Nutritional, anti-nutritional and technological functionality of flour from Libidibia ferrea

Cristiani Viegas Brandão Grisi^[1], Angela Maria Tribuzy de Magalhães Cordeiro^[2], Andressa Samara de Carvalho Ferreira^[3], Agdylannah Felix Vieira^[4], Ana Paula Trindade Rocha^[5], Gilmar Trindade de Araújo^[6]

[1] crisgrisi.gere@gmail.com. Universidade Federal da Paraíba / Pós-graduação em Tecnologia Agroalimentar. [2] atribuzycordeiro@gmail.com. Universidade Federal da Paraíba / Pós-graduação em Tecnologia Agroalimentar. [3] andressasamaracf@gmail.com. Universidade Federal da Paraíba / Departamento de Engenharia de Alimentos. [4] agdylana@hotmail.com. Universidade Federal de Campina Grande / Pósgraduação em Engenharia Agrícola. [5] ana_trindade@yahoo.com.br. Universidade Federal de Campina Grande / Pós-graduação em Engenharia Agrícola. [6] gilmartrindade@ufcg.edu.br. Universidade Federal de Campina Grande / Pós-graduação em Engenharia Química

ABSTRACT

The purpose was to develop new ingredients for the food industry. The flours were obtained from the bark and the fruit of juca (Libidibia ferrea) by grinding and drying in an air circulation greenhouse. The flours were analyzed in terms of nutritional, anti-nutritional, antioxidant, and technological functionality propriety. The flours developed are rich in carbohydrates with values ranging from 89.29 g/100g for the fruit and 81.76 g/100g for the bark. Flours showed low water and fat absorption index, high compacted and real density, and intermediate flow values by the Hausner's ratio and Carr's index. The hygroscopicity of the flours ranged from 5.56 g/100g for the bark and 10.31 g/100g for the fruit, influencing the solubility property. The anti-nutritional compounds do not discourage the technological application of flours since studies indicate the action of tannic and phytic acids as antioxidants. The flour shows high total phenolic compound and antioxidant activity in vitro (DPPH and FRAP methods), due to the flavonoids compounds as catechin and myricetin identified by HPLC method. Therefore, fruit flour is the best one when compared to the botanical parts, and indicated as an ingredient to improve sensory characteristics such as crispness, increased sensation, and retention of food flavor.

Keywords: antioxidant. carbohydrate source. crispy food. new ingredient. water absorption.

RESUMO

O objetivo foi desenvolver um novo ingrediente para indústria de alimentos. As farinhas do fruto e da casca do jucá (Libidibia ferrea) foram obtidas por trituração e secagem em estufa. As farinhas foram analisadas quanto as propriedades nutricionais, antinutricionais e funcionalidades tecnológicas. As farinhas são ricas em carboidratos com valores de 89,29g/100g para o fruto e 81,76g/100g para a casca do jucá. As farinhas apresentaram baixa retenção de água e de gordura, elevada densidade compactada e real, e valores intermediários de fluidez pelo fator de Hauser e pelo índice de compressibilidade de Carr. A higroscopicidade variou de 5,56 g/100g para a casca e 10,31g/100g para o fruto do jucá, influenciando na propriedade de solubilidade. Os compostos antinutricionais identificados não desestimulam as aplicações tecnológicas das farinhas, visto que estudos apontam a ação dos ácidos tânico e fítico como antioxidantes. As farinhas apresentaram elevado teor de compostos fenólicos e atividade antioxidante pelos métodos in vitro (DPPH e FRAP), devido aos flavonoides, como catequina e miricetina, identificados por cromatográfica líquida. Portanto, a farinha do fruto é melhor comparada entre as partes botânicas, e indicada como ingrediente para melhorar as características sensoriais como, crocância, aumento da sensação e da retenção do sabor do alimento.

Palavras-chave: antioxidante. absorção de água. alimento crocante. fonte de carboidrato.

1 Introduction

The food industry seeks new ingredients to innovate its products, basing its studies on the adequacy of technological processes (FELKER *et al.*, 2018), resulting in significant changes in composition, structure, physicochemical behavior, or nutritional value (MEMON *et al.*, 2020). Within the scope of consumers, there is a need to develop new ingredients (LIMA *et al.*, 2019), whose functionality and versatility meet them in terms of bioactive compounds (AYESSOU *et al.*, 2014; DRAKOS *et al.*, 2017; MILLAR *et al.*, 2019; OLIVEIRA *et al.*, 2020)

Flours have been featured among these new ingredients (LIMA *et al.*, 2019; MEMON *et al.*, 2020; MILLAR *et al.*, 2019; SUMMO *et al.*, 2019; TAMSEN; SHEKARCHIZADEH; SOLTANIZADEH, 2018). The flours are defined as products obtained from edible parts of one or more species of cereals, legumes, fruits, seeds, tubers, and rhizomes by milling and/or other technological processes (BRAZIL - MINISTRY OF HEALTH, 2005). Flours are often used in many recipes as a carbohydrate source and for the texture improvement of processed foods (THIYAJAI *et al.*, 2016).

The flours are usually developed from different sources, such as corn, potato, wheat, and more recently rice (THIYAJAI *et al.*, 2016). The use of juca as an alternative to replace traditional flours in the cooking recipes need to be studied. The physiochemical properties and technological functionality aspects of partial/whole substitution of juca flour in noodles, spaghetti, biscuits, and some crispness food were not found in the literature.

The impairment of the nutritional value of natural sources as juca is attributed to the presence of different compounds usually known as anti-nutritional factors that act as direct or indirect antagonists of nutrient availability (OLGUIN *et al.*, 2003). Some of these compounds are tannic, phytic, and oxalic acids. These chemicals in plants come from their defense mechanisms, acting efficiently against fungi, bacteria, and herbicidal actions (MUNHOZ *et al.*, 2018). Due to the possible impact on the nutritional value of foods, current Brazilian legislation recommends the determination of anti-nutritional compounds for the development of new ingredients (BRAZIL – Ministry of Health, 1999).

