Effect of drying on nutritional composition, antioxidant capacity and bioactive compounds of fruits co-products

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Abstract

Fruit coproducts fruit, made of peels, seeds and pulp, discarded during the industrial processing, contain lots of health beneficial compounds, however, high moisture content limits its use. Drying is a low cost and great potential alternative for using. This study aimed to evaluate the phenolic compounds content, in vitro antioxidant capacity and total carotenoid, anthocyanins and vitamin C contents of pineapple (Ananas comosus), banana (Musa sp.), lychee (Litchi chinensis) and papaya (Carica papaya) peels, fresh and oven dried at 55 °C. Phenolic compounds, total carotenoids, anthocyanins, vitamin C contents and the antioxidant capacity of flours, were also significantly higher, indicating that the drying process promoted the concentration of these components, and constitute an excellent alternative to use these coproducts as a source of nutrients.

Keywords: Ananas comosus; Musa sp.; Litchi chinensis; Carica papaya.

Practical Application: Provides waste data that can be used as raw materials due to its high nutritional value, supporting the development of new products.

1 Introduction

Fruit consumption has become increasingly important in the human nutrition due to its nutrient composition and potential health effects (Feliciano et al., 2010). They can be consumed fresh or processed, for example in the form of juices, pulps, jellies, among others.

During processing, various parts of fruits and vegetables such as peels, stems, seeds and pulp are removed, which results in a considerable nutritional loss since these parts contain large amounts of nutritional compounds (Ayala-Zavala et al., 2010). Peels, seeds, and unused pulp of fruits generated in the different processing stages are considered co-products and usually discarded (Ajila et al., 2007).

The amount of co-products resulting from fruit processing can be equal to or even higher than the amount of processed product. For papaya, mango, and pineapple, for example, co-products can represent up to 60% by weight of fruit (Ayala-Zavala et al., 2010).

The manufacturing process can minimize co-products disposal, thus obtaining value-added products (Ayala-Zavala et al., 2010). Fruits and vegetables processing co-products and extraction of compounds to be used as food additives have been extensively studied, as previously reported by Ayala-Zavala et al. (2011).

Furthermore, several authors have reported that these wastes can be used as functional food ingredients, since they are rich in dietary fiber and antioxidant compounds (Sun-Waterhouse et al., 2010). Therefore, it is necessary to investigate the portions not commonly consumed in fruits and their chemical characterization, which can contribute to a better use of these materials (Abdennacer et al., 2015).

Morais et al. (2015) studied phenolic compounds in pulp, seeds, and peels in natura, and found the highest levels in peels. This result is consistent with the study of Ayala-Zavala et al. (2011), who reported that the functional compounds in fruits are located preferably in the peel and seeds, and to a lesser extent in the pulp. Silva et al. (2014) have reported that agro-industrial co-products are good sources of bioactive compounds, and the exploitation of these abundant and inexpensive materials can be done by pharmaceutical and food industries, with opportunities for new nutraceuticals and/or pharmaceuticals, reduction of industrial waste, and lower costs, thus providing positive economic and environmental impacts.

Fruit peels have limited use due to their high moisture content, which favors the proliferation of microorganisms and degradation of chemical compounds (Torres et al., 2010). Drying process is an alternative method of food conservation, once it enables weight reduction, reducing transportation and packaging costs, prolongs the shelf life of the product by reducing the water content, and increases the potential for full utilization of fruits and development of new products (Zambrano-Zaragoza et al., 2013; Queiroz et al., 2015).

Flour made from dried fruits has assumed great importance and attracted the attention of the food industry and consumers (Alves & Perrone, 2015), representing a promising alternative for use of co-products, including fruit peels (Queiroz et al., 2015).
This study aimed to evaluate the nutritional compounds of pineapple, banana, lychee, and papaya peels subjected to drying for the preparation of flours.

