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Comparative Evaluation of Agricultural Residues in the Production of Dietary Fibers

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Abstract. The well established health benefits associated with dietary fibers have not only increased consumer interest in fiber rich products, but also research interest in new fiber sources. In this study, we compared the potential of three agricultural residues, pequi peels, coffee husks and wheat bran, as substrates for the production of fiber rich powders. Dietary fiber contents ranged from 39.8 to 66.9g/100g, with the lowest and highest values corresponding to pequi peels and coffee husks, respectively. The amount of soluble fibers was higher for pequi peels and coffee husks in comparison to wheat bran, thus both pequi peels and coffee husks are probably more versatile in terms of applications, given the enhancement of hydration properties. Even though coffee husks presented high contents of phenolic compounds, the concentration in pequi peels was significantly higher. All fibers presented high antioxidant activity, with direct correlation to the amount of phenolics.

Keywords: Antioxidant capacity, Coffee husks, *Caryocar Brasilense* Camb, Pequi, Wheat bran.

1. Introduction

The well known fact that grains, fruits and vegetables are good sources of dietary fibers has prompted investigations on the feasibility of employing fruits, vegetables and cereals as fiber sources [1]. Corresponding industry by-products are of particular interest as alternative sources of dietary fibers, given that they are inexpensive and usually available in large quantities. Pequi (*Caryocar brasiliense* Camb.) is typical fruit found in the Brazilian cerrado. This fruit is economically exploited by the regional population, and consumed after cooking (usually mixed with rice) and also used for preparation of liquor, ice cream and traditional dishes [2]. The fruit peel represents ~80% of the total fruit mass and is usually discarded. Coffee is the most relevant agricultural product in Brazil, with an average yearly production of 2.5 million tons. Coffee husks are the major solid residues from coffee processing. For every kg of coffee beans produced, approximately 1 kg of husks are generated. Although some alternative uses for coffee husks have been proposed such as supplement for animal feed, direct use as fuel, fermentation for the production of a diversity of products, production of adsorbents and others, there is still a need to find other alternative uses for this solid residue because of the high amounts generated [3]. Given that no literature data on the characterization or use of such agricultural residues (both pequi peels and coffee husks) as a source of dietary fibers were found, we evaluated the potential of these materials as substrates for the production of fibers with antioxidant capacity. Wheat bran was used as a reference.

2. Materials and Methods

2.1. Materials

The pequi (*Caryocar brasilense* Camb.) fruits were acquired at CeasaMinas, Minas Gerais State Agricultural Production Distribution Center (Contagem, MG, Brazil). Fruits were visually and manually

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inspected and only healthy fruits were employed. Coffee husks were provided by Minas Gerais State Coffee Union (Sindicafé, Belo Horizonte, MG). The peels were manually removed from the pequi fruits and cut into smaller pieces (2 x 2 cm). The exocarp was removed and the samples were blanched. Samples were blended with water (1:1) using a mixer (model 712, Fisaton, São Paulo, Brazil). The obtained mixture was spread out on trays and dried at 50 ± 2 °C for 24 h and then sieved through a 425-μm mesh (PF). Dry coffee husks were blanched, submitted to drying (50°C for 6h) and sieved through a 425-μm mesh (CF). Blanching consisted on immersion in hot water (90°C) followed by immersion in cold water (4°C), 3 min each [4]. Commercially available wheat bran was dried and ground to the same conditions (WF).

