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ORIGINAL PAPER

Evaluating the nutritional and bioactive composition of grape, jaboticaba, and hibiscus by-product flours as potential ingredients for the food industry

Maria Olivia dos Santos Oliveira[1,2] · Gabriel Luis Castiglioni[1] · Julião Pereira[1] · Mariana Alencar da Macena[2] · Rômulo Alves Morais[2,3] · Glêndara Aparecida de Souza Martins[2,3] et al. *[full author details at the end of the article]*

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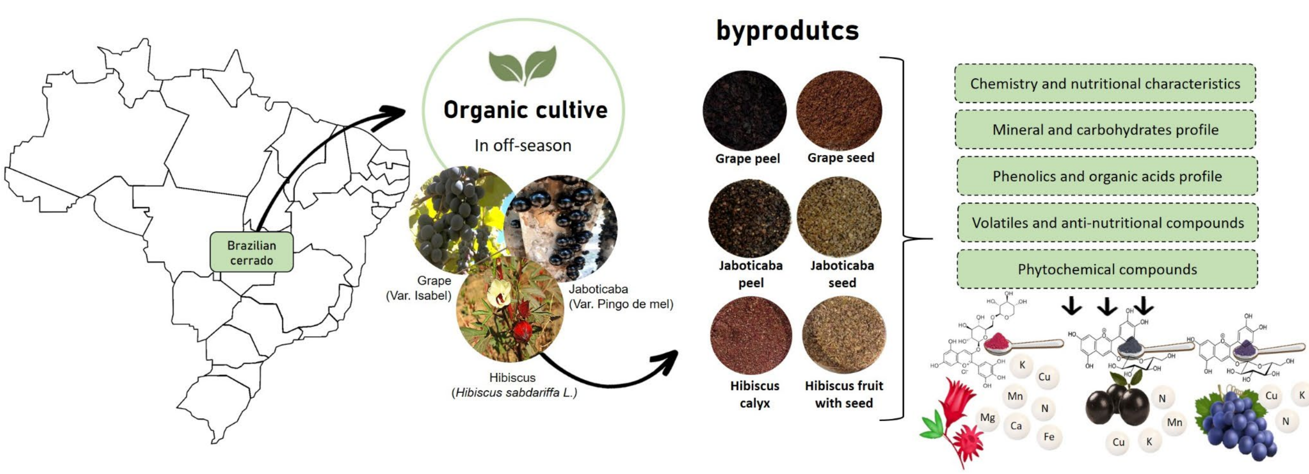
Abstract
Grapes, jaboticaba, and hibiscus are renowned for their antioxidant properties, which contribute significantly to human health. However, after industrial processing, their peel and seeds are often discarded as waste, representing up to 50% of the raw material. This study aimed to evaluate the physicochemical and nutritional composition, mineral, carbohydrates, organic acids, and phenolic profile, in addition to assessing the anti-nutritional compounds, phytochemical, and volatile compounds present in flours made from the peels and seeds of grapes, jaboticaba, and hibiscus. Moisture content ranged from 8.24 to 14.86 g/100 g, with low water activity (0.29–0.56) ensuring extended shelf life. Hibiscus flours were particularly rich in protein, providing up to 31.18% of the recommended daily intake. Grape seed oil stood out for its high unsaturated fatty acid content, while the ash content (up to 9.28 g/100 g) suggests potential for mineral fortification. Key minerals included nitrogen (658–2327 mg/100 g), potassium (480–2080 mg/100 g), and iron (4.92–15.67 mg/100 g) especially abundant in hibiscus flour, highlighting its potential in combating anemia. Phytochemical analysis revealed high levels of carotenoids and flavonoids, known for their antioxidant properties, along with significant anthocyanin and tannin concentrations. Finally, phenolic compounds, such as catechin and gallic acid, were widely detected, with emphasis on catechin in grape peel flours (149.30 mg/100 g) and ellagic acid in jaboticaba peel flours (41.40 mg/100 g), known for their antioxidant and anti-inflammatory properties. The results suggest that grape, jaboticaba, and hibiscus byproduct flours offer substantial nutritional and functional benefits, with promising applications in food industries.

Graphical Abstract



Keywords Byproducts · Waste · Flour · Nutritional composition · Phytochemical compounds · Volatile compounds

✉

Introduction

Changes in lifestyle and consumer habits translate into new requirements and needs. Nowadays, people seek agility in preparing food containing nutrients and compounds that positively affect their health [1]. The attribution of nutritional and sustainable values positively influences consumers choice of food [2, 3]. Given this, consuming foods rich in phytochemicals with antioxidant potential has been promoted, as they can control auto-oxidation, interrupting the propagation of free radicals and directly inhibiting their formation, consequently reducing oxidative stress. Therefore, consuming fruits, vegetables, and nuts reduces the risk of some chronic and degenerative diseases [4, 5].

In this sense, some fruits and vegetables have aroused interest in human nutrition as a source of natural antioxidants and, consequently, an important role in health. Among these fruits, we can highlight grapes (*Vitis labrusca*), jaboticaba (*Plinia cauliflora*), and hibiscus (*Hibiscus sabdariffa*). Despite belonging to different botanical families, these three fruits are widely recognized for their high levels of bioactive compounds, such as anthocyanins and flavonoids, which contribute to their vibrant colors and antioxidant properties [6–8]. Grapes and jaboticaba are rich in phenolic compounds, which help prevent cardiovascular diseases, while hibiscus, known for its diuretic capacity, is also a source of natural antioxidants. Furthermore, the three fruits are used both in food and in folk medicine, being transformed into juices, teas, and natural supplements, with beneficial effects on health [8–10].

After processing these fruits in the industry, peels and seeds are considered waste and are generally discarded. The production of grape juice and wine, for example, results in a large amount of waste, representing 10 to 30% of the initial weight of the fruit [11]. The peel and seeds of the jaboticaba represent approximately 50% of the total fruit [12]. Given the above, the underutilized portions of the grape, jaboticaba, and hibiscus fruits exhibit attractive properties for the pharmaceutical, cosmetics, and food industries. They can be incorporated as natural ingredients in various products, acting multifunctionally in different processes and as a natural pigment [13]. In addition to reducing food waste, this practice can diversify menus and increase the variety of nutrients ingested, thus generating new business opportunities for the food industry and family farming and adding value to the final products.

Therefore, fruit waste emerges as a sustainable solution to several environmental, economic, and public health problems by integrating new bioactive compounds into the production chain and minimizing ecological impacts [14, 15]. This waste, often improperly discarded, is rich in nutrients and functional compounds, such as fibers, polyphenols, flavonoids, and essential oils, which can be extracted and used in the food, cosmetics, and pharmaceutical industries. In addition, processing these by-products contributes significantly to minimizing disease vectors, such as flies and rodents, which proliferate in environments with decomposing organic matter [16, 17]. The correct reuse of waste thus reduces the risk of disease transmission and health problems in urban and rural areas. In this way, the sustainable use of waste favors the circular economy and protects the environment, ensuring greater food security and the preservation of ecosystems [18, 19].

However, complete information on this waste, its availability, potential uses, comparisons and restrictions on use, particularities, and influence of cultivation outside the harvest period still need to be fully elucidated. Given this, this study's main objective is to evaluate the nutritional composition of grape, jaboticaba, and hibiscus peels and seeds. They are investigating the levels of antioxidants, bioactive compounds, and antinutritional compounds in grape, jaboticaba, and hibiscus peels and seeds. Furthermore, we seek to evaluate the potential of these by-products as a source of natural pigments for application in the food industry, aiming to expand the options for natural dyes and maximize the use of agro-industrial waste.

Materials and methods

Plant materials

Fresh grape berries (cultivar Isabel) were harvested ripe before the harvest period (May, beginning of the dry season) in Itaberaí– GO, Brazil (Latitude: 16º 01' 13" S, Longitude: 49º 48' 37" W). Fresh jaboticabas (Honey Drop cultivar) were harvested ripe after the harvest period (December, rainy season) in Hidrolândia-GO, Brazil (Latitude: 16º 57' 44" S, Longitude: 49º 13' 41" C). The hibiscus was collected from a Family Farmers Association, located in Bela Vista de Goiás– GO, Brazil (Latitude: 16º 58' 22" S, Longitude: 48º 57' 12" W) after the average fruiting period (July, dry season). All raw materials were produced organically, free of pesticides. All raw materials were harvested in 2022 and 2023. After collection, the vegetables were packaged in low-density polyethylene packaging and transported for processing. The ripe grapes and jaboticabas were selected (removing impurities and damaged fruits), washed in running water, sanitized in a sodium hypochlorite solution at a concentration of 200 ppm for 15 min, and then rinsed. The grape and jaboticaba fruits were pulped in a pulper (Macanuda DM-SP1 model), where rotary sieves separated the pulp from the peel and seeds. The samples were dried at 45 °C in an oven with forced air circulation. Subsequently, seeds and calyx for the hibiscus were separated and ground in a mill (TE-633, TEC MILL), obtaining powders with a particle size of 20 mesh. Finally, the flours obtained were packaged in low-density polyethylene bags and stored in a freezer at −18 °C until analysis.