2 Material and methods

2.1 Material

Pineapple, banana, lychee, and papaya peels were used. Ripe pineapple (Ananas comosus), banana (Musa sp.), and papaya (Carica papaya) fruits were provided by CEASA - GO (State of Goias Supply Centers) and transported in polyvinyl chloride (PVC) trays to the Laboratory of Food Chemistry and Biochemistry, Faculty of Pharmacy, Federal University of Goias. The fruits were selected for the absence of defects, washed with mild soap and running water, sanitized with sodium hypochlorite solution 100 mL L$^{-1}$ for 15 minutes and drained. Then, the fruits were peeled manually with a knife. Pineapple, banana, and papaya peels were packed in low-density polyethylene bags and subjected to physicochemical characterization.

Previously sanitized lychee (Litchi Chinensis) peels from pulp processing were provided by the company Frutos do Brasil. The peels were packed in low-density polyethylene bags and transported to the Laboratory of Food Chemistry and Biochemistry, Faculty of Pharmacy, Federal University of Goias, for physicochemical characterization.

Approximately two-thirds of the total amount of fruit peels were dried in a forced air circulation oven at 55 °C to constant weight.

After drying, peels were ground in micro knife mill Willye type (Tecnal, TE-648), resulting in four samples, which were encoded as PPF (pineapple peel flour), BPF (banana peel flour), LPF (lychee peel flour), and YPF (papaya peel flour). The remaining amount of peel, coded as PP (pineapple peel), BP (banana peel), LP (lychee peel), and YP (papaya peel), was used in natura, as a control for the physicochemical analyses.

2.2 Methods

Proximate composition

The moisture content was determined by oven drying at 105 °C to constant weight; ash was determined by the gravimetric method of incineration in a muffle at 550 °C, and nitrogen was determined by the Kjeldahl method, considering the conversion factor of 6.25 for crude protein, according to Association of Official Analytical Chemists (2012). Total lipids content was determined by the Bligh & Dyer (1959), and total carbohydrates content was calculated by difference, by subtracting the values obtained for moisture, ash, protein, and lipids from 100, as proposed by the Association of Official Analytical Chemists (2012). The results were expressed as gram per 100 g of sample.

Total energy value

The energy value (TEV) was determined by multiplying the percentage of protein, lipid and carbohydrate contents by the respective energy values of 4, 9 and 4 Kcal (Atwater & Woods, 1896). The results were expressed as kcal per 100 g of sample.

Obtaining ether, ethanolic, and aqueous extracts for determination of total phenolic compounds and in vitro antioxidant capacity

The phenolic and antioxidant compounds were extracted sequentially using solvents with different polarities. For that, 2.5 g sample was mixed with ethyl ether in the dark, in the ratio 1:20 (w/v) for 1 hour, using a magnetic stirrer, at room temperature. The extract was filtered through Whatman #1 filter paper, transferred to a volumetric flask, and the volume was adjusted with the extraction solution. The remaining residue was used for obtaining the subsequent extracts, using alcohol and distilled water under the same conditions of the ether extraction. Then, the extracts were placed in amber glass bottles and stored in a freezer at -18 °C for determination of total phenolic compounds and in vitro antioxidant capacity.

Total phenolics compounds

The total phenolics were determined in the ether, ethanolic, and aqueous extracts in spectrophotometer (Rayleigh, UV-1800) at 750 nm, according to Waterhouse (2002), using a gallic acid standard curve in the range of 5 to 50 mg of gallic acid per L, and the results were expressed as mg of gallic acid equivalents (GAE) per gram of sample.

Antioxidant capacity

In vitro antioxidant activity was determined in the ether, ethanolic, and aqueous extracts using three methods (ABTS, FRAP, and DPPH). The antioxidant activity by ABTS (2,2' azinobis (3-sulfonic acid ethyl benzothiazoline 6)) method was determined according to the methodology described by Rufino et al. (2007). The absorbance was measured in a spectrophotometer (UV-1800 Rayleigh) at 734 nm at 6 minutes after sample addition. A standard curve in the range of 100-2000 uM Trolox was used and the results were expressed as mM Trolox equivalents per gram of sample.

The antioxidant activity by the FRAP method (Ferric Reducing Antioxidant Power) was performed according to Rufino et al. (2006). The absorbance was measured in a spectrophotometer (Rayleigh UV-1800) at 593 nm, using a standard curve of ferrous sulfate (FeSO$_4$) in the range of 500 to 2000 μM, and the results were expressed as mM of FeSO$_4$ per gram of sample.