2.2. Methods

Visual color was measured using a Hunter colorimeter model ColorFlex (Hunter Associates Laboratory, Reston, VA) with standard illumination D_{65} and colorimetric normal observer angle of 10°. Measurements were based on the CIE $L^*a^*b^*$ three dimensional cartesian (xyz) color space represented by: Luminosity (L^*), ranging from 0 (black) to 100 (white) – z axis; parameter a^*, green–red color component – x axis; and parameter b^*, blue–yellow component -y axis. However, a^* and b^* values were converted to chroma (c^*) and hue angle (h), since these parameters can be directly associated to color intensity (c^*) and tone (h):

$$c^* = \left[a^{*2} + b^{*2}\right]^{1/2} \quad (1)$$

$$h = \tan^{-1}\left[b^*/a^*\right] \quad (2)$$

An enzymatic–gravimetric method was used to determine the total dietary fibre content [5]. Briefly, the samples were initially submitted to gelatinization (100 °C for 15 min) with α-amylase, followed by enzymatic digestion with pepsin and pancreatin, both for 60 min at 40 °C. Subsequently, insoluble dietary fiber (IDF) was filtered and washed with warm distilled water. The filtrate and washed water were combined, 95% ethanol at 60 °C was added and the mixture was let stand for 60 min in order to precipitate the soluble dietary fiber (SDF). The residues were weighed after overnight drying at 105 °C in a hot air oven. Total dietary fiber (TDF) was calculated as the sum of IDF and SDF.

The preparation of extracts followed the method described by Hassan and co-workers [6], with a few modifications. The prepared fiber powders (250 mg) were placed in test tubes sequentially extracted with 20 ml of 50% (v/v) methanol and 20 ml of 70% (v/v) acetone at 25 °C on an orbital shaker (Marco Q-250, São Paulo, Brazil) at 200 rpm for 1 h. The mixture was centrifuged at 3500rpm, for 10 min using a Celm centrifuge (São Paulo, Brazil). The supernatants were then combined and brought to a final volume of 100 ml with distilled water. The extract was kept at -18 °C for further analysis. The total amount of extractable phenolics (TP) was then determined according to the Folin–Ciocalteau method [7]. In summary, 1 ml of the sample extract was added to 5 ml of Folin–Ciocalteu reagent followed by addition of 4 ml of sodium carbonate solution. The mixture was stirred and allowed to stand for 120 min. The absorbance was read at 760 nm using a UV/Vis spectrophotometer (Micronal AJX 1900, São Paulo, Brazil). The results were expressed as mg of gallic acid equivalents (GAE) per g of dry matter based on a calibration curve of gallic acid at concentrations ranging from 0.02 mg/ml to 0.07 mg/mL.

In vitro antioxidant capacity was evaluated by reaction with 2,2′-azino-bis-3-ethylbenzthiazoline-6-sulphonic acid (ABTS). The ABTS assay was based on the method described by Floegel et al. [8] with modifications. In summary, 50mL of ABTS solution (3.8 g/L) were mixed with 88 μL of potassium persulfate (37.8g/L) and kept in the dark for 16h. The blue-green $ABTS^+$ solution was diluted in ethanol until absorbance of 0.70 ± 0.050 at 734 nm. Different dillutions (1:10, 1:20, 1:30, 1:40 and 1:50 v/v) of PF, CF and WF extracts in methanol were prepared. Then, 30 μL of the solution were mixed with 300μL of $ABTS^+$ solution and incubated for 6 min. The decrease of absorbance was monitored at 734 nm until absorbance readings became stable. Results were expressed as μM trolox equivalents per grams of dry sample (μM TE/ g dry weight).

3. Results and Discussion

Results obtained for color parameters of the produced powders are displayed in Table I. PF and CF luminosity values are low compared to WF and also to other types of peel-based powders such as orange (L*~80), but similar or higher than to date fibre concentrates (43<L*<55) and grape pomace (30<L*<47) [9]-12]. Luminosity values are an important parameter when darkening of the the food product affects its aceitability, and such values must be monitored not only for the ingredients but also for the final products. For example, Soares Júnior and co-workers [13] evaluated the effect of adding pequi peel powder as a substitute to wheat flour in cookies. The luminosity of the produced cookie was evaluated, ranging from 55 (pure wheat flour) to 35 (50% pequi peel powder). These authors observed a decrease in product luminosity with the addition of pequi peel powder. It is interesting to notice that the luminosity of the control sample (cookie prepared with pure wheat flour) is actually similar to the luminosity of the pequi peel powder (see Table I), and thus the decrease in luminosity during product processing is probably associated to caramelization of sugars and Maillard reactions that will naturally occur during any thermal processing taking place in food preparation. Diferences in color tone (hue angle) were observed between the fibers obtained from wheat bran, coffee husks and pequi peel, with the decrease in hue angle value associated with tone changing from yellow towards orange. In general, color intensity values for the developed fiber powders are not particularly high, with the smallest intensity of WF visually providing a greyish aspect.