Nutritional composition and physicochemical parameters

Nutritional composition and other analyses were performed on grape peel flours (GPF), grape seed flours (GSF), jaboticaba peel flours (JPF), jaboticaba seed flours (JSF), hibiscus calyx flours (HCF), and hibiscus fruit flours with seeds (HFFS). The nutritional composition and physicochemical characteristics of buritirana pulp, shells, and seeds were determined according to the Association of Official Analytical Chemists [20]: moisture (925.09), ash (923.03), lipids (920.85) recovered with hexane for six hours, proteins (920.87), fiber (991.43), carbohydrates by difference. The total energy value was estimated considering the conversion factors of 4 kcal/g for proteins and carbohydrates and 9 kcal/g for lipids and expressed in kcal/100 g of the sample. Titratable acidity (947.05) and pH (981.12). The activity water (*aw*) was determined for termoconstanter electric hygrometric (Aqualab CX-2). Color analysis was performed at 25 °C using a digital colorimeter (Minolta CR4000) Light source in L^*, a^*, b^*, Chroma, and ºHue color space in the CIELAB system. All analyses were performed in sextuplicate, and results were expressed as mean ± standard deviation.

Mineral profile

The minerals were extracted through two digestion methods: nitro-perchloric, with a controlled temperature of 210 ºC, and sulfuric, with a controlled temperature of 350 ºC, following the methodology described by Borges et al. [21]. The first digestion aimed to analyze the levels of magnesium (Mg), potassium (K), phosphorus (P), boron (B), sulfur (S), copper (Cu), manganese (Mn), zinc (Zn), and iron (Fe). The product of the second digestion was used to detect total nitrogen by distillation in a micro-Kjeldahl. Phosphorus, boron, and sulfur were quantified using a molecular absorption spectrophotometer (model 700 plus, FEMTO, São Paulo, Brazil), operating in the light range of 660 nm for the phosphorus mineral. In comparison, boron and sulfur were analyzed in the light range of 420 nm. Potassium was detected by flame photometry (Model 400, Corning, New York, USA), while magnesium, zinc, copper, and iron were measured using a flame atomic absorption spectrophotometer (Model AAnalyst-400, Perkin Elmer, Waltham, USA). The results were expressed as mean ± standard deviation (mg/100 g).

Antinutritional compounds

Cyanogenic compounds were analyzed using the Guignard test, where plum seeds were used as a control to compare the presence of these compounds since they contain cyanogenic glycosides, precursors of hydrocyanic acid [22]. The results were expressed in the absence or presence of cyanogenic compounds. The total oxalate content was determined according to Pinheiro et al. [23]. In this case, oxalic acid was hot extracted with sulfuric acid, precipitated, and quantified by titration of oxalate with potassium permanganate. The results were mean ± standard deviation (mg/100 g).

Ratio minerals determination

Mineral ratios are often more important than individual mineral levels themselves because they help determine nutritional interrelationships and also provide information about the many possible factors that may be represented by a disruption of their ratios, such as disease states, physiological and developmental factors, and the effects of diets [24]. The mole of minerals was determined by dividing the mass of minerals by their atomic weight (K = 39 g/mol, Na = 23 g/mol, *P* = 31 g/mol, Fe = 56 g/mol; Zn = 65 g/mol; Ca = 40 g/mol) and then the mineral ratio was calculated by dividing the mineral of the first mole by the mineral of the second mole [24]. The critical factor for mineral ratios will be compared with the standard reference, which is considered acceptable.

Phytochemicals

The method described by Lee and Francis [25] was followed to determine total anthocyanins and flavonoids. First, 1.0 g of the sample was weighed, and then 10 mL of the extracting solution of 95% ethanol acidified with 1.5 N HCl in the proportion (85:15 *v/v*) was added; the samples were then homogenized for 1 min in a vortex, and then placed in vials wrapped in aluminum foil, left to rest overnight at 4 °C. Immediately after, the material was filtered, and readings were taken for anthocyanins ($\lambda = 535$ nm) and flavonoids ($\lambda = 374$ nm) in a digital spectrophotometer (Model UV-1800, Beijing Rayleigh Analytical Instrument Corporation, China). Carotenoid content was determined according to Kimura et al. [26]. Briefly, 1.0 g of the samples were weighed and macerated with 30 mL of the solvent. The mixture was then added to a separatory funnel containing 50 mL of petroleum ether and filtered thrice until the residue became colorless. Then, 50 mL of acetone was added to the extract, followed by carefully adding distilled water (300 mL). The absorbance of the extract was read on a digital spectrophotometer. The absorbance was then measured at 450 nm, and the molar extinction coefficient of β-carotene in petroleum ether (2592) was used for calculation.

Chlorophyll was extracted and determined by the methodology proposed by Ferreira et al. [27] with modifications. For the extraction of chlorophyll pigment, 2 g of the samples were diluted with 40 mL of 80% acetone and 5 mg of $CaCO_3$ (calcium carbonate) macerated for approximately 1 min and transferred to a 25 mL flask wrapped in aluminum. The sample was filtered, and the reading was performed using a spectrophotometer with a wavelength of 652 nm. The total tannin content was determined according to the methodology of Swain and Hills [28]. For extraction, 1.5 g of the samples were added to 20 mL of distilled water at 80 °C/5 min with stirring. After this process, filtration was performed, and the extract obtained was used for analysis. Using the Folin-Denis reagent and saturated sodium carbonate, the reading was performed in a digital spectrophotometer at 760 nm. All results were expressed as mean ± standard deviation (mg/100 g).

Carbohydrates profile and organic acids

To analyze the sample's carbohydrate and organic acid profile, 2.5 g was diluted in 30 mL of Milli-Q water. The samples were then homogenized and left for 4 h at room temperature (25ºC). After this process, the samples were centrifuged. Then, they were filtered through a hydrophilic PTFE (polytetrafluoroethylene) filter with a porosity of 0.22 µm and kept at -18 ºC until being injected into the chromatograph [15]. The determination and quantification of carbohydrates and organic acids were performed by high-performance liquid chromatography - HPLC, under the following analysis conditions: Agilent equipment, model 1260 infinity II, equipped with a refractive index detector-RID at 40 ºC, using a Supelcogel C610H chromatographic column (30 cm × 7.8 mm) - Sigma-Aldrich, Supelguard C610H pre-column (5 cm × 4.6 mm) - Sigma - Aldrich®, the mobile phase used was H_3PO_4 0.1% (deionized water and phosphoric acid), with a flow rate (mobile phase flow) of 0.5 mL/min, total run time of 18 min and oven temperature of 40 ºC. Glucose, xylose, fructose, cellobiose, and arabinose were used as carbohydrate standards; on the other hand, citric, ascorbic, malic, and tartaric acids were used as standards for identification of organic acids; the compounds were identified by comparison with the retention time of each one. The results were expressed as mean ± standard deviation (mg/100 g).

Phenolic profile

The phenolic compound profile was extracted from the samples using the method proposed by Sousa et al. [14]. For extraction, 1 g of the samples was diluted in 30 mL of 70% ethanolic solution and subjected to an ultrasonic bath at room temperature for 25 min. Subsequently, the samples were centrifuged and filtered using a hydrophilic PTFE filter (0.22 µm). Stock solutions containing different phenolic acids, such as caffeic, chlorogenic, ellagic, and gallic acids, as well as catechin and flavonoids such as kaempferol, myricetin, naringin, quercetin, and rutin, were prepared in methanol at a concentration of 10 mg/mL, and standards were used to compare retention times. The compounds were analyzed using an HPLC-DAD chromatographic system (Agilent model 1260 Infinity II) with a diode array detector. Compounds were identified based on absorption wavelengths. Chromatographic separation was conducted on an Agilent® Zorbax C_{18} column (250 mm × 4.6 mm, 5 µm), with a total run time of 30 min. Method parameters included column temperature maintained at 30 °C, mobile phase A containing 5% formic acid, and mobile phase B consisting of acetonitrile, with a flow rate of 0.8 mL/min. Needle washing was performed with 100% acetonitrile, and the injection volume was 20 µL. The chromatographic gradient used was: 0 min (10% B); 3 min (15% B); 18 min (40% B); 24 min (40% B); 27 min (66% B); 33 min (70% B); and 40 min (80% B). Individual injections confirmed the retention times of all standard compounds, and the results were expressed in µg/g.