The antioxidant activity by the DPPH (2,2-diphenyl-1-picrylhydrazyl) assay was determined according to the methodology described by Brand-Williams et al. (1995). The degree of discoloration of the DPPH radical by the action of antioxidants was measured using a concentration of 0.2 mg g$^{-1}$, in a spectrophotometer (UV-1800 Rayleigh) at 517 nm after 20 minutes of reaction. The results were expressed as % discoloration of the DPPH radical.

Total carotenoids

Total carotenoids were determined in the samples PP, BP, YP, PPF, BPF, and YPF, according to the methodology described by Higby (1962). Readings were performed at 450 nm, and the results were expressed as mg carotenoids per 100 g sample.
2.2.7 Total anthocyanins

Total anthocyanins were determined in the LP and LPF samples by the colorimetric method as described by Lees & Francis (1972), with modifications by Barcia et al. (2012). The quantification of anthocyanins was based on molar extinction coefficient of cyanidin-3-glucoside, which is the main anthocyanin present in fruits. The results were expressed as mg of cyanidin-3-glucoside per 100 g sample.

Vitamin C

The vitamin C content was determined by the colorimetric method, as described by Strohecker & Henning (1967). The extraction was performed using oxalic acid under stirring, and after filtration, the quantitation was performed using 2,4-dinitrophenylhydrazine and ascorbic acid as standards. The results were expressed as mg of ascorbic acid per 100 g sample.

2.3 Statistical analysis

The experiment was conducted in a completely randomized design. Analyses were performed in triplicate, with four replications. The results were submitted to analysis of variance (ANOVA) using the SISVAR software. The average results for peels and flours were compared by t-test, and the averages of the different extracts (ether, ethanolic, and aqueous) were compared by Tukey’s test, at 5% significance level. The values were expressed as mean ± standard deviation. The correlations between data were calculated using the Pearson correlation coefficient (r).

3 Results and discussion

The proximate composition of the samples is shown in Table 1.

Moisture is one of the most important parameters for the stability of flours during storage. With respect to the standard established in the current Brazilian legislation (15g 100 g⁻¹) (Brasil, 2005), and evidently lower values than those observed in the peels.

The ash contents of the pineapple, banana and papaya peels (4.83 ± 0.31, 12.82 ± 10.60, 11.51 ± 0.09, respectively) were significantly higher than those of their flours (4.36 ± 0.04, 10.60 ± 0.17, 10.64 ± 0.05).

There were no significant differences in protein content of pineapple, lychee and papaya peels and flours (5.75 ± 0.38, 4.12 ± 0.33, 16.60 ± 1.05 for peels and 5.34 ± 0.09, 4.89 ± 0.86, 16.30 ± 1.26 for flours, respectively). According to Brazilian legislation, for a product to be considered a source of protein, it must contain a minimum of 6 g of protein per serving, which for all types of flours corresponds to 50 g (Brasil, 2012; Brasil, 2003), which allows FCM to be considered a protein source.

The lipid contents of the flours evaluated (1.69 ± 0.18, 9.39 ± 0.26, 3.01 ± 0.13 and 2.70 ± 0.03, for FCA, FCB, FCL and FCM, respectively) were significantly lower than those of the peels (2.14 ± 0.12, 10.08 ± 0.14, 3.75 ± 0.27 and 3.64 ± 0.10, for CA, CB, CL and CM, respectively).

Carbohydrate levels varied from 67.50 ± 0.73 to 89.38 ± 0.30, and represented the major chemical components of the samples evaluated. The flours presented significantly higher carbohydrate levels than the bark, with the exception of LC and FCL, which did not present a significant statistical difference. Foods rich in carbohydrates can be used to energetically enrich food, either by direct consumption or by inclusion in the development of new products (Abud & Narain, 2009).

It is important to remember that the carbohydrate content is represented by the starch, cellulose, hemicellulose, lignin, pectin and other biopolymers present in fruit peels and therefore the fiber contents of the samples evaluated are included in the values found for total carbohydrates.

