food Technology**, Campinas, v.17, n.3, p.198-203, 2014.

ANVISA - Agencia Nacional de Vigilância Sanitária. Resolução RDC n° 270 de 22 de setembro de 2005. **Regulamento técnico para óleos vegetais, gorduras vegetais e creme vegetal**. Brasília, 2005 Disponível em: http://portal.anvisa.gov.br/documents/10181/2718376/RDC_270_2005_.pdf/8f80bf4d-a38a-4699-9f8f-582186b3797d. Acesso em: 14 maio 2020.

AOCS - American Oil Chemists Society. **Official methods and reccomended practices of the American Oil Chemists Society**. 5th ed. Champaign, 2003.

CODEX ALIMENTARIUS (FAO/WHO). **Codex standard for named negetable oils, Codex Stan 210 (Amended 2003)**. Roma, 2003. Disponível em: https://mvo.nl/media/voedselveiligheid/codex_standard_named_vegetable_oils.pdf Acesso em: 19 jun. 2020.

ARAÚJO, A.C.M.A.; MENEZES, E.G.T.; TERRA, A.W.C.; DIAS, B.O.; OLIVEIRA, E.R.; QUEIROZ, F. Bioactive compounds and chemical composition of Brazilian Cerrado fruits' wastes: pequi almonds, murici, and sweet passionfruit seeds. **Food Science and Technology**, Campinas, v.38, n.1, p.203-214, 2018.

DAMIANI, C.; ALMEIDA, T.L.; COSTA, N.V.; MEDEIROS, N.X.; SILVA, A.G.M.; SILVA, F.A.; LAGE, M.E.; BECKER, F.S. Perfil de ácidos graxos e fatores antinutricionais de amêndoas de pequi crua e torrada. **Pesquisa Agropecuária Tropical**, Goiás, v.43, n.1, p.71-78, 2013.

HARTMANN, L.; LAGO, R.C.A. Rapid preparation of fatty acid methyl esters from lipids. **Laboratory Practices**, London, v.22, p.475-477, 1973.

INSTITUTO ADOLFO LUTZ. **Normas analíticas do Instituto Adolfo Lutz**: métodos físico-químicos para análise de alimentos. São Paulo, 2008. 1020 p.

LIMA, A.L.; SILVA, A.M.O.; TRINDADE, R.A.; TORRES, R.P.; MANCINI-FILHO, J. Composição química e compostos bioativos presentes na polpa e na amêndoa do pequi (*Caryocar brasiliense*, Camb.). **Revista Brasileira de Fruticultura**, Jaboticabal, v.29, n.3, p.695-698, 2007.

LIMA, J.R.; PINTO, G.A.S.; MAGALHÃES, H.C.R .**Óleo da amêndoa de castanha-de-caju**: métodos de extração. Fortaleza: Embrapa Agroindústria Tropical, 2018. (Boletim de Pesquisa e Desenvolvimento, 165).

LORENZI, H. **Árvores brasileiras**: manual de identificação e cultivo de plantas arbóreas nativas do Brasil. 4.ed. Nova Odessa: Plantarum, 1992. v.1, 368p.

MOODLEY, R., KINDNESS, A.; JONNALAGADDA, S.B. Elemental composition and chemical characteristics of five edible nuts (almond, Brazil, pecan, macadamia and walnut) consumed in Southern Africa. **Journal of Environmental Science and Health**, London, v.42, n.5, p.585-591, 2007.

NAVARRO, S.L.B.; RODRIGUES, C.E.C. Macadamia oil extraction methods and uses for the defatted meal byproduct. **Trends in Food Science & Technology**, Amsterdan, v.54, p.148-154, 2016.

OLIVEIRA, M.E.B; GUERRA, N.B.; MAIA, A.H.N; ALVES, R.E.; MATOS, N.M.S.; SAMPAIO, F.G.M.; LOPES, M.M.T. Características químicas e físico-químicas de pequis da Chapada do Araripe, Ceará. **Revista Brasileira de Fruticultura**, Jaboticabal, v.32, n.1, p.114-125, 2010a.

OLIVEIRA, M.L.M.; PINHEIRO, D.C.S.N.; TOMÉ, A.R.; MOTA, E.F.; VERDE, I.A.L.; PINHEIRO, F.G.M.; CAMPELLO, C.C.; MORAIS, S.M. In vivo topical anti-inflammatory and wound healing activities of the fixed oil of *Caryocar coriaceum* Wittm. Seeds. **Journal of Ethnopharmacology**, v.129, p.214-219, 2010b.

PROTIMIZA. **Protimiza experimental design**. 2020. Disponível em: experimental-design.protimiza.com.br. Acesso em: 12 maio 2010.

RAMOS, K.M.C.; SOUZA, V.A.B. Características físicas e químico-nutricionais de frutos de pequizeiro (*Caryocar coriaceum* wittm.) em populações naturais da região meio-norte do Brasil. **Revista Brasileira de Fruticultura**, Jaboticabal, v.33, n.2, p.500-508, 2011.

SILVA, M.A.P.; MEDEIROS FILHO, S. Morfologia de fruto, semente e plântula de pequi (*Caryocar coriaceum* Wittm.). **Revista Ciência Agronômica**, Fortaleza, v.37, n.3, p.320-325, 2006.

TORRES, L.R.O.; SANTANA, F.C.; TORRES-LEAL, F.L.; MELO, I.L.P.; YOSHIME, L.T.; MATOS-NETO, E.M.; SEELAENDER, M.C.L.; ARAÚJO, C.M.M.; COGLIATI, B.; MANCINI-FILHO; J. Pequi (*Caryocar brasiliense* Camb.) almond oil attenuates carbono tetrachloride-induced acute hepatic injury in rats: antioxidant and anti-inflammatory effects. **Food and Chemical Toxicology**, Oxford, v.97, p.205-216, 2016.