Headspace– Gas chromatography coupled with mass spectrometry analysis (HS-GC-MS)

The analyses were performed on a gas chromatograph (Shimadzu Nexis GC2030) coupled to a mass spectrometer (Shimadzu QP2020 NX), equipped with a capillary column (SH-Stabilwax-MS: 30 m × 0.25 mm × 0.25 µm). The samples were previously heated via headspace at 80 °C for 30 min, and 2 g was injected into the chromatograph. The split mode was used with a 10:1 ratio and 3 min equilibration. The oven temperature was initially programmed at 40 °C/min and held for 1 min. The temperature was increased by 5 °C/min to 160 °C; then it was increased by 10 °C/min to 250 °C and held for 15 min. The total analysis time was 52 min. Helium 5.0 was used as a carrier gas, with a pressure of 45.1 kPa, a flow rate of 0.94 mL/min, and a linear velocity of 35 cm/s. The injector, interface, and ion source temperature were maintained at 220 °C. The mass spectrometer in scanning mode recorded ions in the 20 to 400 m/z range with a scan time of 150 ms. Compounds were identified when the retention time corresponded to at least 80% of those available in the computerized library.

Fourier transform infrared spectroscopy (FTIR-ATR)

Fourier transform infrared spectroscopy analyzed and compared the samples' chemical structures. For this analysis, 2 g of the samples were subjected to infrared radiation by the Frontier equipment in the spectral region range of 4000–500 cm^{-1}, resolution of 4 $cm^{-1,}$ and 20 scans/min. The resulting FT-IR spectral pattern was then analyzed and combined with the corresponding characteristic functional and bonding groups.

Statistical analysis

A completely randomized design (CRD) was conducted in duplicate, with 3 replicates each. All analyses were performed in triplicate. Differences, when necessary, were detected by Tukey's test for comparison of means at a significance level of 5% ($p < 0.05$) using the Sisvar® software.

Results and discussion

Nutritional composition, physicochemical analysis, and mineral profile of the flours produced

Table 1 presents the results regarding the nutritional composition, physicochemical characterization, mineral profile, and antinutritional properties of flours prepared from the different samples. Moisture content ranged from 8.24 g/100 g (GSF) to 14.86 g/100 g (GPF). The flours exhibited low water activity (aw), ranging from 0.29 to 0.57, below the threshold of 0.6. This low aw extends shelf life by reducing the availability of free water necessary for microbial growth. These values also allow the flours to be stored at room temperature, as moisture content and water activity are directly related to stability, quality, composition, and yield [29].

The protein content of the flours ranged from 2.81 to 17.46 g/100 g, with the flours obtained from the processing of Hibiscus parts having the highest concentrations of 12.59 g/100 g for HCF and 17.46 g/100 g for HFFS, respectively (Table 1). A healthy adult's recommended daily protein intake varies worldwide, but the average is 0.8 g per kilogram of body weight per day. Thus, a healthy person weighing 70 kg requires 56 g per day [30]. Therefore, 100 g of HFFS would meet approximately 31.18% of the required daily protein intake. On the other hand, the lipid content ranged from 0.38 to 8.65 g/100 g. These values are lower than those found in lipid-rich foods such as baru (42.00 g/100 g), chia seeds (29.70 g/100 g), or avocado (64.09 g/100 g) [31–33]. GSF (8.65 g/100 g) may be potentially promising for products that seek to provide a moderate amount of lipids and present the desired sensory and nutritional characteristics since, according to Sabir et al. [34], grape seed oil has in its composition primarily unsaturated fatty acids, such as linoleic acid (69.60%), followed by oleic acid (31.20%) and palmitic acid (12.90%).

Regarding the ash content, it was observed that GPF, followed by HCF, presented the highest results (9.28 and 6.43 g/100 g, respectively) (Table 1). These results indicate that these flours may be rich in minerals, which can add nutritional value and be used to develop new formulations. GPF, with the highest ash content (9.28 g/100 g), demonstrated especially promising potential for fortifying foods with essential minerals. On the other hand, the crude fiber content ranged from 3.60 to 53.57 g/100 g. The recommended dietary intake (RDI) of fiber for healthy adults is between 25 and 35 g in a diet with 2,000 kcal [5]. Therefore, the consumption of GSF (53.57 g/100 g) and HFFS (27.31 g/100 g) represents more than 100% of the recommended fiber intake. GSF exhibited the lowest carbohydrate content (17.66 g/100 g) and the lowest total energy value (181.28 g/100 g), a good alternative for people with a diet rich in fiber and other compounds and low caloric intake. Foods with low carbohydrate and high fiber content tend to have a lower glycemic index, resulting in a slower and more gradual increase in blood sugar levels [35]. This prevents glycemic spikes, controls blood glucose, and benefits people with diabetes or insulin resistance [36]. Furthermore, the high fiber content increases satiety and acts as a prebiotic, feeding beneficial bacteria in the intestine [35].

Beyond moisture and water activity, pH and acidity are important parameters in determining food preservation [37]. The pH and titratable acidity values ranged from 1.88 to 4.78 and 1.53 to 8.7 g of citric acid/100 g, respectively (Table 1). Hibiscus calyx flours (HCF) stood out for their high acidity, followed by JPF, HFFS and GPF. High acidity can help preserve the product since it limits the growth of pathogens; however, in a possible technological application of the flour, pH and acidity adjustments may be necessary to meet specific formulation, sensory, texture, or stability requirements of the final product [38]. Furthermore, some chemical reactions, such as fermentation or the effectiveness of certain preservatives, depend on a specific pH [39].

Luminosity (L^*) is related to the amount of light reflected by the surface of the flours. Given this parameter, it can be observed that the lightest flours were those from seeds, which also presented higher values for the parameters a^* (reddish tones) and b^* (yellowish tones), except for the hibiscus seed flour, which tended more towards the yellowish tone. The grape and jaboticaba peels were darker, with

Table 1 Nutritional composition, physicochemical characteristics, and mineral profile of flours made from grape, Jaboticaba, and hibiscus byproducts

Parameters (g/100 g)	Samples					
	GPF	GSF	JPF	JSF	HCF	HFFS
Moisture	14.86±0.02	8.24±0.01	10.51±0.08	11.02±0.05	11.92±0.02	10.15±0.01
Protein	2.81±0.19	6.19±0.32	8.06±0.39	6.82±0.23	12.59±0.45	17.46±0.58
Lipids	0.90±0.07	8.65±1.34	0.57±0.01	0.38±0.15	1.09±0.05	3.97±0.12
Ash	9.28±0.18	5.69±0.11	3.40±0.11	2.28±0.06	6.43±0.01	3.34±0.20
Carbohydrates	59.87±0.06	17.66±0.02	67.24±0.92	75.10±0.12	56.86±0.07	36.94±0.05
Fiber	12.54±2.12	53.57±3.91	10.22±1.83	3.60±0.05	12.48±4.77	27.31±0.75
Energetic value *	293.51±0.20	181.28±0.12	306.33±0.48	331.49±2.28	292.61±0.22	256.73±0.22
*aw***	0.29±0.001	0.33±0.003	0.32±0.002	0.33±0.004	0.57±0.001	0.37±0.002
Acidity***	1.78±0.01	1.60±0.12	1.98±0.01	1.53±0.03	8.7±0.03	1.89±0.04
pH	3.52±0.007	4.13±0.009	3.21±0.01	4.78±0.02	1.88±0.01	3.12±0.01
Color						
*L**	41.49±0.36	47.11±0.19	41.88±0.14	60.05±0.34	51.79±0.95	65.13±1.14
*a**	4.37±0.25	10.54±0.30	4.35±0.06	5.37±0.37	13.05±0.07	6.48±0.34
*b**	0.63±0.24	10.19±0.46	1.93±0.17	10.97±1.13	6.68±0.41	15.79±1.01
ºHue	12.09±2.68	44.04±0.62	23.33±1.61	64.11±0.88	27.10±1.65	67.68±1.36
Chroma	4.47±0.28	14.00±0.53	4.93±0.11	12.57±1.18	14.02±0.11	17.01±0.99
Minerals (m/100 g)						
Nitrogen	658±0.52	1663±0.63	1070±0.64	991±0.25	1327±0.41	2327±0.25
Phosphorus	98±0.01	217±0.06	109±0.09	150±0.12	260±0.14	431±0.14
Potassium	2080±1.61	480±0.52	960±0.24	920±0.42	1760±0.36	1080±2.87
Calcium	137±0.21	326±0.14	82±0.11	55±0.17	951±0.21	430±0.69
Magnesium	64±0.12	120±0.42	81±0.87	122±0.56	202±0.61	264±0.63
Sulfur	17±0.11	33±0.36	24±0.12	66±0.02	96±0.03	126±0.16
Boron	1.66±0.23	1.03±0.01	0.92±0.02	0.92±0.14	5.52±0.68	1.61±0.17
Copper	0.94±0.12	1.14±0.03	2.47±0.15	1.40±0.21	1.29±0.21	1.06±0.01
Iron	4.92±2.11	4.95±1.56	6.92±1.41	4.23±2.1	15.67±3.45	5.90±1.96
Manganese	nd.	0.38±0.12	3.09±0.03	3.18±0.09	1.78±0.08	3.10±0.02
Zinc	0.62±0.02	0.55±0.03	1.56±0.14	1.29±0.13	1.93±0.19	3.22±0.11
Antinutrients (mg/100 g)						
Oxalate	27.31±4.78	13.02±3.08	6.79±1.11	20.21±0.73	10.27±5.77	6.73±0.98
Cyanogen ****	Absence	Absence	Absence	Absence	Absence	Absence