Libidibia ferrea is commonly known as "pau ferro" or "jucá" in Brazil. There is a spontaneously growing plant from Northeast to Southeast Brazil (COSTA; GUILHON-SIMPLICIO; SOUZA, 2015; PEREIRA

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et al., 2012), it used in different areas ranging from environmental restoration to applications as medicine, due to its proven therapeutic power in ethnobotanical and phytochemical studies (BARROS *et al.*, 2014). The juca bioactive substances are found in all parts of the plant (FERREIRA; SOARES, 2015). Those substances can be extracted by infusion for use as a medicinal tea, without any critical toxic effects (CARVALHO *et al.*, 2011). Pharmacological studies have demonstrated that juca exhibits antibacterial, antiviral, anti-inflammatory, anti-tumor, antihypertensive and hypoglycemic properties (CUNHA *et al.*, 2017; DIAS *et al.*, 2013; FIGUEREDO *et al.*, 2017; NASCIMENTO *et al.*, 2015; PORT'S *et al.*, 2013).

Libidibia ferrea has potent antioxidant effects and antibacterial activity against oral pathogens (COSTA; GUILHON-SIMPLICIO; SOUZA, 2015). A highlight is rich in polar compounds of pharmacological interest, especially in their fruits and bark, parts more utilized in traditional medicine (FERREIRA; SOARES, 2015). Despite its prominence as a medicine, the literature does not report the use of juca flour as a carbohydrate source.

Thus, to contribute further to the knowledge of *L. ferrea*, our research aimed at the evaluation of physicochemical properties and technological functionality aspects of flour produced by the drying process from the fruit and the bark juca. The properties examined include chemical composition (moisture, protein, lipid, ash, carbohydrate, iron content), antinutritional compounds (tannic, phytic and oxalic acid), total phenolic compound and antioxidant activity, as well as the densities, porosity, water activity, pH, hygroscopicity, solubility, and absorption capacity.

2 Materials and methods

2.1 Materials

The fruits and bark of the juca (*Libidibia ferrea*) collected during the dry period (from December 2015 to March 2016) were purchased from the commercial centers of João Pessoa - PB, Brazil.

All the reagents used were of analytical grade. The acids were purchased from Perkin-Elmer (Sulfuric acid, boric acid, sodium hydroxide, hydrochloric acid, phytic acid, oxalic acid, and tannic acid). The petroleum ether, ethanol, iron, and sodium carbonate were purchased from NEON. The 2,2'-diphenyl-2-picrylhydrazyl (DPPH), Folin-Ciocalteau, 2,4,6-tris (2-pyridyl)-s-

triazine (TPTZ), Folin-Denis, and Wade reagents; and the reagents used HPLC grade as acetic acid, acetonitrile, methanol, phenolic acid, and flavonoid standard were purchased from Sigma–Aldrich Chemical Co. (St. Louis, MO, USA).

2.2 Juca flour preparation

The bark and the fruits of the juca were heated (40 °C) in a greenhouse with air circulation for 24h. Then crushed in Willey knife mill (SOLAB-SL31, Brazil), with the fixed rotation of 1750 RPM and 10 mesh stainless steel sieve (1,64-1,76mm Tyler) coupled to the equipment. The resulting flours were stored in plastic bags and vacuum-sealed until the analyses were performed. This product has a patent application under filing number BR 10 2019 0014210.

2.3 Proximal composition and total energy value

All analyses were carried out according to standard methods of A.O.A.C (1995). Moisture was determined by the gravimetric method, from sample weight loss after oven drying at 105 °C; the protein concentration was determined by the Kjeldahl method using a conversion factor of 5.75 for products of plant origin; the lipid concentration was obtained for Soxhlet extraction method with petroleum ether; the ash was calculated after heating the sample at 550 °C. Total carbohydrate was calculated by difference (100 - sum of protein, fat, ash, and moisture), and the reducing and non-reducing sugars were determined by the Lane-Eynon method. Results were expressed in g/100g on a dry basis. The total energy value was expressed in kilocalories (kcal) / 100g, estimated from the Atwater coefficients, using 4.0 kcal / g for carbohydrates and proteins, and 9.0 kcal/ g for lipids (MERRIL; WATT, 1973).

2.4 Iron content

Iron content was determined by the orthophenanthroline method described in NBR 13934 ABNT (1997) by reading in a spectrophotometer UV-Vis absorbance at 510nm. The results calculated by standard curve of iron (y=0.0502x - 0.0123, R² = 0.98) and expressed in mg iron/100 g dry basis sample.

2.5 Anti-nutritional compounds

Tannic acid was determined by the standard curve by the Folin-Denis method according to Rangana (1979). The tannins were extracted from samples by boiling (70°C) the samples in distilled water for 1h. The supernatant was obtained by centrifugation and mixed with Folin–Denis reagent and sodium carbonate solution after 30 minutes. Absorbance was measured spectrophotometrically at 760 nm. The result calculated by the standard curve of tannic acid (y=0.0564x+0.2268 $R^2 = 0.95$) and expressed as mg of tannic acid/100g dry sample.

The phytic acid content was determined by the methodology of Chang and Xu (2009). The phytate was extracted with HCl for 16 hours of stirring at 25 °C, then the sample was centrifuged at 1000 rpm for 20 minutes at 10 °C. Phytate content was measured at 500 nm using Wade's reagent after 10 minutes centrifugation at 5500 rpm at 10 °C and using water as white. The result calculated by the standard curve of phytic acid (y=0.0002x+0.0066 R² = 0.97) and expressed in mg of phytic acid/100g dry sample.

Oxalic acid was extracted with HCl, precipitated, and quantified by titration of calcium oxalate with potassium permanganate, according to the methodology described by Moir (1953). The result was expressed in mg of oxalic acid/100g dry sample.

2.6 Total phenolic content and antioxidant activity

The total phenolic compound (TPC) and antioxidant activity (AA) were assessed on an aqueous extract (1:10 m/v) prepared as follows: 1 g of flour was mixed with 10 mL of solvent in a centrifuge tube and stirred for 2 h in the dark. Then, the tube was centrifuged at 4400 rpm for 3 min to recover the supernatant.

The TPC of the flours was determined by Folin-Ciocalteu's method described by Rossi and Singleton (1965). 20µL of the sample was pipette onto a glass tube and 60µL with the addition of Folin-Ciocalteu reagent and 2740µL of distilled water. After 1 min, 180µL of sodium carbonate (15%) was added, the mixture for 1 min again, and left to stand for 2 hours to action. The absorbance readings of the samples and standard were measured at 760 nm. The standard curve of gallic acid at concentrations of 1 to 20 mg / L (y=0.098x+0.01 R² = 0.99) was used to calculate the results expressed in gram gallic acid equivalent per gram of sample (mg GAE / g).