Table I: Color parameters.

Sample	Luminosity (L*)	Hue angle (h)	Color intensity (c*)
WF	65.83 ±0.82a	70.35 ±0.32b	19.55 ±0.22c
PF	55.19 ± 0.01b	72.55 ± 0.11a	30.64 ± 0.11a
CF	47.12 ±0.19c	67.16 ±0.05c	25.22 ±0.10c

Average value ± standard deviation. Values followed by the same letter in the same column do not differ significantly (Tukey test, 95% confidence).

The results for dietary fiber contents are shown in Fig 1. TDF and IDF are higher for both CF and WF in comparison to PF. CF total dietary fiber contents (~67 g/100g in average) are similar to other fruit peels such as orange and lemon (~67-80 g/100g), and quite high in comparison to fruits and vegetables such as apple (~13 g/100g), tomato (~19 g/100g) and carrots (~26 g/100g). Although smaller than the other produced fiber powders, TDF values for PF are in the same range of carrot peels (~39-55 g/100g) and commonly employed fiber sources such as rice and wheat bran (~27-45 g/100g) [1] and [14]. The soluble and insoluble nature of dietary fibres not only affect their technological functionality, but also the associated physiological effects. IDF are associated to porosity, low density and ability to increase faecal bulk and decrease intestinal transit [1]. IDF values were the highest for CF, followed by WF and PF. Values are in the same range of other fruit and vegetable peels including mango (~39 g/100g), lemon (~42 g/100g), orange (~52 g/100g) and carrots (~34-51 g/100g) and significantly high in comparison to fruits including apple, orange, dates and tomatoes (~5-12 g/100g) [1] and [6]. High IDF values were expected for WF, since this is typical of grain-based fiber powders that are basically comprised of cellulose, hemicellulose and lignin. Nonetheless, the higher values obtained for CF point towards the feasibility of employing such residue as a substitute for grain milling by-products in formulations of the so-called "all bran" cereals. SDF are also of interest from both technological and functional points of view, in association to their capacity to increase viscosity and to reduce the glycemic response and plasma cholesterol as well as prebiotic action, and its higher nutritional value [1]. SDF values were significantly higher for both PF and CF in comparison to WF, and slightly high in comparison to some fruits including apple, peach and tomato [1].

The results obtained for hydration properties are displayed in Table II. WRC, SWC and ORC values are indicative of possibilities about the use of fibers as ingredients in food products. For example, dietary fibres with high OHC allow the stabilization of high fat food products and emulsions. Dietary fibres with high WRC can be used as functional ingredients to avoid synaeresis and modify the viscosity and texture of some formulated foods. The water retention capacity (WRC) is the quantity of water that remains bound to the hydrated fiber following the application of an external force [14]. WRC values were similar for CF and PF and slightly lower for WF, but all values were high in comparison to residues from juice extraction (peel and

pomace) of grapefruit, lemon, orange and apple (~1.6-2.3 g/g) [14] and [15]. ORC values for CF are high in comparison to PF, WF, as well as fruit peel and pomace (~1.5-3.3 g/g) [15] and thus especially CF could be interesting as an additive in foods that require some emulsification levels, such as ice creams. SWC values obtained for CF and PF are higher than WF and in the range reported for residues from juice extraction of grapefruit, lemon, orange and apple (~6-9 g/g) [15]. The better hydration properties of CF and PF in comparison to WF are attributed to their higher content of SDF.