Values expressed as mean±standard deviation (n=6); GPF=Grape peel flour; GSF=Grape seed flour; JPF=Jaboticaba peel flour; JSF=Jaboticaba seed flour; HCF=Hibiscus calyx flour; HFFS=Hibiscus fruit flour with seeds. Note: * = energetic value expressed in Kcal/100 g; ***aw*=water activity; *** Titratable acidity=value expressed in g of citric acid/100 g; **** = cyanogenic compounds expressed in absence and presence; nd=not detected

luminosity of 41.49 and 41.88, respectively, with parameters *a** (4.35 and 4.37, respectively) tending towards red tones and *b** (0.63 and 1.93) towards blue tones. These values follow the line of the original raw material, reflecting its natural characteristics. The hue angles *ºHue* (ºH) confirm the results, where the grape and jaboticaba peels flours tend towards bluish-red or red-violet, and the calyx flour tends towards yellowish-red. Grape seeds present red tones, and jaboticaba and hibiscus seeds present yellowish-red tones. The color intensity (*C**) varied from 4.47 to 17.01, indicating more neutral colors without brightness.

Eleven minerals were quantified in the flours of the raw materials analyzed and presented in Table 1. Generally, the commonly inedible portions (seeds) presented the highest mineral contents, which justifies the total processing of these raw materials. The most abundant macronutrients among the flours evaluated were nitrogen (658−2327 mg/100 g) and potassium (480−2080 mg/100 g). Flours from hibiscus co-products showed the best results, especially HFFS, with the highest content of nitrogen (2327 mg/100 g), P (431 mg/100 g), Mn (3.10 mg/100 g) and Zn (3.22 mg/100 g), while HCF showed the highest content of K (1760 mg/100 g), Ca (951 mg/100 g) and B (5.52 mg/100 g). According to Morais et al. [5], the high nitrogen content indicates high protein content, as found in the results presented in Table 1 for nutritional data.

Hibiscus flours are sources of calcium (Ca), where HCF with 951 mg/100 g, close to the daily recommendation of the macronutrient (1000 mg day^{-1}), and HFFS requires the consumption of 232 g to provide the necessary RDI. The main micronutrient among the flours was iron (Fe), emphasizing HCF. Although the bioavailability of iron in vegetables is generally lower than in animal origin, the iron content found in the flours (4.23 to 15.67 mg/100 g) was higher than that found in flours of vegetables consumed daily, such as beetroot, carrot, and spinach (2.10, 1.28 and 12.60 mg/100 g, respectively) [40]. The ingestion of 100 g of hibiscus calyx flour provides the recommended daily iron intake. Iron is essential for oxygen transport, immune system functions, and combating anemia. Considering that one-third of the world's population suffers from anemia, grape, jaboticaba, and hibiscus flours may be valuable in the diet to increase iron intake and help combat this condition [41].

The results suggest that the consumption of hibiscus calyx flour (HCF) and hibiscus fruit and seed flour (HFFS) may be necessary for building bones, preventing blood clots, and also for nerve and muscle contraction [42]. Absorption occurs mainly in the small intestine (90%) and, to a lesser extent, in the colon. There are two absorption processes: transcellular transport, regulated by vitamin D, and paracellular transport, which depends on calcium solubility and the time it remains in the intestine. For calcium to be bioavailable, it must be absorbed without precipitating in the intestines, facilitated by precipitation inhibitors and complexing ligands. The absence of precipitation inhibitors and the interaction of calcium with other components can reduce its bioavailability, impairing bone health [43].

Both jaboticaba peel and seed flours contain macronutrients essential to the diet, with particularly significant nitrogen levels, especially in JPF (1070 mg/100 g). The values exceeded those reported by Inara et al. [44] for the whole fruit, dry peel, and seed fractions. These flours can be valuable dietary additions to support general health and well-being, providing important nutrients that support crucial bodily functions. These macronutrients in adequate amounts reinforce the importance of including these flours in the daily diet to promote optimal health and prevent nutritional deficiencies [45]. Other components of the flour can influence the bioavailability of minerals for absorption. The interaction of certain nutrients can affect the immune system and cause allergic reactions. Identifying and understanding these interactions is essential for selecting vegetables that meet specific dietary criteria and improve nutrient absorption. Mineral ratios are often more important than individual levels, as they reflect nutritional interrelationships and can indicate pathological conditions, physiological factors, and dietary effects [24, 46].

The oxalate levels found in the flours were relatively low (6.73 and 27.31 mg/100 g) (Table 1) compared to those found by Gómez-Mejía et al. [47] in Albariño grape seeds (38.9 mg/100 g). Also, they compared with calyxes and hibiscus seeds grown in Egypt, with 33.3 and 115.2 mg/100 g, respectively [46]. The recommended oxalate intake is up to 60 mg/day^{-1}, especially for people with kidney stones. Therefore, the flours analyzed, with their low oxalate content, are suitable for safe consumption within the recommendations. They offer a nutritious option that can be incorporated into specific diets without significantly increasing the risk of kidney stone formation [48]. On the other hand, cyanogenic compounds are considered antinutritional since such compounds release hydrocyanic acid, which, through the action of enzymes, causes toxicity, which can lead to poisoning [49]. Therefore, their absence is recommended, as observed in all flours analyzed (Table 1).

Mineral proportions of the flour

The mineral proportions of the evaluated flours are shown in Table 2. Calcium-phosphorus (Ca/P) ratios of the six ranged from 0.28 to 2.83. The recommended Ca/P ratio should be >0.5 to ensure calcium absorption in the small intestine [50]. For Khatun et al. [51], the diet is considered good if the Ca/P ratio is more significant than 1.0 and poor if this ratio is below 0.5 for good intestinal utilization of Ca and P. In this study, the GPF, GSF, JPF, HCF, and HFFS flours obtained ratios above the minimum requirement (0.5). GPF, GSF, and HCF were above 1.0, indicating that these flours present a favorable absorption of calcium in the intestine for bone formation and maintenance, especially in children and the elderly [52]. The flour's calcium-potassium (Ca/K) ratios ranged from 0.06 to 0.53. An ideal Ca/K level is 4, so calcium absorption in the intestine and bone development

Table 2 Mineral proportions of grape, Jaboticaba, and hibiscus byproducts flours

Proportions	GPF	GSF	JPF	JSF	HCF	HFFS	Limits
Calcium / Phosphorus	1.08	1.16	0.58	0.28	2.83	0.77	>0.5
Calcium / Potassium	0.06	0.66	0.08	0.06	0.53	0.39	<4
Calcium / Magnesium	1.28	1.63	0.61	0.27	2.82	0.98	1
[Potassium / (Calcium+Magnesium)]	8.76	0.94	4.54	3.65	1.40	1.27	<2.2
Calcium / Iron	0.04	0.09	0.02	0.02	0.08	0.10	-
Iron / Zinc	9.19	10.44	5.16	3.81	9.41	2.13	>2
Oxalate / Calcium	0.009	0.004	0.015	0.023	0.001	0.003	1

GPF=Grape peel flour; GSF=Grape seed flour; JPF=Jaboticaba peel flour; JSF=Jaboticaba seed flour; HCF=Hibiscus calyx flour; HFFS=Hibiscus fruit flour with seeds are positive. The relationship between these minerals is called the thyroid ratio because calcium and potassium are vital in regulating thyroid activity [53]. Gemede [24] indicates that the low Ca/K ratio may be related to increased thyroid expression. Since the Ca/K ratios of flour are low, about the limit, the ingestion of other sources of calcium when consuming flour would be a strategy to improve the ratio.