2.6.1 Antioxidant activity (AA)

The AA was evaluated by the 2,2-diphenyl-1picrylhydrazyl (DPPH) radical scavenging capacity assay method described by the Brand-Williams; Cuvelier and Berset (1995). 20µL of the sample was pipette onto a glass tube and 280µL with additional ethanol. Then it was diluted with DPPH solution to 3mL of the final volume, and incubated for 30 minutes at 25 °C. The absorbance readings of the samples were measured at 517nm, with a quartz cell with 1 cm of the optical path. The DPPH• solution in ethanol was used as blank. The results were calculated with percent inhibition of the free radical DPPH • relative to the blank (%).

The AA of ferric reduction was determined by FRAP's method (RUFINO *et al.*, 2006). Under darkened condition, the FRAP reagent was prepared with a 300 mmol/L acetate buffer (pH 3.6), 2,4,6-tris (2-pyridyl)-s-triazine (TPTZ) (10 mmol/L) in a solution of HCI (40 mmol/L) and water solution of FeCl₃ (20 mmol/L). 90µL of the sample was pipette onto a glass tube and 270µL of ultrapure water and 2700µL of FRAP reagent. Then it was mixed and incubated for 30 minutes at 37 °C. The absorbance readings of the samples and standard were measured at 595 nm. The standard curve was performed with Fe₂SO₄ at concentrations of 100 to 2000 mmol / L in ethanol (y=0.0002x - 0.0215 R² = 0.99). The results were expressed in micromol Fe₂SO₄ equivalent / g extract.

2.6.2 HPLC analysis

The chromatographic analyses were carried out in high-performance liquid chromatography (HPLC) Shimadzu (Kyoto, Japan). The analysis of identification and quantification of phenolic compounds in the flours used the methodology described by Alcântara et al (2019). The samples were eluted with a gradient system consisting of solvent A (2% acetic acid, v/v) and solvent B (acetonitrile: methanol, 2:1, v/v), used as the mobile phase, with a flow rate of 1 mL/min. The temperature of the column was maintained at 25 °C and the injection volume was 20 µL. The gradient system started from 90% A at 0 min to 80% A at 10 min, 70% A at 15 min, 60% A at 25 min, 50% A at 30-40 min, 75% A at 42 min, and 90% A at 44 min. The peaks of the phenolic compounds were monitored at 280 nm. Identifications were made by comparing retention times with phenolic and flavonoid acid standards. The LabSolutions (Shimadzu) software was used to control the LC–UV system and for data processing.

2.7 Bulk and tapped densities

Densities were calculated as the ratio by mass/ volume, expressed in g/ml, according to Achor *et al.* (2015). For bulk density, samples were weighed into a 5 ml graduated cylinder without tapping, to determine the total mass of this volume occupied. Tapped density was determined from the mass of the sample in the measuring cylinder after 50 manual taps consecutive on the countertop surface at a height of 10 cm, to determine the volume occupied. Carr's index and Hausner's ratio were determined from the values of the bulk and tapped densities results obtained (JINAPONG; SUPHANTHARIKA; JAMNONG, 2008).

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2.8 True density

True density was determined by the liquid displacement method using the oil as the immersing fluid as described by Pragati; Genitha and Ravish (2014) and computed according to the following equation, with m_c , a mass of solid (g), and v_c , spent volume of oil (ml).

$$\rho_{t} = (m_{s}) / (10 - v_{s})$$
(1)

2.9 Porosity

The porosity (ϵ) was determined from the values of bulk (ρ_{b}) and true (ρ_{t}) densities when fitted into the equation according to the method of Drakos *et al.* (2017).

$$\varepsilon = (1 - (\rho_b / \rho_t)) . 100$$
 (2)

2.10 Water activity (Wa) and the potential of hydrogen (pH)

Water activity was determined by the free water meter (AQUALAB: 4TEV-EUA) at 25 ° C and calibrated with silica (0% RH). The pH of the samples was directly determined by the digital potentiometer (CIENLAB-MPA-210-BRASIL) at 25 °C, previously calibrated with pH 7.0 and 4.0 buffer solutions.

2.11 Hygroscopicity

Hygroscopicity was determined according to the method proposed by Caparino *et al.* (2012). 1g of the sample was weighed in an airtight container and placed in the desiccator with 75% relative humidity (saturated NaCl solution) at 25 °C for 7 days. The result was calculated by the ratio of the mass of water absorbed to the mass of the dry sample and expressed in g /100g dry sample.

2.12 Solubility

Solubility was determined using the procedure developed by Dacanal and Menegalli (2009). 1 g of sample to a vessel containing 100 ml of distilled water under agitation, maintaining the height of the vortex at 30 mm. After 1 min of agitation, the solution was quickly filtered and the filter containing the nondissolved particles was oven-dried at 105 °C for 24h. The solubility was evaluated from the fraction of non-dissolved material and expressed as g/100 g dry sample.

2.13 Water (WAC) and oil (OAC) absorption capacity

The absorption capacity test consisted of adding 1 g of sample to a centrifuge tube containing 10 ml of distilled water or refined soya- bean oil under agitation for 3 min. The samples were allowed to stand for 30 min and then centrifuged at 2500 rpm for 10 min. The supernatants were drained off, and the wet sediment was weighed. Water or oil absorption capacity was expressed as g of water or oil held per g dry sample (DRAKOS *et al.*, 2017).

2.14 Statistical analysis

ASSISTAT software version 7.7 was used to analyze the result by ANOVA and the Tukey test at a 95% level of significance. Data are reported as means \pm standard, each replication consisted of 3 independent measurements.

3 Results and Discussion

3.1 Proximate composition analysis, iron content and total energy value

The proximal composition and the total energy provide information on common nutrients. Based on our research there is limited data in the literature describing the relationship of nutrient content and anti-nutritional compounds of the juca flour. In table 1 are expressed in the values of reducing sugars, non-reducing sugars, protein, lipid, ash, moisture, and energy values for the fruit and the bark of the juca flour.