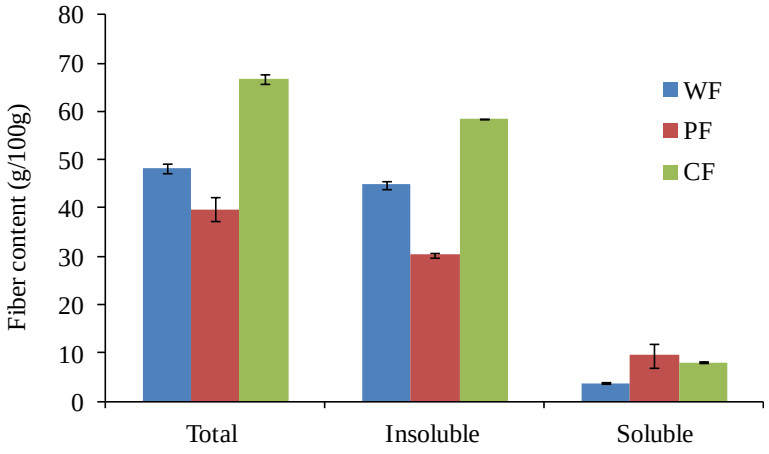

Fig. 1: Dietary fiber contents

Table II: Fiber hydration properties.

Sample	CF	PF	WF
WRC (g water/ g sample)	4.32 ±0.03a	3.74 ±0.44a	2.75 ±0.14b
ORC (g oil/ g sample)	4.91 ± 0.01a	1.23 ±0.03b	1.26 ±0.1b
SWC (mL water/ g sample)	8.05 ± 0.09b	11.34 ±0.92a	4.13 ±0.52c

Average value ± standard deviation. Values followed by the same letter in the same line do not differ significantly ($p > 0.05$).

The results obtained for total phenolics and antioxidant activity are displayed in Table III. Both CF and PF presented significantly higher phenolics content and antioxidant activity than WF. PF phenolic contents are significantly high in comparison to literature data. Examples include mango peels (4400-7000 mg GAE/100g), grape processing residues (6700 mg GAE/100g) and carrot peels (877-1017 mg GAE/100g) [6], [12] and [14]. A correlation between TP and antioxidant activity can be seen. Both CF and PF presented significantly higher values of antioxidant activity in comparison to WF. Such values are also significantly high in comparison to fruits and other agricultural residues including cranberries (~98 µM TE/g), mango peel and pulp (~15-38 µM TE/g) and marolo pulp (~132 µM TE/g) [6],[8] and [16].

Table III: Total phenolics and in vitro antioxidant activity

Sample	Total phenolics (mg GAE /100g)	Antioxidant activity (µM TE/g)
WF	0.75 ±0.05e	6.15 ± 0.03c
CF	515.41 ±12.46b	607.14 ± 42.62b
PF	15490.90 ±21.70a	986.9 ±33.5a

Average value ± standard deviation. Values followed by the same letter in the same column do not differ significantly (Tukey test, 95% confidence). GAE = galic acid equivalent.

4. Conclusions

Results indicate that both pequi peel and coffee husks could be suitable sources of dietary fibers and phenolic compounds. Coffee husks exhibited higher contents of total dietary fiber (~58 g/100g) in comparison to pequi peel (~30 g/100g), both with dominance of insoluble dietary fiber fraction. Nonetheless, soluble fiber contents were significantly higher for both pequi peel and coffee husks in comparison to wheat

bran, resulting in better hydration properties. Both coffee husks and pequi peel resulted in fiber rich powders with significantly higher phenolics content and antioxidant activity than wheat bran powder, pointing towards the feasibility of employing such residues in nutraceutical and functional food applications.

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6. References

[1] M. Elleuch, D. Bedigian, O. Roiseux, S. Besbes, C. Blecker and H. Attia. Dietary fibre and fibre-rich by-products of food processing: Characterisation, technological functionality and commercial applications: A review, *Food Chem.* 2011, 124: 411-421.

