Physical-chemical characterization, bioactive compounds, and antioxidant activity of pulp and peel of the Jamelão

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ABSTRACT

The high production of jamelão (Syzygium cumini) during harvest and the lack of information about it emphasize the development of new processing technologies for this fruit since it has been proven that its different parts have functional properties. The objectives of this work were to evaluate the physical-chemical characteristics and to determine the content of bioactive compounds and the antioxidant activity present in the pulp and peel of the jamelão in order to assess which one has the greatest industrial potential. For this, the fruit was pulped and separated into peel and pulp. The peel was subsequently dehydrated, resulting in three samples: pulp, fresh peel, and dried peel. It was observed that drying the peel provided an increase in the values of ash, lipids, acidity, and soluble solids to a higher or equal level to those of the pulp. In addition, total phenolic compounds, total flavonoids, total monomeric anthocyanins, and total antioxidant activity of the fresh peel were found to be ten times higher than those of the pulp. Thus, the peel, a by-product of jamelão, has great potential to be used in the food, pharmaceutical and cosmetic industries.

Key words: Syzygium cumini; phytochemicals; antioxidants.

INTRODUCTION

Brazil is a country of great territorial extension and it is considered to have the greatest flora diversity in the world, with approximately 55,000 species cataloged, including a large number of fruit species. However, there are still hundreds of potentially lucrative fruits that are not regularly studied and quite unknown. Thus, getting to know them opens new perspectives for the creation of products and technologies for an expanding market (Santos et al., 2020a).

The jamelão (Syzygium cumini) is a plant that can adapt to different climatic conditions, making its cultivation possible in several parts of Brazil, such as the Northern, Northeastern, and Southeastern regions; featuring several names in Brazilian Portuguese, such as: jamelão, jambolão, azeitona-roxa (purple olive), and jamun (Corrêa, et al., 2018). It is rich in minerals, such as phosphorus and magnesium, vitamin C, carotenoids, flavonoids, ellagic acid, and tannins (Soares & Pereira, 2020).

The high production of jamelão (Syzygium cumini) during the harvest and the lack of information about its processing highlight the adequacy of conventional technologies and the development of new processing technologies, in order to promote a more profitable use, adding value to this fruit (Santos et al., 2018; Neves-Brito et al., 2021).

Different jamelão parts (pulp, peel, and seed) have been studied by other authors due to their functional properties, such as the presence of high levels of anthocyanins, phenolic compounds, flavonoids, and carotenoids, demonstrating the jamelão’s antioxidant, antidiabetic, antifungal, and antibacterial potentials (Corrêa et al., 2018; Brandão et al., 2019; Neves-Brito et al., 2021).
Previous studies on the evaluation of bioactive and nutritional compounds from residues (peels and seeds) of other fruits have also been carried out in recent years as raw material for obtaining antioxidant extracts, thus adding value to a substance that would normally be discarded (Almeida et al., 2020; Aquino, et al., 2020; Silva et al., 2020; Santos et al., 2020a).

Regarding processing, the jamelão fruits have also been used to make products such as jellies (Yenrina et al., 2017), nectar (Soares et al., 2020), powdered and pasteurized juices (Tavares et al., 2017; Oliveira et al., 2016), coloring (Brito et al., 2017), frozen yogurt (Bezerra et al., 2015), fermented drinks (Oliveira et al., 2016), wine (Venugopal & Anu-Appaiah, 2017), and in functional foods (Venugopal & Anu-Appaiah, 2017), and in functional foods frozen at -18 ºC until being used. Before their use, the fruits were submitted to the drying process.

However, to make such products, the food industry uses the pulp and discards the peel, nevertheless, the latter is also rich in phenolic and antioxidant compounds (Brito et al., 2017; Frauches, 2017; Neves-Brito et al., 2021). Thus, our objective is to promote the exploitation of these abundant and cheap materials among the food, pharmaceutical, and cosmetics industries in the development of new nutraceuticals, flours, flavoring extracts, antioxidants, and dyes for incorporation in new products, providing positive economic and environmental impacts (Silva et al., 2020).

Thus, the purpose of the work was to characterize the pulp and peel of the jamelão, both physically and chemically, as well as regarding bioactive compounds and the production of antioxidant extracts from these raw materials, comparing them and evaluating which one has a greater potential for application by industries.