The total phenolic compounds of peels and flours are presented in Table 2. The phenolic extraction with ethyl ether was not effective, thus the results corresponded to the ethanolic and aqueous extracts. As reported by Fu et al. (2011), the better solubility in ethanol and water demonstrated that these solvents are commonly used for phenolic extraction from plant materials.

The results showed that the content of phenolic compounds was significantly higher in the flours as compared to the peels, indicating the concentration of phenolic compounds during drying. Although the ethanolic extract of the banana peel had insufficient phenolic content for the correct quantification, the result found for the banana peel flour indicates the presence of these compounds in the sample.

Nunes et al. (2016), in a study on the effect of drying on compounds present in guavas, identified a greater diversity of

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Table 1. Proximal composition and total energetic value of fresh shells of pineapple, banana, lychee, papaya and their respective flours (dry basis).

|        | Moisture | Ashes      | Proteins | Lipids   | Carbohydrates | TEV     |
|--------|----------|------------|----------|----------|---------------|---------|
| PP     | 82.05 ± 0.09 | 4.83 ± 0.31 | 5.75 ± 0.38 | 2.14 ± 0.12 | 87.27 ± 0.46 | 391.39 ± 1.10 |
| PPF    | 5.36 ± 0.06  | 4.36 ± 0.04  | 5.34 ± 0.09 | 1.69 ± 0.18 | 88.60 ± 0.18 | 391.01 ± 1.00 |
| BP     | 85.19 ± 0.34 | 12.82 ± 0.59 | 9.60 ± 0.40 | 10.08 ± 0.14 | 67.50 ± 0.73 | 391.12 ± 2.70 |
| BPF    | 9.25 ± 0.17  | 10.60 ± 0.17 | 7.93 ± 0.71 | 9.39 ± 0.26 | 72.00 ± 0.76 | 404.54 ± 1.43 |
| LP     | 71.11 ± 0.35 | 2.75 ± 0.09  | 4.12 ± 0.33 | 3.75 ± 0.27 | 89.38 ± 0.30 | 407.74 ± 1.16 |
| LPF    | 8.45 ± 0.11  | 3.36 ± 0.07  | 4.89 ± 0.86 | 3.01 ± 0.13 | 88.74 ± 0.86 | 401.60 ± 0.73 |
| YP     | 89.83 ± 0.05 | 11.51 ± 0.09 | 16.60 ± 1.05 | 3.64 ± 0.10 | 68.26 ± 1.12 | 372.14 ± 0.63 |
| YPF    | 14.18 ± 0.21 | 10.64 ± 0.05 | 16.30 ± 1.26 | 2.70 ± 0.03 | 70.36 ± 1.27 | 370.95 ± 0.30 |

Values expressed by mean ± standard deviation, in g per 100g of sample. PP = pineapple peel. PPF = pineapple peel flour. BP = banana peel. BPF = banana peel flour. LP = lychee peel. LPF = lychee peel flour. YP = papaya peel. YPF = papaya peel flour. TEV = Total energy value. Equal lower case letters, in the same column, for each sample and its flour, do not differ statistically from each other by the T-test (p < 0.05).
phenolic compounds in fruit powders than in fresh fruits, which was also attributed to the concentration of the components caused by the loss of water during the drying process.

Several factors affect the levels of bioactive compounds in fruits and derivatives, such as the type of crop, climate, variety, and time of year (Deng et al., 2010).

When comparing all extracts, it was observed that, for the peel extracts, the best phenolics extraction was observed for the ethanolic extract when compared to the aqueous extract. As for flours, the aqueous extract exhibited better extraction than the ethanolic extract. This can be due to the water loss leads to a rearrangement of molecules, which can modify its properties, including solubility.

The in vitro antioxidant activity of the samples is shown in Table 2 and was assessed using three methods. For the ABTS assay, the antioxidant activity was significantly higher in flours, when compared with the samples in natura, with the exception of the CL and FCL aqueous extracts. The best antioxidant activities were found in the aqueous extracts, except for the FCL.