Calcium and magnesium are essential for the body's functioning, as they are associated elements and are considered the "gold standard" for health issues related to minerals. Studies report that a high Ca/Mg ratio is associated with an increase in insulin levels [54], and a corresponding reduction is associated with calcium deficiency, leading to a decrease in the proportion available for the formation of bones and teeth [55]. The Ca/Mg ratios in the flours were between 0.27 and 2.82. GPF, GSF, and HCF flours were above 1.0, the minimum value required for calcium absorption [56]. Grape peels, seed flours, and hibiscus calyx flours presented higher amounts of calcium relative to magnesium and, therefore, did not interfere with magnesium absorption. Calcium supplementation would be necessary in diets that rely solely on JPF, JSF, and HFFS flours as food sources. A low Ca/Mg ratio may suggest low insulin levels, impacting blood sugar control. It may also indicate increased production of adrenal cortex hormones, such as cortisol, which is involved in the stress response and metabolism [54].

This Ca/Mg ratio has been reported to depend on dietary potassium levels due to the synergism between potassium and magnesium [54]. Therefore, the milliequivalent ratio [K/(Ca + Mg)] has been reported to be a better indicator of magnesium bioavailability in the presence of potassium than the calcium/magnesium ratio alone [52]. In this study, the [K/(Ca + Mg)] ratios of GSF, HCF, and HFFS flours were lower than the critical limit (2.2), indicating that the calcium and magnesium contents were higher than the potassium contents. Therefore, the consumption of 100 g of these flours does not present a risk of hypermagnesemia in humans [52]. Studies indicate calcium (Ca) can interfere with iron absorption (Fe). However, there is still no exact proportion of Ca/Fe intake to avoid interference in absorption, as it depends on several individual factors, such as age, sex, health status, and the general composition of the diet. To minimize interference with iron absorption, it is recommended to avoid consuming large amounts of calcium (more than 500 mg) along with iron-rich meals, and including vitamin C in the diet can help mitigate these effects [57, 58].

Pérès et al. [59] reported that iron did not impair zinc absorption up to a Fe ratio of 2:1 in normal rats. From this ratio, a dose-dependent effect was observed, where increasing the iron-to-zinc ratio led to a more significant inhibition of zinc absorption, up to a ratio of 5:1. However, when increasing the ratio to 10:1, there was no additional inhibition of zinc absorption. In iron-deficient rats, no changes in zinc absorption and retention were observed. The Fe/Zn ratio in the flours ranged from 2.13 to 10.44, indicating that the iron present possibly did not impair zinc absorption. Thus, consuming the analyzed flours may help correct iron and zinc deficiencies, impacting growth, cognitive development, and immune function. Keyata et al. [46] obtained Ca/P (9.5), Ca/K (8.9), and Fe/Zn (7.9) ratios in red hibiscus calyxes grown in Egypt. They found mineral ratios for hibiscus seeds, Ca/P (2.8), Ca/K (1.8), and Fe/Zn (1.7). No proportions of these nutrients were found in the literature for jaboticaba and grapes.

To evaluate whether calcium absorption is affected by oxalate content in the flours, the oxalate/Ca ratio was calculated (Table 2). An oxalate/calcium ratio greater than 1 could compromise calcium absorption [24]. Reduced calcium intake leads to more excellent intestinal oxalate absorption, possibly increasing the risk of stone formation. Calcium supplements may reduce oxalate absorption because calcium binds to dietary oxalate in the intestinal lumen [60]. Considering the calcium contents shown in Table 2 and the oxalic acid content in Table 1, the oxalate/Ca ratio was less than 1 in all flour samples, with mean values ranging from 0.001 to 0.021. Thus, oxalic acid calcium does not compromise calcium bioaccessibility.

Phytochemical compounds

The phytochemical composition of the flours is detailed in Table 3. The studied samples carotenoid content (Table 3) presented high concentrations, emphasizing GPF, HCF, and JPF (18.56, 15.19, and 11.33 mg/100 g, respectively). However, all the flours evaluated can be considered sources of carotenoids since foods containing more than 0.02 mg/g of carotenoids are considered rich sources of these compounds [61]. The carotenoid content in grape and jaboticaba berries is essential for the industry as precursor of important aromas [62].

Flavonoid content (Table 3) was highest in JSF (234.99 mg/100 g) and JPF (156.14 mg/100 g). As largest class of phenolic compounds in nature and are widely known for their health-promoting effects and functional properties, including anticancer, antioxidant, anti-inflammatory, and antiviral properties [63]. On the other hand, the flours made from jaboticaba and grape peels (JPF– 121.83 mg/100 g; GPF– 113.32 mg/100 g), as well as hibiscus calyx (HCF– 112.09 mg/100 g) presented the highest number of anthocyanins. The main anthocyanins in jaboticaba and grape peel are cyanidin 3-glucoside and delphinidin 3-glucoside [64]. In hibiscus calyx, they are delphinidin 3-*o*-sambubioside

Table 3 Phytochemical compounds present in grape, Jaboticaba, and hibiscus mineral proportions of grape, Jaboticaba, and hibiscus byproducts flours

Analysis (mg/100 g)	Samples					
	GPF	GSF	JPF	JSF	HCF	HFFS
Carotenoids	18.56 ± 1.95	6.97 ± 0.03	11.33 ± 0.54	4.27 ± 0.27	15.19 ± 0.05	2.0 ± 0.60
Flavonoids	139.15 ± 3.07	36.32 ± 1.16	156.14 ± 0.38	234.99 ± 0.61	115.86 ± 2.80	19.83 ± 1.21
Anthocyanins	113.32 ± 2.20	15.41 ± 1.16	121.83 ± 0.06	8.47 ± 0.08	112.09 ± 1.72	4.0 ± 0.03
Tannins	1361.61 ± 6.28	304.12 ± 3.56	1539.22 ± 2.12	684.21 ± 3.96	21.25 ± 0.32d	36.41 ± 0.76
Chlorophyll	37.16 ± 0.27	5.22 ± 0.35	40.95 ± 0.122	5.40 ± 0.48	32.85 ± 0.23	7.45 ± 0.11
Chlorophyll a	20.03 ± 0.16	4.90 ± 0.26	22.56 ± 0.64	2.22 ± 0.33	22.58 ± 0.14	5.18 ± 0.12
Chlorophyll b	6.11 ± 0.07	1.66 ± 0.11	14.22 ± 0.10	2.93 ± 0.06	6.32 ± 0.17	1.46 ± 0.13

Values expressed as mean ± standard deviation (*n* = 6); GPF = Grape peel flour; GSF = Grape seed flour; JPF = Jaboticaba peel flour; JSF = Jaboticaba seed flour; HCF = Hibiscus calyx flour; HFFS = Hibiscus fruit flour with seeds

and cyanidin 3-*o*-sambubioside (responsible for the bright red color) [65].

The tannin content was concentrated in the GPF and JPF samples, 1361.61 and 1539.22 mg/100 g, respectively. In this sense, higher concentrations of tannins are found in grapes with thicker peels, such as in this study. The tannins in grape peels are more astringent, while those in the seeds and stalks tend to be more bitter. The tannin contents of HCF and HFFS were lower than those found by Keyata et al. [46] in calyxes and seeds of red hibiscus sabdariffa grown in Ethiopia. The antinutritional action of tannins is reported in concentrations above 5% of the consumed portion, hurting palatability (astringency) and reducing the digestibility of proteins, vitamins, and minerals [66]. In this study, all percentages were below 5% in 100 g of sample. On the other hand, these antinutrients are thermolabile, sensitive to heating, and can be eliminated after cooking [29].