Samples	Fruit flour	Bark flour	
Reducing sugars (g/100g)	41.38±0.34 ^b	75.15±0.00ª	
Non-reducing sugars (g/100g)	43.61 ± 0.35ª	4.51±0.00 ^b	
Protein (g/100g)	0.86 ± 0.12^{b}	3.95 ± 0.17^{a}	
Lipid (g/100g)	$2.83 \pm 0.40^{\circ}$	2.06 ± 0.25^{a}	
Ash (g/100g)	2.58 ± 0.00^{b}	7.61 ± 0.00^{a}	
Moisture (g/100g)	4.44 ± 0.02^{a}	4.62 ± 0.23^{a}	
Energetic value (kcal/100g)	386.07	411.33	

 Table 1 – Results of proximal composition and total energy value.

* Values are mean ± SD (n=3). **Different superscript letters indicate significant differences (p<0.05) by Tukey test.

Source: Prepared by the authors.

The major macronutrient found in both parts were carbohydrates with values ranging from 89.29 g/100g for the fruit and 81.76 g/100g for the bark. Nutritional and technological point of view the presence of carbohydrates in large quantities adds value to the plant, as they are the main energy source of living beings and contribute to various texture properties of food products.

Among the carbohydrates, the reducing sugars in the shell presented the highest value, 75.15g glucose per 100g sample for bark, while the fruit was 41.28g glucose per 100g sample. For non-reducing sugars, these values ranged from 4.51 g sucrose per 100g of bark to 43.61 g sucrose per 100g of fruit. This high value of the fruit is mainly due to hydrocolloid polysaccharides such as galactomannans, present in juca seed, which has physical properties as emulsifiers, stabilizers, gel in aqueous solutions and thin films as reported by Cunha *et al.* (2017).

The high carbohydrates content of juca should be the environmental characteristics of the plant, since it is a native of Brazil and is distributed throughout the tropical and subtropical region of the country, especially in the North and Northeast (COSTA; GUILHON-SIMPLICIO; SOUZA, 2015), causing the plant suffers from a high level of incidence of sunlight and UV radiation (FIGUEREDO *et al.*, 2017). Also, the regions have low rainfall, causing plants to perform high photosynthesis rates, consequently causing an excess of carbon and nitrogen in their structure (FERREIRA; SOARES, 2015). However, the fruit and bark of the juca presented low protein content, ranging from 0.86 g/100g to 3.95 g/100g.

The lipid content of the bark and the fruit of juca showed no statistically significant differences (p < 0.05), ranging from 2.06 g/100g to 2.83 g/100g, respectively. These values are considered low when compared to leguminous plants (OLGUIN *et al.*, 2003).

The ash obtained for the bark was 7.61 g/100g, much higher than that found in the fruit of the juca (2.58 g/100g), thus showing the mineral richness of the juca since the ash content relates the number of minerals present in the food.

Micronutrient deficiencies, especially iron, are a major public health problem in the developing world, like Brazil (AYESSOU *et al.*, 2014). Besides that, iron was investigated due to the current Brazilian legislation, which defines iron-enriched flour, when it has in its composition from 4 to 9 mg iron/100g flour (BRAZIL – Ministry of Health, 2017). The values of iron content in the fruit and bark of juca flour ranged from 2.20 mg iron per 100g sample and 2.30mg iron per 100g sample, respectively. These showed the need to add more iron to the flour composition to classify it as iron-enriched.

The moisture values were low and below 5%. typical of dry products, due to the drying process of the raw material to obtain the flour, as well as the storage with controlled humidity and temperature. The lower moisture content found in the current study is important for the food processing industry. Moisture content directly influences other parameter control as absorption of water and fat, and the storage shelf life the grain flour (MEMON et al., 2020). Moisture content greater than 15% is usually not acceptable for long term storage of flour as described in the requirements RDC N. 263 of September 22nd, 2005 (BRAZIL -Ministry of Health, 2005). The values found for juca flours are complying with the requirements of Brazilian legislation and the flour can usually acceptable for long term storage.

The flour of fruit and bark of the juca are high in carbohydrates value, because of that providing a total energy value high as well (386.07 kcal/100g and 411.33 kcal/100g, respectively), which corresponds to approximately 20% of the caloric needs of an adult on a 2000 kcal diet (MUNHOZ *et al.*, 2018).

3.2 Anti-nutritional compounds and bioactive compounds

Tannic acid and phytic acid results are presented in table 2. Significant statistic difference (p<0.05) is observed within the samples.

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Table 2 – Results of anti-nutritionaland bioactive compounds.

Samples	Fruit flour	Bark flour
Tannic Acid (mg TA/ 100g)	88.91 ± 0.07a	87.83 ± 0.15b
Phytic Acid (103mg PA/ 100g)	3.86 ± 0.22a	1.80 ± 0.06b
Oxalic Acid (mg OA/ 100g)	0.00 ± 0.00a	0.00 ± 0.00a
TPC (mg GAE/g)	219.75 ± 0.10a	212.19 ± 0.96b
DPPH (%)	93.75 ± 0.10a	93.00 ± 0.96a
FRAP (mmol Fe2SO4/g)	315.23 ± 0.14a	300.72 ± 0.36b

* Values are mean ± SD (n=3). **Different superscript letters indicate significant differences (p<0.05) by Tukey test. Source: Prepared by the authors.

The tannic acid content of fruit flour (88.91 mg/100g) is higher than that of bark flour (87.83 mg/ 100g). The difference in tannin concentration varies according to plant tissues, as well as depending on age, plant size, time, and place of collection, as explained by Munhoz et al. (2018) in your search.

The phytic acid content of fruit flour (3.86 g/100g) is higher than that of bark flour (1.80 g/100g), with the same behavior found in tannic acid content. Phytic acid can chelate several important divalent cations (e.g. Fe, Zn, Ca and Mg) forming insoluble complexes and making them unavailable for absorption and utilization in the small intestine (SUMMO *et al.*, 2019). Phytate has also been implicated in decreasing protein digestibility by forming complexes and also by interfering with enzymes such as trypsin and pepsin. Phytic acids can also affect starch digestions by combining with digestive enzymes or bind minerals such as Ca, via phosphate linkages (RAJ BHANDARI; KAWABATA, 2006).

The occurrence of oxalic acid can decrease calcium absorption and aiding the formation of kidney stones, most of the urinary stones formed in humans are calcium oxalate stones (RAJ BHANDARI; KAWABATA, 2006). Oxalic acid was not detected in both samples, stating its absence in the flour composition developed. As well as, it was not found in the literature data reporting these values for juca fruits and bark, as it is commonly detected in leaves as explained by Ponka (2006) in his research.