Nunes et al. (2016) also observed an increase in antioxidant activity of guava subjected to oven-drying process. The authors reported that the changes in the phenolic profile were due to the drying process, with a wide variation of antioxidant activity in the individual compounds. Heat induces numerous chemical reactions, such as Maillard reaction, Strecker degradation, and hydrolysis of esters and glycosides, leading to generation of new antioxidant compounds, as reported by Tian et al. (2016), which perceived an increase in the antioxidant activity of samples submitted to cooking.

For the FRAP assay, the antioxidant activity was significantly higher in flours, with the exception of the CL, FCL, CM and FCM ether extracts, and CA, FCA, CL, FCL, CM, and FCM ethanolic extracts. The aqueous extracts also exhibited antioxidant activity, except for FCB, CL, and CM.

Opposite behavior was observed in the DPPH assay. The antioxidant activity of peels was significantly higher than the flours, with exception of CA and FCA ethanolic extracts, and CM and FCM ether extracts. The aqueous extracts showed better antioxidant activity, except for FCL and CM.

According to Halliwell (1996), the natural antioxidant defense in fruits and vegetables is associated with three main groups: ascorbic acid and phenolic compounds as hydrophilic antioxidants, and carotenoids as lipophilic antioxidants. However, the antioxidant activity of fruits is affected by several factors, including environmental aspects, ripening, fruit variety, type of extraction solvent, and extraction conditions (Muniz et al., 2006). Almeida et al. (2011) pointed out that the antioxidant potential of fruits is also influenced by the action of different antioxidant compounds with synergistic and antagonistic effects between them.

Table 3 shows the carotenoids concentrations of all samples, except for the LP and LPF, once it was not possible to obtain the extracts, probably because the characteristic color of carotenoids was not observed in these samples. Flours exhibited an increase in total carotenoids, indicating that the heat treatment did not cause degradation of these compounds, which presented increased concentration due to water loss.

The quantification of anthocyanins was carried out in the samples LP and LPF, once their color profile characterized the presence of this pigment. As can be seen in Table 3, higher anthocyanins content was observed in lychee flours (9.57 mg 100 g⁻¹), when compared to peels (1.25 mg 100 g⁻¹), due to pigment concentration during drying.
Queiroz et al. (2015) studied the composition of lychee peel flours and found anthocyanins content of 99.55 mg 100 g⁻¹, which is higher than that found in this study. This difference may be due to darkening of lychee peel, and consequent loss of the characteristic red color after harvesting, which has been attributed to rapid anthocyanins degradation and water loss, besides the activity of oxidative enzymes, such as polyphenol oxidases and peroxidases (Lima et al., 2010).

Vitamin C content (Table 3) of flours were up to 10 times higher than those found in peels. Due to the heat sensitivity of vitamin C, high degradation of this nutrient was expected (Guiamba et al., 2016); however, the results showed high concentration of this vitamin in the samples subjected to drying under the temperature studied.

The values were higher than those of traditional fruits in natura, such as strawberry (71.80 mg 100g⁻¹), guava (218 mg 100g⁻¹), cashew (219 mg 100g⁻¹), and acerola (1457.69 mg 100g⁻¹), the latter considered a source of vitamin C (Freire et al., 2013).

The correlation between the content of phenolic compounds, carotenoids, anthocyanins, vitamin C, and in vitro antioxidant capacity assessed by ABTS, FRAP, and DPPH assays was established through the Pearson correlation test, to determine the main contributors to the antioxidant capacity of peels (Table 4), and flour (Table 5). The phenolic compounds were the main

### Table 3. Total carotenoids, total anthocyanins and vitamin C in pineapple, banana, lychee, papaya and their flours.

|                 | Carotenoids* | Antocianinas** | Vitamin C*** |
|-----------------|--------------|----------------|--------------|
| PP              | 0.009⁰ ± 0.00| -              | 571.55⁰ ± 43.53 |
| PPF             | 0.03³ ± 0.00 | -              | 2823.56° ± 135.97  |
| BP              | 0.003³ ± 0.00| -              | 272.03° ± 36.09   |
| BPF             | 0.04³ ± 0.00 | -              | 1203.41° ± 50.87  |
| LP              | 1.25³ ± 0.14 | -              | 347.87° ± 15.02   |
| LPF             | 9.5³ ± 0.07  | -              | 2110.23° ± 210.90 |
| YP              | 0.06³ ± 0.00 | -              | 210.62° ± 32.29   |
| YPF             | 0.13³ ± 0.00 | -              | 2331.13° ± 257.57 |