[2] M.T.C. Machado, B.C.B.S. Mello, M.D. Hubinger. Study of alcoholic and aqueous extraction of pequi (*Caryocar brasiliense Camb.*) natural antioxidants and extracts concentration by nanofiltration, *J. Food Eng.* 2013, in press, doi:10.1016/j.jfoodeng.2012.12.007.

[3] A.S. Franca, L.S. Oliveira Coffee processing solid wastes: current uses and future perspectives. In: Columbus F (Ed) *Agricultural Wastes*, Nova Publishers, New York, 2009.

[4] C. Ndiaye, S.Y. Xu, Z. Wang, Steam blanching effect on polyphenoloxidase, peroxidase and colour of mango (Mangifera indica L.) slices, *Food Chem.* 2009, 113: 92–95.

[5] N.G. Asp, C.G. Johansson, H. Halmmer, M. Siljestrom, Rapid enzymatic assay of insoluble and soluble dietary fiber. *J. Agric. Food Chem.* 1998, 31: 476-482.

[6] F.A. Hassan, A. Ismail, A.A. Hamid, A. Azlan, S.H. Al-Sheraji, Characterisation of fibre-rich powder and antioxidant capacity of Mangifera pajang K. fruit peels. *Food Chem.* 2011, 126: 283-288.

[7] V.L. Singleton, R. Orthofer, R.M. Lamuela-Raventos, Analysis of total phenols and other oxidation substrates and antioxidants by means of Folin– Ciocalteu reagent. *Method. Enzymol.* 1999, 299: 152–178.

[8] A. Floegel, D.-O. Kim, S.-J. Chung, S.I. Koo, O.K. Chun, Comparison of ABTS/DPPH assays to measure antioxidant capacity in popular antioxidant-rich US foods, *J. Food Comp. Anal.* 2011, 24: 1043-1048.

[9] J.A. Larrauri, P. Rupérez, F. Saura-Calixto, Effect of drying temperature on the stabilitity of polyphenols and antioxidant activity of red grape pomace peels. *J. Agric. Food Chem.* 1997, 45: 1390-1393.

[10] M.C. Garau, S. Simal, C. Rosselló, A. Femenia, Effect of air-drying temperature on physico-chemical properties of dietary fibre and antioxidant capacity of orange (Citrus aurantium v. Canoneta) by-products, *Food Chem.* 2007, 104: 1014-1024.

[11] C. Borchani, S. Besbes, M. Masmoudi, C. Blecker, M. Paquot, H. Attia, Effect of drying methods on physico-chemical and antioxidant properties of date fibre concentrates, *Food Chem.* 2011, 125: 1194-1201.

[12] A. Tseng, Y. Zhao, Wine grape pomace as antioxidant dietary fibre for enhancing nutritional value and improving storability of yogurt and salad dressing, *Food Chem.* 2013, 138: 356-365.

[13] M.S. Soares Júnior, R.C. Reis, P.Z. Bassinello, D.B.C.L. Lacerda, S.N. Koakuzu, M. Caliari, Qualidade de biscoitos formulados com diferentes teores de farinha de casca de pequi (Quality of cookies prepared with different amounts of pequi peel flour), *Pesqui. Agropecu. Trop. (UFG)* 2009, 39: 98-104, (in portuguese).

[14] P. Chantaro, S. Devahastin, N. Chiewchan, Production of antioxidant high dietary fiber powder from carrot peels. *LWT - Food Sci. Technol.* 2008, 4: 1987-1994.

[15] F. Figuerola, M.L. Hurtado, A.M. Estévez, I. Chiffelle, F. Asenjo, Fibre concentrates from apple pomace and citrus peel as potential fibre sources for food enrichment. *Food Chem.* 2005, 91: 395-401.

[16] V.R. Souza, P.A.P. Pereira, F. Queiroz, S.V. Borges, J.D. Souza Carneiro, Determination of bioactive compounds, antioxidant activity and chemical composition of Cerrado Brazilian fruits, *Food Chem.* 2012, 134: 381-386.