Chlorophyll, the pigment responsible for the green color of plants and vegetables, is present in greater quantities in fruits when immature [67]. As these fruits ripen, chlorophyll is degraded, decreasing the green color and the appearance of other colors, such as purple in jaboticabas and shades of purple and red in grapes [68]. The shell flours presented the highest total chlorophyll contents, JPF (40.95 mg/100 g), followed by GPF (37.16 mg/100 g) and HCF (32.85 mg/100 g); the other flours presented low results for this compound. The differences in chlorophyll levels between seeds and shells are attributed to distinct biological functions. While leaves and shells are mainly responsible for photosynthesis and contain high levels of chlorophyll to capture solar energy, seeds are nutrient storage structures and protect the developing embryo [69]. Therefore, seeds do not need large amounts of chlorophyll to perform their specific biological functions, resulting in lower contents of this pigment than shells and leaves. JPF presented the highest total chlorophyll content among the shell flours (40.95 mg/100 g), which may indicate that they are in a younger stage of development. Since chlorophyll degrades during maturation, anthocyanin biosynthesis occurs [70].

Analysis of the profile of individual compounds

The identification and quantification of carbohydrates were performed by high-performance liquid chromatography (HPLC-DAD/UV-Vis) and are presented in Table 4. In general, the grape and jaboticaba peel flours had the highest sugar concentrations, as expected, based on their carbohydrate content found in Table 1. Fructose and glucose were the main carbohydrates in GPF (21.82 g and 16.27 g/100 g, respectively), as already described by Walker et al. [71], and sucrose and fructose were the main carbohydrates of JPF (23.52 and 16.02 g/100 g, respectively). Sucrose and its constituents, glucose and fructose, are responsible for most of the non-structural sugar content of many ripe fruits [72]. These carbohydrates provide a metabolic substrate, acting as carbon storage, osmoregulation, transport, signaling, and, in their wild ancestors, attracting animals to disperse the fruits/seeds [73]. Fructose, obtained through the hydrolysis of inulin or sucrose (fructose + glucose), is the sweetest-tasting sugar, followed by sucrose.

The sugars concentration in GPF and JPFoffer advantages and disadvantages, depending on the context of consumption and dietary goals. Sugars such as glucose, fructose, and sucrose are quick energy sources that benefit athletes or individuals needing a quick energy boost [74]. In addition, these flours can be used as natural sweeteners in recipes, reducing the need for refined sugar [75]. However, on the other hand, high concentrations of sugars can raise the glycemic index of foods, which can be problematic for individuals with diabetes or those trying to control blood sugar levels [75]. Furthermore, for those monitoring their caloric intake, sugars can contribute to an unwanted increase in calories in the diet [74].

The World Health Organization suggests that a maximum of 50 g of sugar be consumed daily, corresponding to 10% of an adult's diet, considering a 2,000-calorie diet [5]. Therefore, GPF and JPF flours should be moderate and integrated into a balanced diet since they contain 42.78 and 50.08 g/100 g of total sugars. Although they have nutritional

Table 4 Profile of carbohydrates, organic acids, and individual phenolics of flours prepared from grape, Jaboticaba, and hibiscus processing byproducts

Analysis	GPF	GSF	JPF	JSF	HCF	HFFS
Carbohydrates (g/100 g)						
Glucose	16.27 ± 0.11	3.31 ± 0.01	10.32 ± 0.87	3.26 ± 0.28	2.70 ± 0.56	1.10 ± 0.08
Fructose	21.82 ± 0.78	3.84 ± 0.06	16.02 ± 0.02	4.35 ± 0.17	2.38 ± 0.88	1.00 ± 0.11
Sucrose	4.69 ± 0.02	1.89 ± 0.08	23.52 ± 1.13	3.96 ± 0.10	5.45 ± 0.05	0.97 ± 0.99
Cellobiose	0.86 ± 0.28	nd.	0.22 ± 0.98	nd.	18.38 ± 0.10	4.04 ± 1.03
Total sugars	42.78 ± 0.57	9.04 ± 0.04	50.08 ± 0.86	11.57 ± 0.23	28.91 ± 0.49	7.11 ± 0.84
Organic acids (mg/100 g)						
Citric Acid	nd.	nd.	nd.	nd.	nd.	275.98 ± 0.33
Malic Acid	nd.	nd.	nd.	nd.	nd.	nd.
Tartaric Acid	988.77 ± 1.63	1452.42 ± 1.74	4381.54 ± 3.54	7000.58 ± 1.95	1042.78 ± 2.30	606.72 ± 0.12
Ascorbic Acid	10.98 ± 0.40	7.61 ± 0.71	1.15 ± 0.03	0.56 ± 0.01	48.48 ± 0.95	101.38 ± 1.42
Phenolic profile (mg/100 g)						
Caffeic	2.01 ± 0.11	0.98 ± 0.02	6.63 ± 0.40	21.94 ± 0.33	nd.	0.21 ± 0.04
Chlorogenic	10.18 ± 0.96	2.71 ± 0.06	nd.	nd.	nd.	31.83 ± 0.85
Ellagic acid	31.53 ± 0.33	nd.	41.40 ± 0.95	nd.	nd.	25.02 ± 0.54
Gallic acid	51.08 ± 0.47	20.89 ± 0.51	49.86 ± 2.88	54.57 ± 2.91	50.28 ± 0.43	nd.
Catechin	149.30 ± 1.52	256.36 ± 0.23	607.38 ± 3.01	nd.	nd.	nd.
Kaempferol	nd.	nd.	nd.	nd.	nd.	nd.
Myricetin	nd.	0.05 ± 0.01	nd.	6.81	233.17 ± 0.57	nd.
Quercetin	295.01 ± 0.99	104.32 ± 2.58	238.14 ± 0.33	540.48 ± 1.66	nd.	nd.
Naringin	nd.	nd.	nd.	nd.	27.54 ± 0.11	nd.
Rutin	2.01 ± 0.05	0.98 ± 0.01	6.63 ± 0.18	21.94 ± 2.17	nd.	0.21 ± 0.06

Values expressed as mean ± standard deviation ($n = 2$); GPF = Grape peel flour; GSF = Grape seed flour; JPF = Jaboticaba peel flour; JSF = Jaboticaba seed flour; HCF = Hibiscus calyx flour; HFFS = Hibiscus fruit flour with seeds

value and functionality, it is important to be cautious with excessive consumption, especially for individuals with specific dietary restrictions. Seed flours (GSF, JSF, and HFFS) presented low sugar concentrations, especially HSSF. HCF was low in glucose and sucrose but had a higher sucrose content (5.45 g/100 g) than GPF, GSF, JSF, and HFFS (4.69, 1.89, 3.96, and 0.97 g/100 g, respectively). The accumulation, metabolism, and transport of sugars, mainly sucrose, strongly depend on environmental and physical parameters, such as high CO_2, cold, and drought [76]. Water scarcity during the hibiscus cultivation period may have triggered an increase in sucrose content in HCF, which was not cultivated on a large scale like grapes and jaboticabas. Cellobiose is a reducing sugar and a significant component of the plant cell wall, found in plants as a consequence of cellulose hydrolysis; it has different enzymatic activities in the small intestine and presents the same chemical properties as maltose [77]. In this study, HCF flour demonstrated the highest content (18.38 g/100 g), which was not detected in GSF and JSF flours.

Regarding organic acids, as seen in Table 4, citric, malic, tartaric, and ascorbic acids were identified. However, only tartaric and ascorbic acids were quantified in all flours. Citric acid was detected only in HSSF. Tartaric acid (Table 4) is naturally found in several fruits, mainly grapes, bananas, and tamarind. It is a crucial acidity controller and directly influences pH and sensory characteristics [78]. This acid was considered the majority in this study, especially in JSF and JPF, with 7000.58 and 4381.54 mg/100 g, respectively. The results obtained for ascorbic acid, better known as vitamin C (Table 4), were high in HCF (48.38 mg/100 g) and HSSF (101.38 mg/100 g) flours. Therefore, hibiscus calyx and fruit flours with seeds have high amounts of this compound since the daily recommendation for vitamin C is 45 mg to 75 mg for women and 45 mg to 90 mg for adult men [79].