The presence of tannic acid and phytic acid does not only represent a negative aspect. Since studies prove its action as anticarcinogenic and antioxidant, besides acting complexing minerals, enzymes, and proteins (MILLAR *et al.*, 2019; MURTHY *et al.*, 2019).

The antioxidant activity occurs mainly due to their potential for ox-reduction, which enables them to act as reducing agents, donating hydrogen, and neutralizing free radicals. As reported in table 2, both samples presented the high content of total phenolic compound (TPC) ranging from 212.19 mg GAE/ g for bark and 219.75 mg GAE/ g for fruit flour.

Antioxidant activity (AA), the samples are equal values of DPPH radical scavenging capacity assay (93%) and FRAP's method has significant differences (p<0.05) between the samples (315.23 for fruit flour and 330.72 for bark flour). Although the antioxidant activity measured by the method of free radical scavenging (DPPH) has a different mechanism of action than the method of the reduction potential of iron ions, the results showed similar trends. Antioxidant activity is associated with phenolic compounds since extracts of higher TPC also had greater antioxidant activities regardless of the method used.

Table 3 shows the quantification of the main phenolic substances present in juca flours were determined by HPLC. The peaks were positively evidenced and confirmed based on the retention time corresponding to the existing reference standards.

The phenolic compounds present in juca flour are potential sources of natural antioxidants for commercial operation, confirm the results obtained in the spectrophotometric methods of TPC, DPPH, and FRAP. There are typically compounds of benzoic acids (7), cinnamic acids (4), and their esters and flavonoids (6). The main phenolic compounds identified and quantified in both samples are the flavonoids catechin, myricetin, rutin, quercetin, and the acids syringic, vanillic, dihroxybenzoic, gallic, p-coumaric, and caffeic, what is following the literature. Araújo *et al.* (2014) analyzed aqueous crude extract and revealed the presence of gallic acid, catechin, epicatechin, and ellagic acid. Barros *et al.* (2014) studied extracts ethanolic of fruits and bark of juca and identified compounds as gallic acid and epicatechin.

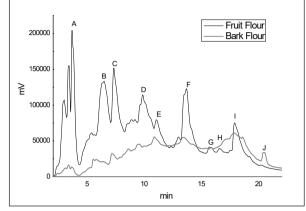
Phenolic compound	Fruit Flour (mg/g)	Bark Flour (mg/g)	
Hydroxybenzoic acid			
Syringic Acid	94.40	3.00	
Vanillic acid	79.00	9.20	
Salicylic acid	59.60	ND	
Dihroxybenzoic Acid	19.20	6.00	
Ellagic Acid	5.20	17.60	
Gallic acid	17.40	0.80	
Hydroxybenzoic acid	6.40	0.20	
Hydroxycinnamic acid			
p-Coumaric acid	60.80	5.40	
Ferulic acid	46.20	ND	
Caffeic acid	34.80	6.60	
trans-Cinnamic acid	0.40	0.40	
Flavonoid			
Catechin	330.00	20.60	
Myricetin	158.20	38.40	
Rutin	72.40	2.20	
Quercetin	36.80	5.60	
Chrysin	1.20	1.40	
Kaempferol	0.60	0.60	

Table 3 -	Phonolic	compounds	nresent in i	iuca flours
Table 5 –	I HEHOIC	Compounds	DIESEIIL III	uca nours.

*ND- not identified.

Source: Prepared by the authors.

The major compounds identified in both samples were the flavonoids, representing 58.59% for fruit flour and 58.30% for bark flour of phenolic compounds identified. The catechin was the most important detected and quantified compound in the juca flours, in concentrations up to 330.00 mg/g in juca fruit flour. The catechin has several therapeutic properties on human health, as anti-inflammatory, antiviral, and antibacterial effects (FERREIRA; SOARES, 2015). Figure 1 – HPLC chromatogram of the phenolic compounds of the juca flours. Peaks: A, Gallic acid; B, Dihroxybenzoic Acid; C, Catechin; D, Vanillic acid; E, Syringic Acid; F, p-Coumaric acid; G, Rutin; H, Ellagic Acid; I Myricetin; J, Quercetin



Source: Prepared by the authors.

The second main compound identified was the myricetin, in concentrations up to 158.20 mg/g in juca fruit flour. Myricetin shows the highest antioxidant capacity when compared with other flavonoids (quercetin, kaempferol, and rutin). That action is attributed to increases in the number of phenolic hydroxyl groups in this compound (ALCÂNTARA *et al.*, 2019). Myricetin is known to present antioxidants and decrease the risk of prostate cancer (ABU-REIDAH *et al.*, 2015).

The acids salicylic (59.60 mg/g) and ferulic (46.20 mg/g) were identified only in the juca fruit flour. Thus, it is possible to verify the influence of the botanical parts of the plant.

The information on the molecular structure of compounds justifies the high antioxidant activity observed by two of the methods used (DPPH and FRAP) in this study. Analyzing the molecular structure of the major compounds: the catechin consists of two aromatic rings and one hydroxyl group; and myricetin consists of three aromatic rings with six hydroxyl groups. Thus, these compounds provide high availability of the hydroxyl groups to react with free radicals or reduce other compounds (ALCÂNTARA *et al.*, 2019).

This work shows that *L. ferrea* is a promising candidate for the development of flour rich in carbohydrates and bioactive compounds. Those results have encouraged research on the technological and functional properties of this product.

3.3 Densities, Hausner's ratio, Carr's index, and Porosity

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Table 4 shows the results of densities, Hausner's ratio, Carr's index, and porosity. There were statistically significant differences (p < 0.05) between the samples for most results.

Table 4 – Results of densities, Hausner'	S
ratio, Carr's index, and porosity.	

Samples	Fruit flour	Bark flour
Bulk density (g/ml)	0.60 ± 0.00^{a}	0.60 ± 0.00^{a}
Tapped density (g/ml)	0.83 ± 0.00^{a}	0.76 ± 0.01^{b}
True density (g/ml)	$0.86 \pm 0.01^{\circ}$	0.77 ± 0.01^{b}
Hausner's ratio	1.38 ± 0.01^{a}	1.27 ± 0.02^{b}
Carr's index (g/100g)	27.68 ± 0.33ª	21.13 ± 0.47^{b}
Porosity	$0.30 \pm 0.00^{\circ}$	0.22 ± 0.02^{b}

* Values are mean ± SD (n=3). **Different superscript letters indicate significant differences (p<0.05) by Tukey test.

