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Editorial Board

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Editorial

Fruit and Vegetable Derived Waste as a Sustainable Alternative Source of Nutraceutical Compounds

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Dietary phytochemicals are widely investigated in the field of chemistry, biology, nutrition, and medicine for their potential health-promoting effects. Indeed, many *in vitro* and *in vivo* studies provide evidence that a number of these compounds are involved in the prevention and/or control of chronic disorders such as cancer and cardiovascular diseases. The exponential growth of plant waste production from the agrofood industry is a critical global issue, considering its storage, disposal, environmental impact, and potential health risks. However, the exploitation of plant wastes/by-products for the recovery of added-value compounds offers new avenue for industrial growth and waste management. Indeed, the research and development of new functional foods and health products from low-cost raw materials is of great importance in nutraceutical, cosmetic, pharmaceutical, and agribusiness sectors. Besides, optimizing the processing methods of waste products in order to reduce biomass utilization and environmental risks, as well as to improve recovery of added-value compounds, represents an urgent and necessary technological innovation for the benefit of mankind. In an industrial point of view, moreover, the utilization of food waste for recovering nutraceuticals is economical not only in production line but also in their disposal.

The purpose of this special issue is to feature the scientific knowledge on the nutraceuticals associated with plant waste products derived from fruits and vegetables and their *in vivo* and *in vitro* bioactivities. The information disseminated through this issue is hoped to serve as an interdisciplinary link between biochemistry of nutrition, functional foods, and food technologies. Knowledge of not only quantity and quality of nutrients and nonnutrients present in such functional foods but also their bioactivity may provide broader and valuable information on the food quality field of research.

This special issue about "Fruit and vegetable derived waste as a sustainable alternative source of nutraceutical compounds" covers research articles from different perspectives. Overall, most of the papers have been related to (i) extraction and characterization of bioactive compounds from plant by-products as sources of health-related beneficial compounds; (ii) process optimization; (iii) development of new products and functional foods; and (iv) *in vitro* and *in vivo* bioactivities of nutraceutical components present or extracted from plant food wastes.

Among the submitted manuscripts, five papers have been selected to be part of this special issue. The paper authored by V. Lele et al. deals with the development of chewing candy (CC)—nutraceutical formulations from juices and by-products of juices of the fruits sea buckthorn (*Hippophae rhamnoides* L.) and quince (*Cydonia oblonga* L.) with antimicrobial properties against a panel of pathogenic bacteria strains. Two texture-forming agents (agar and gelatin) were tested for CC formulation. The results obtained in this study indicated that all samples (juices and juice by-products) displayed antimicrobial activity against all the

pathogens tested, and the largest inhibition zones against Bacillus and *Proteus mirabilis* were observed for sea buckthorn juice and quince juice, respectively. Moreover, the addition of all samples (sea buckthorn and quince juices and juice by-products) increased the antioxidant activity and total phenolic content of CC. Therefore, taken together all results, not just juice but also juice by-products, have great potential as desirable antimicrobial ingredients for the food industry with the best acceptability values found for CC prepared with agar and sea buckthorn juice by-products and with gelatin and quince juice.

Abundant residues are generated by industrial processing of blackberry in juices and concentrates. The study by Zafra-Rojas et al. analyses chemicals, minerals, organic acids, antioxidants, and dietary fiber of Mexican blackberry (*Rubus fruticosus* cv Tupy) residues and compares it with a prune-based commercial product. The results show that these residues possess bioactive components and functional properties higher than the commercial sample. Indeed, they are a very rich source of malic acid, phenols, and anthocyanins that contribute to a remarkable antioxidant capacity as measured by the ABTS assay. In addition, the residues can reduce iron and contain high amount of dietary fiber with elevated water retention and swelling capacity. Due to these characteristics, this waste matter could be considered as a potential source of useful and healthy components.

The Bigarade is a bitter orange (*Citrus aurantium* L. cv Bigarade) whose unpleasant taste mainly restricts its utilization to industrial extraction of essential oils. The study carried out by Lagha-Benamrouche et al. was aimed at debittering the peel of these fruits to obtain a jam preparation with appreciable sensorial quality. At the same time, a number of analyses have been carried out to check physicochemical characteristics, bioactive components, and reducing power of the jam in comparison with the original bitter fruit. The results show that the debittering process, including treatment with salt (NaCl), heat, and water decreases acidity, sugars, proteins, bioactive compounds, and reducing power, whereas increases the ash rate. Nevertheless, this jam still remains an interesting source of bioactive compounds with antioxidant potential, to be considered for dietary purposes. This may add new interest to the exploitation of this fruit cultivated in Algeria.