MATERIALS AND METHODS

Raw material

The jamelão fruit studied was purchased in the central market of the Brazilian city of Aracaju, in the State of Sergipe (SE), having been placed in polyethylene packaging and frozen at -18 ºC until being used. Before their use, the fruits were defrosted at 5 ºC and sanitized with sodium hypochlorite at 200ppm. They were then manually peeled, with the pulps being separated from the seeds in a mechanich pulper machine (Itametal- compact model no. 189), with the seeds being then discarded. The samples were divided into pulp and peel in two separate batches, with one sample in natura, which was stored in polyethylene packaging and frozen at -18 ºC, and the pulp sample, with the latter being submitted to the drying process.

Dehydration process for obtaining jamelão peel powder

The jamelão peel samples were weighed and dried in a hot-air oven (Tecnal-model TE 394/2) at a temperature of 40 ºC for approximately 20 hours, until a final moisture content of approximately 7-10% was reached. The dried peels were then crushed in a grinder (Wyle – model TE 650) to obtain the powder, which was stored in a vacuum laminated packaging.

Physicochemical characterization of jamelão residue

The samples of jamelão in natura (pulp and peel) and the peel powder were analyzed in terms of the moisture, ash and ash contents, pH, soluble solids, titratable acidity and color (Zenebon et al, 2008). The analyses were carried out threefold.

Chlorophyll, total carotenoids and β-carotene contents

The contents of these compounds were determined according to the Lichtenthaler (1987), method from the reading of the filtered extract in a spectrophotometer at 646.8 and 663.2 nm for chlorophyll, and at 470; 646.8; 663.2 nm for carotenoids.

Ascorbic acid

Ascorbic acid was determined through the colorimetric method, as established by Cuniff and Association of Official Analytical Chemists (1995).

Extract preparation

In order to determine the content of bioactive compounds and total antioxidant activity, 5g of the pulp and 5g of the peel in natura were separately homogenized with 50 mL of 70% ethanol and submitted to ultrasonic bath for 60 minutes and protected from light. The extracts were subsequently vacuum filtered and concentrated in a Rotary evaporator (model Buchi R-3) at 40 ºC and stored in amber glass vials for further analyses.

Total phenolic compounds (TPC)

Total phenolic compounds were determined according to the traditional Folin-Ciocalteu spectrophotometric method adapted by Swain & Hillis (1959) and modified by Thaipong et al. (2006), with the results expressed as milligrams of gallic acid equivalents (mg GAE / g of dry extract), calculated using a standard curve of gallic acid, drawn for concentrations between (20-160 mg/L), for comparing the results.

Total flavonoids (TF)

The content of total flavonoids was determined according to the method described by Boroski et al. (2015) with some modifications. The results were compared using a standard curve of quercetin (0.0-100 mg / L) (diluted in 70% ethanol).
Total monomeric anthocyanins (TMA)

The levels of TMA were determined using the pH differential method described by Giusti & Wrolstad (2001) with some modifications, calculated as cyanidin-3-glucoside (MW=449.2 g / mol and ε=26.90 L / mol.cm⁻¹) and the results expressed as mg of 3-cy-glucosidde/g of dry material.

Antioxidant capacity

The antioxidant activity of the extracts produced from the samples in natura (pulp and peel) was evaluated using DPPH, ABTS+ and FRAP (ferric reducing/antioxidant power) assays, according to the procedure described by Boroski et al. (2015). The results obtained were compared using standard curves in order to reach the final result.

STATISTICAL ANALYSIS

The mean values of the samples of each assay were compared using Tukey’s test at a 5% significance level, in a completely randomized design using software Assistat 7.7. All analyses were carried out threefold.

RESULTS AND DISCUSSION

Physicochemical characterization of jamelão samples.

The results regarding the values of humidity, ashes, lipids, pH, acidity, and soluble solids are shown in Table 1.

As expected, the pulp has a moisture content that is significantly higher than the peel and that of the peel flour (p < 0.05). Soares & Pereira (2020) found in their study values equal to that obtained in this work, of 84% for the pulp. Santos et al. (2020b) found a slightly higher value of 85.6% for the edible fraction (pulp + peel). In turn, Mussi et al. (2015) obtained a similar ratio between the moisture contents of the residues studied, of 62% for the wet residue and of 4 to 9% for the dry residue.