Phenolic compounds have gained significant interest due to their functional properties, such as antioxidant, anti-inflammatory, anticarcinogenic, and antimicrobial effects [80]. The main phenolic compounds present in the flours were catechin and gallic acid (Table 4). High concentrations of catechin were found in the peel flours (GPF and JPF), 149.30 and 607.38 mg/100 g. While for GSF, the catechin values were only 256.36 mg/100 g (Table 4). Inada et al. [44] evaluated the phenolic composition of jaboticaba seeds and peel and found that gallic acid and ellagic acid were the main non-anthocyanin compounds. In this study, ellagic acid was found to be 41.40 mg/100 g in JPF and not in the JSF sample. The presence of ellagic acid in higher concentrations in jaboticaba peel flour (JPF) is positive due to its health benefits. This polyphenol has antioxidant, anti-inflammatory, and potential anticancer properties, helping to combat free radicals and protect cells against oxidative damage [81]. In addition, it can contribute to cardiovascular health by reducing the oxidation of LDL cholesterol [82].

Abe et al. [83] determined that jaboticaba has the highest levels of ellagitannins, while they did not find ellagic acid in any of the grape cultivars analyzed. Ellagic acid derivatives attract scientific interest due to their antioxidant, antiproliferative, cardioprotective, and angiotensin-converting enzyme inhibition activities [81, 82]. As the fruits ripen, anthocyanin levels increase, and tannin and total ellagic acid contents decrease. At the ripe stage, the contribution to the total ellagic content follows the ratio of 1:3:1 (pulp: peel: seed), with a similar behavior for tannins [83]. Although ellagic acid was not detected in the seeds, it may contain other beneficial compounds. Thus, the presence of ellagic acid in JPF is advantageous, and the combined use of different parts of the plant can maximize the nutritional benefits. On the other hand, caffeic acid is a phenolic acid with high antioxidant and anti-inflammatory activity, typically found in coffee beans, wine, and apples [84]. JSF presented a higher compound content than JPF and other flours. The variety, metabolism, composition, density, and size of the berries, as well as the position of the bunches and environmental conditions, can reflect the production of polyphenol compounds [85]. Chlorogenic acid was detected in HSSF, GPF, and GSF. This compound has beneficial biological actions for glucose regulation and the development of type 2 diabetes, inhibiting Na+-related glucose absorption and insulin secretion [86]. It also reduces inflammatory and oxidative stress [87].

Among the flavonoids, the compound kaempferol was not identified in any sample, and naringin was determined only in HCF (27.54 mg/100 g). This compound is commonly found in citrus fruits, known for its antioxidant and anti-inflammatory effects and potential to improve cardiovascular health [88]. Although it is present in smaller quantities and only in HCF, its presence is beneficial. Quercetin was the main compound in grape and jaboticaba flours, especially in GPF (295.01 mg/100 g) and JSF (540.48 mg/100 g). This powerful antioxidant can help reduce inflammation, improve immune function, and protect against heart disease and cancer, making its presence extremely advantageous. Rutin and myricetin were also identified in the flours, emphasizing HCF, which presented 233.17 mg/100 g. Both compounds have antioxidant and anti-inflammatory properties, contributing to cellular protection and the prevention of chronic diseases [89]. Therefore, these flavonoids' presence and high concentration in grape and jaboticaba flours benefit health.

FTIR measurement

FTIR was used to identify the functional groups of the chemical compositions of GPF, GSF, JPF, JSF, HCF, and HFFS flours. The bands at 3600 to 3200 cm^{-1} corresponding to the O–H stretching can be attributed to moisture [90], as they are more discreet in GSF, JPF, and HCF (Fig. 1). The absorption close to 2900 cm^{-1} represents the C–H bonds, common in carbohydrate and lipid chains. It is observed that they are more intense in GPF and HCF. The bands in the region 1760–1650 cm^{-1} are characteristic of esterified

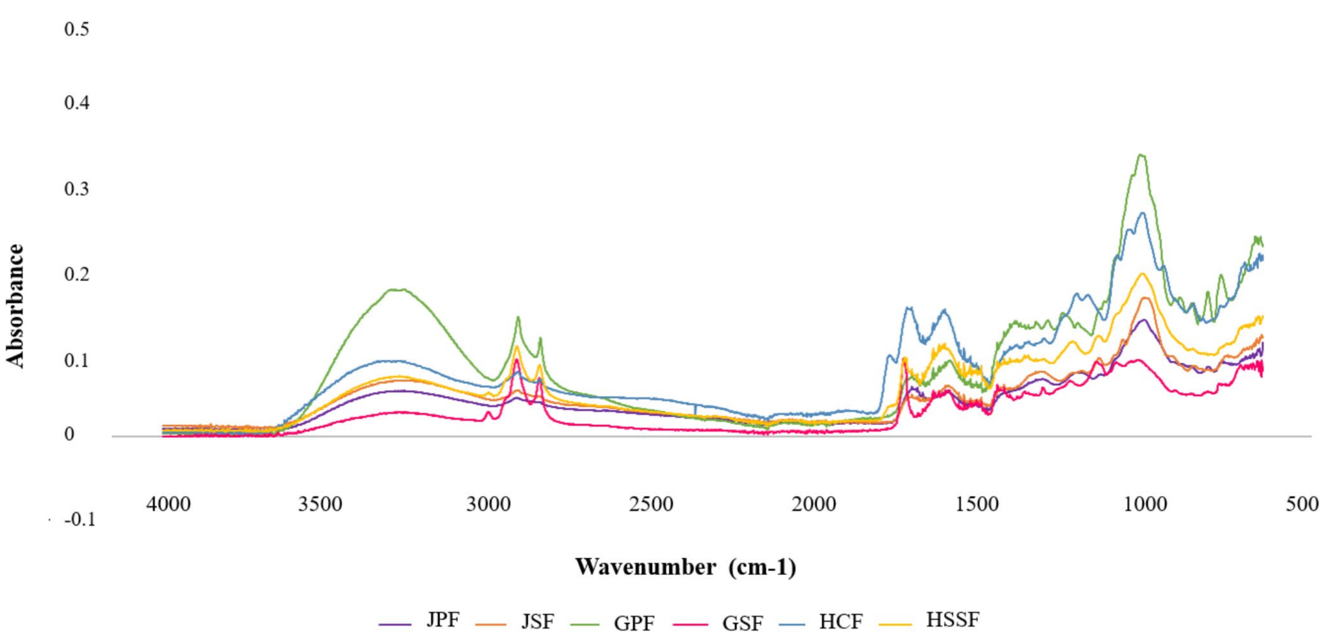


Fig. 1 Fourier transform infrared spectroscopy (FTIR-ATR) of the byproducts flours GPF - grape peel flour, and GSF - grape seed flour; JPF - jaboticaba peel flour, and JSF - jaboticaba seed flour; HCF - hibiscus calyx flour, and HFFS - HFFS = Hibiscus fruit flour with seeds

Table 5 Volatile compounds identified and quantified in flours prepared from byproducts of grape, Jaboticaba, and hibiscus processing