Olive tree culture and oil production are of economic significance in Jordan. The paper authored by Al-Widyan et al. faces the interesting problem of treatment and exploitation of olive oil industry by-products and wastes, in particular the solid waste, a lingocellulosic organic material called olive cake. Considering that the processing at the olive mills, usually performed during the cold season, needs large amounts of hot water and then expensive diesel fuel, the authors propose building a system combining a ground well component (receiving water from tankers that bring the water from nearby springs) and a heat recovery component exploiting the aerobic biological fermentation of the olive waste. A number of analyses are performed to assure that the olive cake can be used for extended periods as a source of fermentation. The authors provide evidence that their system can significantly produce raises in the water temperature before entering the fueled-operated boiler, to satisfy much of the mill needs. Souza et al. have reported that custard apple (*Annona squamosa* L.) bagasse flour, a by-product from custard apple processing, could serve as a promising ingredient in cookies enabling good sensory acceptability. The authors have also shown that the flour and the cookies formulations are rich in essential minerals (Cu, Fe, Mn, Zn, Ca, and Mg) and polyphenols (200 to 658 mg GAE/100 g). Overall, the research highlights that the custard apple bagasse pulp flour can be incorporated in food formulations to improve nutritional and functional properties.

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Research Article

Desamerization of Bitter Jam: Biochemical and Sensory Quality

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This work consists of studying the influence of the desamerization of the mesocarpe on the chemical composition and the sensorial quality of the jam, based on the bitter orange. The results of the various analysis show that desamerization decreases acidity, sugars, protein, and bioactive compound levels (carotenoids, polyphenols, and vitamin C), but desamerized jams still remain an important source of antioxidant compounds with antioxidant potential in the diet. Concerning the sensory analysis of the jams, the results show that the jam desamerized with water presents the same bitterness as the bitter jam and that the salt significantly reduces the bitterness of the jams.

1. Introduction

Citrus fruits are one of the most important fruit crops in the world. They include lemons, mandarins, pomelos, cédrats, oranges, grapefruits, limes, etc. They are eaten as dessert (fresh fruit), jam or juice [1].

Citrus peel is characterized by its high content of dietary fiber (pectin, cellulose, and polysaccharides), minerals, vitamins and secondary metabolites with significant antioxidant potential and contributing to human health such as carotenoids, essential, oils and phenolic compounds [2, 3]. Citrus peel is a natural source of flavonoids, in particular flavanones, flavanone glycosides, and polymethoxylated flavones, which are relatively rare in other plants [4] and are known to have anti-inflammatory and antioxidant properties [5]. The Bigarade is rarely consumed fresh because it is very acidic. The presence of certain flavonoids (neohesperidine, naringine, etc.) in the peels of the Bigarade gives the fruit a bitter taste, which is felt in the jams' preparations [6].

Jam is a means of preserving fruits, and the high sugar content of jam does not allow bacteria, yeast, and molds to grow and also prevents other spoilage. This means that the nutritional qualities of the fruits can be maintained at the same time as providing tasty products [7].

The objective of our work is to study the influence of the desamerization of mesocarpe on the chemical composition and the sensorial quality of the Bigarade jam. Our work is in line with the prospect of valorisation of the bitter orange that is exploited in Algeria only for these essential oils.

2. Materials and Methods

2.1. Collection of Samples. Bitter oranges (*Citrus aurantium* L. cv Bigarade) were collected in the area called Atlas Blideen or Metidjian Atlas which is a region known for the fertility of its soil and its oranges of good quality. This region is located between Blida and Bouïra, two nonremote regions located in the north center of the country. Both regions have a Mediterranean climate with a relatively cold and rainy winter and a hot and dry summer. The harvest was done in a random manner from several trees belonging to the same variety during the month of February 2016.

2.2. Desamerization of Peels and Preparation of Jams

2.2.1. Desamerization. Salt, heat, and water are considered as driving elements in the operation of desamerization process. In this study, the salt factor was variable; five salt levels were fixed during this operation. They correspond to the five percentages (0, 0.3125, 0.625, 1.25, and 2.5%) of salt taken according to the weight of the fruit. The peels of the fruit were cut into pieces and immersed in salt water for 5 hr and then preheated with salt in stainless steel pot for five minutes after boiling. The hot water was poured and then replaced by cold water with the addition of salt for soaking (4 times at 5 hr intervals).

2.2.2. Cooking. The drained peel and the cleaned fruit quarters are cut into small pieces of 2×3 cm, and the seeds are put into a small muslin bag. Weigh the fruits and take the same weight of water and 1.5 weight of sugar to prepare the syrup. The jam is cooked for 45 to 50 min. During the boiling, temperature and sugar concentration were checked. This treatment is stopped when the concentration reaches 60–65 Brix by means of a refractometer.

2.2.3. Physicochemical Analysis

(1) Moisture Content. The thermal drying method was used in the determination of moisture content of the samples [8].

(2) pH and Titratable Acidity. The pH of the jams and pulps is measured using a pH meter. The titratable acidity is determined by neutralizing the acid present in a known amount of sample using a base (NaOH). The evaluation is carried out by titration using a color indicator, phenolphthalein [9].

(3) Rate of Soluble Solids. Soluble solids represent all solids dissolved in water, including sugars, salts, proteins, and carboxylic acids. The rate of soluble solids, expressed in degrees Brix, is determined using a refractometer [8].

(4) Sample Defecation. The aqueous extract of sugary solutions is loaded with many substances (carbohydrates, fats and lipoids, pigments, amino acids, organic acids, mineral salts, reducing substances, which are not carbohydrates, etc.). These substances may disrupt the quantification of sugar. Defecation of samples was carried out according to the Carrez method [10].