The drying process significantly increased the ash content present in the peel, probably due to the concentration of minerals to remove the water. In any case, the levels varied significantly among themselves, with the lowest value for the fresh peel. Santos et al. (2020b) reported ash values of the edible fraction of the jamelão of 0.3%, which were compatible with those obtained for both the pulp and the peel of the fruit.

It was also found that the levels of lipids (Table 1) in the jamelão pulp were higher than in the fresh peel (p < 0.05), however, with dehydration, the values were increased to levels equal to the pulp, showing no significant difference between these samples. And although this fruit is not considered a dietary source of lipids, the results obtained were still superior to those reported by Soares & Pereira (2020) and by Santos et al. (2020c).

Regarding the pH parameter, it was found that the pulp showed a significantly higher value than the peels, similar to those obtained by Santos et al. (2020c) and that it was not affected by the drying process. This pH range characterizes this fruit as an acidic food and can favor the growth of fungi, thus, drying can be a good alternative for the stabilization of the product since the reduction in water activity in acidic foods becomes an important ally in the conservation processes of these fruits (Alves, 2019).

The total acidity and the soluble solids content were significantly higher for the peel flour than for the pulp and fresh peel, and as observed for the ash and lipid content, the removal of water promoted the concentration of the existing compounds.

Santos et al. (2020c) explains that the variation in the chemical composition of the fruits may be due to the environmental characteristics to which these fruits were subjected, to the stage of maturation, the availability of nutrients in the different soils, to the varieties of trees in each region, and the quantity of rain during the harvest.

Table 2 displays that the results of the colorimetric analysis show that there was a significant difference between the studied samples for all the analyzed color parameters. However, the pulp and the raw peel presented closer values when compared to the flour, certainly due to the drying process.

Therefore, the values indicate that, for luminosity (L), the fresh peel and the pulp presented higher values considering the percentage of water contained in them. However, this value was reduced by almost 50% with the removal of water from the peel, thus justifying the darker color of the flour. The same can be observed in the b * coordinate since this sample lost luminosity and darkened with dehydration, ceasing to tend towards yellow and approaching blue.
However, for parameters $a^*$ and $c^*$, which indicate red direction and color intensity, respectively, there was an inversion, where the highest values were observed for flour, considering that drying at lower temperatures promoted the removal of water and an accumulation of anthocyanin pigments, a feature of this fruit. These results confirm the reports by Tavares et al. (2016), who concluded that the color of the jabuticaba ($Syzygium cumini$) varies from green, when unripe, to bluish-purple when ripe.

The values of hue gradients ($H$) found in this work for pulp and peel were around 55°, while for the flour it was close to 0°. And, according to the literature, values that are 360 degrees away indicate a greater presence of blue tones and when close to 0° they indicate red tones (Soares & Pereira, 2020; Soares, 2015). Thus, it can be said that the jabuticaba fruits have a red, purple, or bluish hue and that this is an indicator that they have anthocyanins in their composition.

**Bioactive compounds and antioxidant activity present in jabuticaba samples.**

In Table 3 it is possible to observe that, except for vitamin C, in all other contents the results of the peel were significantly superior to those of the pulp.

Regarding the β-carotene content, Meneses et al. (2018) identified the presence of this compound in different tropical fruits grown in Brazil and observed that mango, guava, and acerola, are excellent sources of this carotenoid, whose contents varied from 215 to 880 µg/100g. Comparing these contents with those obtained for the peel of jabuticaba in this research it can be said that the peel of this fruit is a great source of β-carotene.

Regarding vitamin C, Santos (2017) stated that this is the most abundant vitamin in the jabuticaba. In this work, it was observed that drying did not influence ($p < 0.05$) the contents present in the peel, however, these samples had significantly lower content (approximately 20mg/100g) when compared to the pulp (47.71mg/100g).

In addition to β-carotene and ascorbic acid, the jabuticaba has a high content of other functional compounds, such as phenolic compounds, flavonoids, and anthocyanins (Table 4), on which it was found that the peel has approximately ten times more of these compounds than the pulp, thus showing that this residue might be underutilized by industries.