Values expressed as mean ± standard deviation, * mg total carotenoids, ** mg cyanidin-3-glucoside, and *** mg ascorbic acid per 100 g sample. PP = pineapple peel. PPF = pineapple peel flour. BP = banana peel. BPF = banana peel flour. LP = lychee peel. LFP = lychee peel flour. YP = papaya peel. YPF = papaya peel. Equivalent letters, in the same column, for each sample and its respective flour, do not differ among themselves by the T test (p <0.05).

### Table 4. Pearson’s correlation coefficients for pineapple (a), banana (b), lychee (c) and papaya (d) peels.

|                 | ABTS | FRAP | DPPH |
|-----------------|------|------|------|
| FRAP            | 0.3433 |      |      |
| DPPH            | 0.2958 | 0.0129 |      |
| Phenolics       | 0.4877* | 0.3025 | 0.0420 |
| Carotenoids     | 0.2515 | 0.0064 | 0.1131 |
| Vitamin C       | -0.5959 | 0.0370 | -0.2561 |

|                 | ABTS | FRAP | DPPH |
|-----------------|------|------|------|
| FRAP            | 0.0432 |      |      |
| DPPH            | 0.3955 | -0.2698 |      |
| Phenolics       | -0.1713 | 0.6134* | 0.0769 |
| Carotenoids     | 0.0223 | -0.3868 | -0.0195 |
| Vitamin C       | 0.0520 | -0.3308 | 0.3683 |

|                 | ABTS | FRAP | DPPH |
|-----------------|------|------|------|
| FRAP            | 0.1602 |      |      |
| DPPH            | 0.5061* | -0.3960 |      |
| Phenolics       | 0.0671 | -0.3232 | 0.2383 |
| Carotenoids     | -0.4254 | 0.0693 | -0.4713 |
| Vitamin C       | -0.1067 | 0.4752* | -0.1237 |

*Significant at p <0.05.

### Table 5. Pearson’s correlation coefficients for pineapple (a), banana (b), lychee (c) and papaya (d) flours.

|                 | ABTS | FRAP | DPPH |
|-----------------|------|------|------|
| FRAP            | -0.1290 |      |      |
| DPPH            | 0.2979 | -0.2013 |      |
| Phenolics       | -0.2795 | -0.2362 | 0.1829 |
| Carotenoids     | -0.0895 | 0.0234 | 0.0756 |
| Vitamin C       | -0.1527 | -0.5623 | 0.3283 |

|                 | ABTS | FRAP | DPPH |
|-----------------|------|------|------|
| FRAP            | 0.1062 |      |      |
| DPPH            | 0.5061* | -0.3960 |      |
| Phenolics       | 0.0671 | -0.3232 | 0.2383 |
| Carotenoids     | -0.4254 | 0.0693 | -0.4713 |
| Vitamin C       | -0.1067 | 0.4752* | -0.1237 |

*Significant at p <0.05.
microcomponents that contributed to the antioxidant capacity of pineapple and lychee peels. Vitamin C, in turn, was the main contributor to the antioxidant capacity of banana and papaya peels, and lychee and papaya flours. The antioxidant capacity of papaya flour was also influenced by the carotenoids content.

The results showed that the microcomponents contributing to the antioxidant capacity varied according to the samples and methods of analysis. This is due to the presence of other compounds, as well as the chemical composition of the samples, once the antioxidant effects may be a result of the sum of individual components exhibiting synergistic or antagonistic effects.

4 Conclusion

The drying process at 55 °C, used for the elaboration of pineapple, banana, lychee and papaya peels, promoted a statistically significant increase in phenolic compounds, total carotenoids, total anthocyanins, vitamin C and antioxidant capacity of samples.

In this way, it can be inferred that drying is an excellent alternative for the use of co-products, for example, for the nutritional enrichment of food products.

Acknowledgements

The authors thank the CAPES and CNPq for the financial support.

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