Source: Prepared by the authors.

Density is an important cost parameter in the flour production chain. Low-density materials require larger storage space and increase the cost of logistics and packaging. However, flours with low bulk density values are suitable for the preparation of infant and weaning foods due to their easy digestibility, since this parameter influences the texture of food.

The bulk density for both samples studied is a low value (0.60 g/ml), because of that, it can be used for better texture children's foods. The values of tapped and true density are similar in the same sample due to the specificity of the samples, for fruit flour was 0.83 g/ml and 0.86 g/ml, and for bark flour was 0.76 g/ml and 0.77 g/ml, respectively.

Hausner's ratio and Carr's compressibility percentage index are considered indirect measures of flour flow property. The Hausner's ratio is indicative of particle friction, while Carr's index shows the material's ability to decrease volume. Hausner's ratio for fruit flour (1.38) was higher than bark flour (1.27), showing an intermediate fluidity as stated by Jinapong; Suphantharika and Jamnong (2008). Carr's index for fruit flour (27.68) was higher than for stem shell flour of (21.13), showing average fluidity with values between 16% and 35% as stated by Achor *et al.* (2015).

Porosity is an important property in many aspects. It can indicate the presence of oxygen resulting in more rapid degradation of oxidizing compounds, but it may also improve the water absorption material (DRAKOS *et al.*, 2017). The porosity found in the fruit flour (0.30) was higher than the bark flour (0.22). Pragati *et al.* (2014) were studying the green and ripe banana flour produced through the drying oven found values similar to the present research, this can be attributed due to similar processing.

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3.4 Hydration properties and Oil absorption capacity

Table 5 shows the results of proprieties analysis which influence quality control, packaging type, and storage conditions of the product.

The main hydration properties are measured by hygroscopicity, water-solubility, water absorption capacity (WAC), and water activity (Wa). Hygroscopicity addresses the amount of water spontaneously fixed in the matrix, influenced by density, porosity, and solubility, and is one of the most important carbohydrate properties (PRAGATI; GENITHA; RAVISH, 2014). The hygroscopicity of fruit flour (10.31 g/100g) is higher than the bark flour (5.56 g/100g) influenced by the high carbohydrate content of fruit flour.

The solubility of fruit flour (54.04 g/100g) is lower than that of bark flour (56.67 g/100g), showing a decrease in dispersibility and reconstitution property. This feature can be attributed to the low content of water-soluble substances such as minerals (REYNIERS *et al.*, 2019).

Table	5 – Results of properties of hydration,
	oil absorption capacity, and pH.

Samples	Fruit flour	Bark flour
Hygroscopicity (g/ 100g)	10.31 ± 0.18 ^a	5.56 ± 0.12 ^b
Solubility (g/100g)	$54.04 \pm 0.70^{\circ}$	56.67 ± 0.28°
WAC (gH ₂ O/g)	1.20 ± 0.21^{a}	1.06 ± 0.1⁵a
Wa (25°C)	0.26 ± 0.01^{a}	0.30 ± 0.01^{a}
OAC (gOil/g)	$0.83 \pm 0.02^{\circ}$	0.75 ± 0.02ª
рН (25°С)	3.52 ± 0.03 ^b	$4.64 \pm 0.0^{1}a$

* Values are mean \pm SD (n=3). **Different superscript letters indicate significant differences (p<0.05) by Tukey test. ***WAC, water absorption capacity; OAC, oil absorption capacity; Wa, water activity; pH, potential of hydrogen.

Source: Prepared by the authors.

The water absorption capacity index reflects on the sensory characteristics of food and indicates the amount of water that flour granules are capable of absorbing (FELKER *et al.*, 2018). This is related to the availability of hydrophilic groups (-OH) to bind to water molecules and the gelling ability of starch molecules (REYNIERS *et al.*, 2019). The values did not show a statistically significant difference (p < 0.05) between the samples with low values close to 1 gH₂O/g. Those flours may be suitable for products requiring low water retention and high crispness.

Flour water activities presented values below 0.60, being considered stable to the development of microorganisms and susceptible to oxidative reactions. For this reason, storage in oxygen-impermeable packaging such as vacuum packaging is recommended.

The oil absorption capacity index addresses the combination of fat with nonpolar groups of proteins that are composed of hydrophilic and hydrophobic parts. As well as the availability of lipophilic groups whose oil absorption mechanism is mainly due to the physical entrapment of the oil by capillary attraction (DRAKOS *et al.*, 2017). The values did not show a statistically significant difference (p <0.05) between the samples as values below 1 g oil/g. The low oil absorption capacity is associated with the low protein content of the flour. This feature can play an important role in enhancing the feel and retention of product taste.

When comparing the potential of hydrogenic (pH) of flour, the fruit derivative is classified as very acidic (3.52) while the stem bark flour (4.64) is low acidic. This may be related to the high protein content of bark flour.

4 Conclusions

The present research describes a pioneering study of technological functionality propriety of the juca fruits and bark flour, thereby contributing to the scientific knowledge of this species, as well as facilitating sustainable exploitation of this plant. The objective of this study was achieved completely.

The flours have significant carbohydrate values. Antinutrient compounds such as tannic and phytic acids do not devalue the produced flours once they are potential antioxidant agents. The flour is a source of phenolic compounds with high antioxidant activity, due to the flavonoids compounds as catechin and myricetin.

Therefore, fruit flour is the best one when compared to the studied botanical parts, in terms of carbohydrates, antioxidants, and technological functionality propriety. The fruit flour is suitable for products that require low water and fat retention, to improve the sensory characteristics of crispness, increase the feel and retention of product taste.

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REFERENCES

ABNT -BRAZILIAN ASSOCIATION OF TECHNICAL STANDARDS. NBR No. 13934 - Iron Determination, Orthophenanthroline colorimetric method. . 1997.

ABU-REIDAH, I. M.; ALI-SHTAYEH, M. S.; JAMOUS, R. M.; ARRÁEZ-ROMÁN, D.; SEGURA-CARRETERO, A. HPLC – DAD – ESI-MS / MS screening of bioactive components from Rhus coriaria L. (Sumac) fruits. Food Chemistry, v. 166, n. 1, p. 179–191, 2015. DOI:10.1016/j.foodchem.2014.06.011.