Singh et al. (2016) reported similar results of total phenolic content (TPC) to those found in the present work. However, several authors have reported lower values, such as those of Soares (2015), Brandão et al. (2019), Santos et al. (2020c), and Tavares et al. (2017), who verified levels of 208mg/100g in their studies; from 312 to 434.8 mg/100 g and from 1.56 mg/g to 1.22 mg/g GAE, respectively.

Pacheco (2015) reported that jabuticaba was the fruit that presented the highest TPC value among the fruits studied, confirming its functionality and corroborating the results obtained in this work.

Neves-Brito et al. (2021), reported for jabuticaba peel flour values ranging from 67.66 mg/ g GAE to 90.98 mg / g GAE, these values lower than those obtained in the present study for fresh peel, and greater than pulp.

The variation in the content of phenolic compounds found in the literature can be explained according to the humidity, soil type, climate, to variation in maturity, to genetic differences, or post-harvest storage conditions. Therefore, the method of preparing the extract, differences in the extraction solvent, extraction time, and the base used to measure (g extract or g material) are also sources of variation (Santos, 2017; Santos et al., 2020c; Neves-Brito et al., 2021).

The levels of flavonoids present in the peel were significantly higher ($p < 0.05$) than those found in the pulp (Table 4), possibly due to the greater presence of

### Table 2: Analysis of the color parameters of the jabuticaba samples studied

| Sample      | Parameters |
|-------------|------------|
|             | $a^*$      | $b^*$      | $c^*$ | $L$     | $H$    |
| Pulp        | 6.000±0.900b | 7.675±0.153a | 9.575±0.611b | 29.675±0.709b | 53.950±3.943a |
| Peel        | 2.275±0.419c | 3.225±0.411b | 3.975±0.171c | 45.175±0.713a | 58.266±2.460a |
| Peel powder | 16.775±0.618a | 0.250±0.251c | 16.775±0.618a | 23.575±0.478c | 1.133±0.750b  |

* Values in each column followed by the same letter do not differ statistically at a 5% significance level.

### Table 3: Mean values from the analysis of chlorophyll, carotenoids, β-Carotene and Vitamin C, present in the samples of jabuticaba fruit

| Sample      | Chlorophyll Total (mg/100g) | Carotenoids (mg/100g) | β-Carotene (mg/100g) | Vitamin C (mg/100g) |
|-------------|-----------------------------|-----------------------|----------------------|---------------------|
| Pulp        | 1.24±0.04b                  | 1.09±0.02c            | 0.52±0.00b           | 47.71±3.73b         |
| Peel        | 5.39±0.81a                  | 7.71±1.55a            | 3.48±0.57a           | 20.33±6.00a         |
| Peel powder | 5.46±0.53a                  | 5.03±0.61b            | 2.59±0.34a           | 20.91±3.60a         |

* Values in each column followed by the same letter do not differ statistically at a 5% significance level.
anthocyanins (a class of flavonoid compounds) in the peel of this fruit.

Bezerra (2015) reported values similar to those obtained in this work for the pulp. While higher values were reported by Singh et al. (2016) and lower ones by Tavares et al. (2017).

In this work, we verified anthocyanin values of 113.25 and 1040.2 mg/100 g (Table 4) for pulp and peel, respectively.

Results close to those obtained for anthocyanins found in the jamêlão pulp were verified by Kapoor & Ranote (2015) and Soares & Pereira (2020). While Mussi et al. (2015) found a lower value, 63 mg/100 g, and Branco et al. (2016) found a higher value of 216 mg/100 g. In comparison to the peel, Neves-Brito et al. (2021) and Frauches (2017) found values lower than this research, 886.49 and 575.2 mg/100 g respectively, probably due to the use of powdered peel.

Based on the results found and the works mentioned, it appears that the values of anthocyanins, flavonoids, and phenolics vary greatly between studies since there is a range of intrinsic and extrinsic factors that can act in their composition and determination. However, the values found for the samples, mainly for the peel, show that they are rich in bioactive functional compounds, and with that, the need for further research with the peel of this fruit is verified since it is normally discarded by the agribusiness and it could be incorporated into other products, adding value to them.

The DPPH, ABTS, and FRAP essays were also used in this work to assess the antioxidant capacity of fresh pulp and peel. In Table 5 it is shown that for all the methods analyzed, the peel had significantly higher antioxidant activity than the pulp (p < 0.05), which was already expected due to the results obtained for the bioactive compounds.