Volatile compounds	Odor description	Identification [a]	RT	Relative content (%)					
				GPF	GSF	JPF	JSF	HCF	HFFS
2-methylfuran	Ether-like and chocolate	MS + LRI	2.80	–	–	–	1.66	–	–
Ethyl acetate	Pineapple	MS + LRI	2.90	–	0.43	–	1.21	–	–
Acetaldehyde	Pungent, fruity	MS + LRI	3.12	12.74	6.24	12.82	1.02	56.60	3.80
Glycolaldehyde dimer	Waxy, citrus, and fatty	MS + LRI	3.15	–	10.08	14.31	3.36	–	–
3-Methylbutanal	Fruity, toasted and malty	MS + LRI	3.25	–	–	–	–	–	19.06
Ethanol	Alcoholic and sweet	MS + LRI	3.54	38.68	74.54	31.18	77.80	–	13.25
Pentanal	Cooked beef flavor	MS + LRI	4.15	–	–	–	–	–	0.73
2.3-Butanedione	Strong chlorine-like	MS + LRI	4.16	4.28	–	–	–	–	–
2-methyl-1-propanol	Ethereal	MS + LRI	5.28	–	–	–	1.81	–	–
Butyl ester	Fruity, vinegar, and sour	MS + LRI	5.83	0.86	0.64	–	–	–	–
Hexanal	Herbal and fruity	MS + LRI	6.04	1.76	0.26	–	0.48	0.59	4.48
6.6-dimethyl-2-methylene	Herbal	MS + LRI	6.27	0.76	–	–	–	–	–
5-Decino	nd.	MS + LRI	7.84	–	–	–	–	–	2.89
2-Butenoic acid	Pungent	MS + LRI	8.02	1.06	–	–	–	–	–
1-Penten-3-ol	Pungent and fruity	MS + LRI	8.07	–	–	–	–	0.15	–
Heptanal	Herbal	MS + LRI	8.47	–	–	–	–	0.22	–
d-limonene	Citrus	MS + LRI	8.68	13.60	4.91	–	–	–	0.89
Eucalyptol	Fresh, camphor-like odor	MS + LRI	8.87	–	–	0.28	0.59	–	–
1-Decino	nd.	MS + LRI	9.50	–	–	–	–	–	35.61
2-Pentylfuran	Fruity and green bean	MS + LRI	9.55	–	–	–	–	0.12	–
trans-β-ocimene	Herbal and woody	MS + LRI	9.69	–	–	0.24	–	–	–
γ-terpyrene	Sweat	MS + LRI	9.94	3.03	0.72	–	–	–	–
1-Pentanol	Fermented	MS + LRI	10.39	–	–	–	–	0.15	0.24
o-Cymene	Citric, lemon	MS + LRI	10.65	1.35	–	0.22	–	–	–
Hexyl acetate	Fruity and herbal	MS + LRI	10.85	4.03	0.64	–	–	–	–
Octanal	Aldehydic	MS + LRI	11.31	0.85	–	–	–	0.23	–
6-Methyl-5-hepten-2-one	Mushroom, earthy, woody	MS + LRI	12.62	0.66	–	–	–	–	0.81
Nonanal	Fatty, citrus, and green	MS + LRI	14.09	2.50	–	–	–	–	–
Hexanoic acid	Fatty, cheesy, waxy odor	MS + LRI	14.56	0.98	–	–	–	–	0.17
Copaene	Wood and spice	MS + LRI	15.52	–	–	2.13	–	–	–
Acetic acid	Fruity, vinegar, and sour	MS + LRI	15.80	1.20	1.54	11.05	7.11	36.28	13.81
Furaldehyde	Almond-like odor	MS + LRI	16.04	7.20	–	3.14	0.23	5.81	3.24
2.3-butanediol	Odorless	MS + LRI	17.89	2.83	–	2.10	–	–	–
Linalool	Floral, lavender, lemon	MS + LRI	18.02	–	–	0.68	0.30	–	–
Isoledene	nd.	MS + LRI	18.43	–	–	1.56	–	–	–
Caryophyllene	Herb, Sweet, and spice	MS + LRI	18.95	–	–	2.98	2.53	–	–
Propylene glycol	Slightly estery	MS + LRI	19.16	0.45	–	–	–	–	–
1.2.4-Methene-1 H-indene	Orange and rose	MS + LRI	19.22	–	–	0.75	–	–	–
Alloaromadendrene	Wood and honey	MS + LRI	19.82	–	–	0.55	–	–	–
Benzene acetaldehyde	Grassy	MS + LRI	20.35	–	–	0.38	–	–	0.23
Bicycloskiphelandrene	Floral, citrus and woody	MS + LRI	20.76	–	–	1.11	–	–	–
α-terpineol	Pepper, woody and earth	MS + LRI	21.56	–	–	0.63	0.46	–	–
Naphthalene	Mothball or benzene-like	MS + LRI	21.84	–	–	1.49	1.47	–	–
Cyclohexane methanol	Mild camphor odor	MS + LRI	22.74	–	–	2.19	–	–	–
2.4-decanoic acid	Bartlett Pear	MS + LRI	24.77	0.56	–	–	–	–	–
Nonanoic acid	Aldehydic and spicy	MS + LRI	30.09	–	–	–	–	–	0.81
Agarospirrol	nd.	MS + LRI	30.14	–	–	1.37	–	–	–

[a] MS + LRI, mass spectrum, and LRI in agreement with the literature; MS, mass spectrum in agreement with spectrum in Wiley275 and NBS75 K libraries. The Odor descriptions were based on data reported in the literature [93–97]

and non-esterified carboxyl groups in pectin, mainly in HCF. Bands around 1700 − 1600 cm^{-1} indicate the presence of C = C double bonds, typical in aromatic compounds and some organic acids. In this range, all flours showed peaks, especially HCF flour, which showed peaks with higher intensities. The region between 1200 and 900 cm^{-1} is called the 'fingerprint' for carbohydrates, especially regarding sugar composition [91]. GSF showed the lowest carbohydrate content, as shown in the spectrogram (Fig. 1) and nutritional composition (Table 1). Peaks observed between 1000 and 1500 cm^{-1} are characteristic of phenolic compounds. Pure catechin is observed at 1018 cm^{-1} and 1112 cm^{-1}, attributed to C–O and C–C stretching, and 1143 cm^{-1} (phenyl C–OH) [92].

Volatile compounds

The results of the flours' characterization and identification of volatile compounds are presented (Table 5). In total, 119 different volatile compounds were identified in the flours. JPF stood out with 29 other compounds detected, followed by GPF with 22 types of compounds, HSSF with 16 compounds, JSF with 15 compounds, GSF with 10 compounds, and HCF with 8 different compounds. These compounds mainly belong to the class of esters (27 compounds), terpenes (25 compounds), ketones (17 compounds), carboxylic acids (10 compounds), aldehydes (10 compounds), alcohols (9 compounds), alkanes (9 compounds), lactones (4 compounds), furans (2 compounds), phenols (2 compounds) and 4 other compounds (coumarin, octadecan-2-one, heptadecan-2-one, nonadecan-2-one). Esters and terpenes are the predominant classes, followed by ketones and carboxylic acids. On the other hand, alcohols have the highest peak area ratio about the total peak area of all flours, representing 45.80% of the total area, followed by aldehydes with 19.3% of the total area and esters with 12.7%.

When analyzing the compounds in JPF, the main compound is ethanol (31.18%). Certain vegetables naturally contain ethanol in their matrices due to metabolic processes or the degradation of other organic compounds. The raw materials analyzed may have high levels of precursors that are transformed into ethanol. The headspace extraction method can volatilize and concentrate compounds such as ethanol, increasing their detection. During heating at 80 °C for 30 min, volatile compounds are released and captured more efficiently, which may explain the high content identified. The second major compound identified was Glycolaldehyde dimer (14.31%). Glycolaldehyde dimer is a chemical compound resulting from the union of two glycolaldehyde molecules, a simple aldehyde with the formula $HOCH_2CHO$. This dimer, known as 1,2-ethylene glycolaldehyde, has the formula $HOCH_2CH(OH)CHO$. It is formed through a condensation reaction in aqueous glycolaldehyde solutions, where the molecules interact and combine. The dimer is relevant in organic chemistry studies and may have applications in synthesizing other chemical compounds or understanding reactions involving aldehydes.

Conclusion

In conclusion, the flours analyzed presented a wide nutritional and functional diversity, emphasizing the high concentration of proteins, fibers, minerals, and bioactive compounds, such as flavonoids and carotenoids. Hibiscus, grape, and jaboticaba flours proved promising sources of essential nutrients, such as calcium and iron, and are viable alternatives to enrich diets and combat nutritional deficiencies. In addition, the low water activity of the flour contributes to more excellent stability and longer shelf life. The presence of phenolic compounds and antioxidants reinforces their potential to promote health benefits, such as anti-inflammatory and antioxidant effects. The flours also demonstrated relevant technological properties that can be explored in the food industry, such as replacing sugars and improving product preservation. Therefore, the flours studied are versatile foods with great potential for application in various food formulations and specialized diets. The data indicate that a large proportion of raw materials (waste) are not used directly for consumption, highlighting the importance of strategies for using waste. Transforming this waste into by-products can reduce waste and generate new sources of income, contributing to environmental sustainability and innovation in the food, cosmetics, and pharmaceutical industries.

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Declarations

Competing interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this study.

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Authors and Affiliations

Maria Olivia dos Santos Oliveira[1,2] · Gabriel Luis Castiglioni[1] · Julião Pereira[1] · Mariana Alencar da Macena[2] · Rômulo Alves Morais[2,3] · Glêndara Aparecida de Souza Martins[2,3] · Eduardo Ramirez Asquieri[1]

✉ Maria Olivia dos Santos Oliveira
mariaoliviaeng@gmail.com

[1] Graduate Program in Food Science and Technology, Department of Food Science and Technology, Federal University of Goiás (UFG), Goiânia 74690-900, Brazil

[2] Kinetics and Process Modeling Laboratory, Department of Food Science and Technology, Federal University of Tocantins (UFT), Palmas 77001-090, Brazil

[3] Graduate Program in Food Science and Technology, Department of Food Science, Federal University of Tocantins (UFT), Palmas 7701-090, Brazil