ACHOR, M.; OYENIYI, J. Y.; MUSA, M.; GWARZO, M. S. Physicochemical Properties of Cassava Starch Retrograded in Alcohol. Journal of Applied Pharmaceutical Science, v. 5, n. 10, p. 126–131, 2015. DOI:10.7324/JAPS.2015.501021.

ALCÂNTARA, M. A. *et al.* Effect of the solvent composition on the profile of phenolic compounds extracted from chia seeds. Food Chemistry, v. 275, p. 489–496, 2019. DOI:10.1016/j.foodchem.2018.09.133.

AOAC - ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. Official methods of analysis of the Association of Official Analytical Chemists (method 920.39,C) Arlington: A.O.A.C., 1995.

ARAÚJO, A. A. *et al.* Quantification of polyphenols and evaluation of antimicrobial, analgesic and antiinflammatory activities of aqueous and acetone-water extracts of Libidibia ferrea, Parapiptadenia rigida and Psidium guajava. Journal of Ethnopharmacology, v. 156, p. 88–96, 2014. DOI: 10.1016/j.jep.2014.07.031

AYESSOU, N. C. *et al.* Nutrient composition and nutritional potential of wild fruit Dialium guineense. Journal of Food Composition and Analysis, v. 34, n. 2, p. 186–191, 2014. DOI: 10.1016/j.jfca.2014.01.002.

BARROS, A. O. *et al.* Antioxidant and hepatoprotective activities of Libidibia ferrea bark and fruit extracts. International Journal of Pharmacy and Pharmaceutical Sciences, v. 6, n. 11, p. 1–6, 2014.

BRAND-WILLIAMS, W.; CUVELIER, M. E.; BERSET, C. Use of free radical method to evaluate antioxidant activity. **LWT - Food Science and Technology**, v. 28, n. 1, p. 25–30, 1995. BRAZIL - MINISTRY OF HEALTH. National Health Surveillance Agency, Resolution N. 16 of April 30th, 1999. Approves the Technical Regulation of Procedures for the Registration of Food and or New Ingredients. 1999, p. 3–6.

revista

BRAZIL - MINISTRY OF HEALTH. National Health Surveillance Agency, Resolution N. 263 of September 22nd, 2005 Technical Regulation for Cereal, Starch, Flour and Bran Products. . 2005, p. 6.

BRAZIL - MINISTRY OF HEALTH. National Health Surveillance Agency, Resolution N. 150 of April 13th, 2017. Provides for the enrichment of wheat and maize flour with iron and folic acid. . 2017, p. 1–4.

CAPARINO, O. A. *et al.* Effect of drying methods on the physical properties and microstructures of mango (*Philippine "Carabao" var.*) powder. **Journal of Food Engineering**, v. 111, n. 1, p. 135–148, 2012. DOI: 10.1016/j.jfoodeng.2012.01.010.

CARVALHO, A. F. U. *et al.* Preliminary assessment of the nutritional composition of underexploited wild legumes from semi-arid Caatinga and moist forest environments of northeastern Brazil. **Journal of Food Composition and Analysis**, v. 24, n. 4–5, p. 487–493, 2011. DOI: 10.1016/j.jfca.2011.01.013.

CHANG, S. K. C.; XU, B. Total phenolic, phenolic Acid, anthocyanin, flavan-3-ol, and flavonol profiles and antioxidant properties of pinto and black beans (*Phaseolus vulgaris L.*) as affected by thermal processing. **Journal of Agricultural and Food Chemistry**, Washington, v. 57, n. 1, p. 4754–4764, 2009.

COSTA, L. M.; GUILHON-SIMPLICIO, F.; SOUZA, T. P. *Libidibia ferrea (Mart. Ex tul) L. P. Queiroz var. Ferrea*: Pharmacological, phytochemical and botanical aspects. **International Journal of Pharmacy and Pharmaceutical Sciences**, v. 7, n. 4, p. 48–53, 2015. DOI: 10.5897/JMPR2014.5706.

CUNHA, A. P. *et al.* Polysaccharides from Caesalpinia ferrea seeds – Chemical characterization and antidiabetic effects in Wistar rats. **Food Hydrocolloids**, v. 65, p. 68–76, 2017. DOI: 10.1016/j.foodhyd.2016.10.039.

DACANAL, G. C.; MENEGALLI, F. C. Experimental study and optimization of the agglomeration of acerola powder in a conical fluid bed. **Powder Technology**, v. 188, n. 3, p. 187–194, 2009. DOI: 10.1016/j.powtec.2008.04.076.

DIAS, A. M. A. et al. Wound dressings loaded with an anti-inflammatory jucá (*Libidibia ferrea*) extract

using supercritical carbon dioxide technology. Journal of Supercritical Fluids, v. 74, p. 34–45, 2013. DOI: 10.1016/j.supflu.2012.12.007.

DRAKOS, A. *et al.* Influence of jet milling and particle size on the composition, physicochemical and mechanical properties of barley and rye flours. **Food Chemistry**, v. 215, n. 1, p. 326–332, 2017. DOI: 10.1016/j.foodchem.2016.07.169.

FELKER, F. C.; KENAR, J. A.; BYARS, J. A.; SINGH, M.; LIU, S. X. Comparison of properties of raw pulse flours with those of jet-cooked, drum-dried flours. **LWT - Food Science and Technology**, v. 96, p. 648–656, 2018. DOI:10.1016/j.lwt.2018.06.022.

FERREIRA, M. R. A.; SOARES, L. A. L. *Libidibia ferrea (Mart. ex Tul.) L. P. Queiroz*: A review of the biological activities and phytochemical composition. **Journal of Medicinal Plants Research**, v. 9, n. 2, p. 140–150, 2015. DOI: 10.5897/JMPR2014.5706.

FIGUEREDO, F. G. *et al.* Antimicrobial Activities of Natural Products from *Libidibia ferrea (Mart. ex Tul.) L.P. Queiroz var. ferrea. In:* Antibacterials Synthesis, Properties and Biological Activities. ISBN 9781634858014 (e-Book) Cap 5. p. 115, 2017.

JINAPONG, N.; SUPHANTHARIKA, M.; JAMNONG, P. Production of instant soymilk powders by ultrafiltration, spray drying and fluidized bed agglomeration. **Journal of Food Engineering**, v. 84, n. 1, p. 194–205, 2008.