The EC50 values, obtained by linear regression, for the pulp and peel jamêlão extracts, showed a high coefficient of determination, i.e., \( R^2 = 0.995 \). Also, it was found that the percentage of DPPH inhibition varied from 6 to 23% by changing the volume of extract used from 25 to 100 \( \mu L \); and that the value needed to inhibit 50% of the radical was 246.85 and 55.05 \( \mu g/\text{g of b.s extract} \) (Table 5) for the pulp and peel, respectively.

Higher percentage values were reported by Kapoor & Ranote (2015) who found 91.83% and 88.6% antioxidant activity for fresh jamêlão and processed pulp, respectively, when compared to the DPPH radical. But despite these authors obtaining higher percentages of inhibition, the linearity of the data obtained here applying different concentrations of the extract provided a good antioxidant activity (low EC 50% values) by this method (246.85 \( \mu g/\text{g} \)), which was not noted by Soares (2015), who reported that the EC50 concentration ranged from 639 to 1138 \( \mu g/\text{mL} \).

Regarding the peel, Neves-Brito et al. (2021) realized that the concentration required to inhibit 50% of the DPPH radical was 305.95 \( \mu g/\text{g} \), which is higher than that found in the present work.

On the other hand, when comparing the DPPH methodology with ABTS, it appears that ABTS radicals are more reactive than DPPH ones and, unlike the reaction with DPPH radicals, which involve transferring H atoms, reactions with radicals ABTS + involve an electron transfer process.

Corroborating this fact, it was found in this work that the ABTS method enabled better results in the evaluation of the antioxidant activity of jamêlão extracts than the DPPH method, in agreement with the work of Singh et al. (2016). Still, among the three methods evaluated in the present study, the FRAP method was the one that stood out, considering that it exhibited higher values of antioxidant activity, as demonstrated by Neves-Brito et al. (2021).

Table 4: Mean values of the bioactive compounds: total phenolic compounds (TPC), Total flavonoids (TF) and Total monomeric anthocyanins (TMA)

| Sample | Phenolics (mg GAE/g) | Flavonoids (mg QE/g) | Anthocyanin (mg CE/100g) |
|--------|---------------------|---------------------|-------------------------|
| Pulp   | 14.81±1.13b         | 1.74±1.15b          | 113.25±0.85b            |
| Peel   | 173.9±3.92a         | 10.54±0.53a         | 1040.2±2.52a            |

* Mean values in each column followed by the same letter do not differ statistically at a 5% significance level. GAE=gallic acid equivalent; QE= quercetin equivalent; CE=cyanidin equivalent.

Table 5: Antioxidant activity of jamêlão pulp and peel in natura of dry weight (dw)

| Sample | DPPH µg/g of extract d.w. (EC 50 %) | ABTS (µmol de TE)/g of extract d.w. | FRAP (µmol of Fe (II))/g of extract d.w. |
|--------|-----------------------------------|------------------------------------|------------------------------------------|
| Pulp   | 246.85±59.90a                     | 159.32±13.24b                     | 2485.17±75.90b                           |
| Peel   | 55.05±8.39b                       | 1705.69±139.84a                   | 4681.19±167.21a                          |

* Mean values in each column followed by the same letter do not differ statistically at a 5% significance level.
Thus, the high antioxidant activity found in jamelão may be due to flavonoids, as well as to other phenolic compounds that are not related to this class. Also, the region’s climate may have directly influenced the amount of the compounds present in the fruit since under higher temperatures, as in the region where the research was carried out, plants tend to suffer more stress and, to combat this factor, greater amounts of functional compounds are generated since they also function as a protective barrier.

**CONCLUSION**

In summary, we can conclude that the pulp and peel of the jamelão are rich in bioactive and antioxidant compounds. However, the peel showed results about ten times higher than those of the pulp.

Therefore, the peel, a by-product of jamelão, has a better application potential to be used in the food industry in the form of flour and extracts for incorporation in products such as breads, cakes, and cookies, turning common products into nutritive ones. In addition, it can also be used in the pharmaceutical and cosmetics industries, adding value to this raw material, and contributing to reducing waste and environmental impact.

Future studies to develop new products with the peel flour and to evaluation of its sensorial acceptability with new temporal methods and trained tasters are encouraged.

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