LIMA, C. M. G. *et al.* Development and physicalchemical characterization of sweet potato (*Ipomoea batatas L*) flour with addition of brown flaxseed (*Linum usitatissimum L*). **Brazilian Journal of Development**, v. 5, n. 6, p. 5185–5193, 2019.

MEMON, A. A. *et al.* Impact of flour particle size on nutrient and phenolic acid composition of commercial wheat varieties. **Journal of Food Composition and Analysis**, v. 86, p. 103358, 2020. DOI: 10.1016/j.jfca.2019.103358.

MERRIL, A. L.; WATT, B. K. **Energy value of foods: basis and derivation.** Washington: United States Department of Agriculture, 1973.

MILLAR, K. A.; GALLAGHER, E.; BURKE, R.; MCCARTHY, S.; BARRY-RYAN, C. Proximate composition and anti-nutritional factors of fava-bean (*Vicia faba*), green-pea and yellowpea (*Pisum sativum*) flour. **Journal of Food** Composition and Analysis, v. 82, n. June, p. 103233, 2019. DOI:10.1016/j.jfca.2019.103233.

MOIR, K. W. Determination of oxalic acid in plant Queensland. Journal Agricultural Science, v. 10, n. 1, p. 1–3, 1953.

MUNHOZ, C. L.; GUIMARÃES, R. C.; NOZAKI, V. T.; SANJINEZ-ARGANDOÑA, E. J.; MACEDO, M. L. R. Chemical composition and factors antinutritional of bocaiuva fruit. **Agricultural and Environmental Sciences Sector Magazine**, v. 14, n. 1, p. 212–2224, 2018. DOI:10.5935/ambiencia.2018.15.01.

MURTHY, H. N.; JOSEPH, K. S.; GAONKAR, A. A.; PAYAMALLE, S. Evaluation of Chemical Composition and Antioxidant Activity of Cordia myxa Fruit Pulp. **Journal of Herbs, Spices and Medicinal Plants**, v. 1, p. 1–10, 2019. DOI:10.1080/10496475.2019.1585399.

NASCIMENTO, P. *et al.* Antioxidant and antimicrobial properties of ethanolic extract of *Libidibia ferrea* pods. **Revista Fitos**, v. 9, n. 3, p. 207–216, 2015. DOI:10.5935/2446-4775.20150017.

OLGUIN, M. C. *et al.* Nutritional and antinutritional aspects of an Argentinian soy flour assessed on weanling rats. **Journal of Food Composition and Analysis**, v. 16, n. 4, p. 441–449, 2003. DOI:10.1016/S0889-1575(03)00005-X.

OLIVEIRA, P. M. L. *et al.* Juá fruit (*Ziziphus joazeiro*) from Caatinga: a source of dietary fiber and bioaccessible flavanols. **Food Research International**, v. 129, n. March 2020, p. 108745, 2020. DOI:10.1016/j.foodres.2019.108745.

PEREIRA, L. P. *et al.* Polysaccharide fractions of *Caesalpinia ferrea* pods : Potential anti-inflammatory usage. **Journal of Ethnopharmacology**, v. 139, n. 1, p. 642–648, 2012. DOI: 10.1016/j.jep.2011.12.012.

PONKA, R. Composition of dishes consumed in Camerron. International Journal of Food Science and Technology, v. 4, n. 1, p. 361–365, 2006.

PORT'S, P. S.; CHISTÉ, R. C.; GODOY, H. T.; PRADO, M. A. The phenolic compounds and the antioxidant potential of infusion of herbs from the Brazilian Amazonian region. **Food Research International**, v. 53, n. 2, p. 875–881, 2013. DOI:10.1016/j.foodres.2013.02.010.

PRAGATI, S.; GENITHA, I.; RAVISH, K. Comparative Study of Ripe and Unripe Banana Flour during Storage.

revista

Journal of Food Processing & Technology, v. 5, n. 11, p. 1–6, 2014. DOI:10.4172/2157-7110.1000384.

RAJ BHANDARI, M.; KAWABATA, J. Cooking effects on oxalate, phytate, trypsin and α -amylase inhibitors of wild yam tubers of Nepal. Journal of Food Composition and Analysis, v. 19, n. 6–7, p. 524–530, 2006. DOI:10.1016/j.jfca.2004.09.010.

RANGANA, S. Manual of analysis of fruit and vegetable products. New Delhi: **Tata McGraw Hill Publishing Company**, New Delhi, 1979.

REYNIERS, S.; BRIER, N.; MATTHIJS, S.; BRIJS, K.; DELCOUR, J. A. Impact of mineral ions on the release of starch and gel forming capacity of potato flakes in relation to water dynamics and oil uptake during the production of snacks made thereof. **Food Research International**, p. 1–46, 2019. DOI:10.1016/j.foodres.2019.03.065.

ROSSI, J. A. J.; SINGLETON, V. L. Colorimetry of total phenolics with phosphomolybdic phosphotungstic acid reagents. **American Journal** of Enology and Viticulture, v. 16, p. 144–158, 1965.

RUFINO, M. S. M. *et al.* Metodologia Científica: determinação da atividade antioxidante total em frutas pelo método de redução do ferro (FRAP). **Comunicado Técnico Embrapa**, Fortaleza, CE, v. 125, p. 1–4, 2006.

SUMMO, C. *et al.* Nutritional, physico-chemical and functional characterization of a global chickpea collection. **Journal of Food Composition and Analysis**, v. 84, , p. 103306, 2019. DOI:10.1016/j.jfca.2019.103306

TAMSEN, M.; SHEKARCHIZADEH, H.; SOLTANIZADEH, N. Evaluation of wheat flour substitution with amaranth flour on chicken nugget properties. **LWT** - Food Science and Technology, v. 91, n. 1, p. 580–587, 2018. DOI:10.1016/j.lwt.2018.02.001.

THIYAJAI, P.; SAETANG, P.; KETTAWAN, A.; CHAROENKIATKUL, S.; SRICHAMNONG, W. Promising industrial flour processing and household applications of parboiled germinated brown rice (*Khao dok mali*). **LWT - Food Science and Technology**, v. 73, n. 4, p. 406–411, 2016. DOI:10.1016/j.lwt.2016.06.044.