(5) Total Sugar Content. The total sugar content was determined according to the method of Dubois [11]. Sugar concentrations are determined by referring to the standard glucose curve (10 to 80 μg/ml), and the results are expressed in mg glucose equivalents/100 mg or 100 ml of jam, fresh peels, or Bigarade juice.

(6) Reducing and Nonreducing Sugar Content. This method is based on the reduction of the Fehling liquor in the presence of soda by the reducing sugars present in the sample. For the nonreducing sugars, the defecated solution is hydrolyzed in an acidic and hot medium. We took a volume of this solution and then we proceeded as for reducing sugars. Hydrolysis allows us to determine the total sugars (reducing sugars + hydrolyzable sugars) and to indirectly deduce the level of nonreducing sugars (total sugars-reducing sugars) [12].

(7) Pectin Content. The pectin extraction method used is that described by Multon [13]. It is based on the principle of transformation of pectin into calcium pectate.

(8) Protein Content. The proteins were assayed according to the method of Bradford [14]. A calibration curve is established from a standard solution of BSA (10 to 90 μg/ml).

(9) Ash Content. The determination of ash is based on the destruction of all organic matters under the effect of high temperature ($500 \pm 25^{\circ}$C) [15].

(10) Ascorbic Acid Content. The ascorbic acid content is determined according to the method of Tillmanns cited by Anonyme [16], which is based on the quantitative reduction of 2.6 DPIP (dichlorophenolindophenol) to leuco derived from the reduced form of ascorbic acid (the oxidized form of 2.6 DPIP is pink in acid medium).

(11) Carotenoids. The carotenoid content is determined by the AOAC method [17], and β-carotene was used as standard.

(12) Phenolic Compounds. Ten grams of jam were extracted with 200 ml of methanol-water (700 : 300, v/v) at room temperature for 24 h using a magnetic blender. Then, the extract was vacuum-filtered through sintered glass filter crucibles (porosity 3) and vacuum-filtered using Whatman No. 1 paper. The obtained aqueous organic extract was concentrated, under reduced pressure in rotary evaporation at 40°C, until complete evaporation of organic solvent then reconstituted in pure methanol. The amount of total phenolics in the extract was determined using the Folin–Ciocalteu reagent and gallic acid as standard as described by Meyers et al. [18]. *Proanthocyanidins* were determined by the vanillin reagent assay according to Ba et al. [19], and catechin was used as standard. Colorimetric aluminum chloride method was used for *flavonoids* and *flavonols* determination, using the optimized protocols established by Bahorun et al. [20] and Kumaran and Karunakaran [21, 22], respectively. Quercetin was used as standard.

2.2.4. Determination of the Antioxidant Activity

(1) Reducing Power. The reducing power was determined according to the method of Oyaizu [23]. Quercetin and gallic acid were used for comparison.

(2) Scavenging Activity against the DPPH Radical. The stable radical 1,1-diphenyl 1-2-picrylhydrazyl (DPPH) was used for determination of free radical scavenging activity of the extracts [24]. Quercetin and gallic acid were used for comparison.

Table 1: Effect of desamerization on the physicochemical characteristics of jams and comparison with parts of the fruit.

Jams	pH	Acidity (g CAE/l)	Brix (%)	Moisture (%)
Bitter	2.80 ± 0.00^{f}	27.84 ± 0.4^{b}	59.67 ± 0.58^{cde}	38.433 ± 0.09^{e}
DJW	2.87 ± 0.05^{b}	24.84 ± 0.00^{cde}	59.67 ± 2.52^{cd}	39.494 ± 0.47^{d}
DJ NaCl 2.5%	2.84 ± 0.01^{bc}	24.33 ± 0.53^{cde}	65.33 ± 0.58^{a}	34.790 ± 0.02^{f}
DJ NaCl 1.25%	2.83 ± 0.01^{bcd}	24.35 ± 1.08^{cd}	64.33 ± 0.58^{ab}	36.084 ± 1.31^{c}
DJ NaCl 0.625%	2.84 ± 0.01^{bc}	24.33 ± 0.53^{cde}	62.33 ± 0.58^{c}	39.544 ± 0.63^{c}
DJ NaCl 0.3125%	2.82 ± 0.01^{bcde}	24.96 ± 0.57^{c}	64 ± 1.00^{ab}	33.826 ± 0.21^{g}
Peels	4.16 ± 0.01^{a}	20.91 ± 0.36^{f}	11.67 ± 0.58^{f}	67.00 ± 0.18^{b}
Edible portion	2.51 ± 0.01^{g}	48.00 ± 1.00^{a}	10.67 ± 1.15^{fg}	89.697 ± 0.29^{a}

CAE: citric acid equivalents, DJW: desamerized jam with water, and DJ: desamerized jam. Values with the same letter in each column show no significant difference ($p > 0.05$). Results are ranked in descending order: a > b > c > d > e > f > g.

2.2.5. Sensory Analysis

(1) Grading Test. The subject's task (30 trained subjects) consists in presenting him with a series of coded jams and asking him to classify them in order of intensity of bitterness. The samples are presented simultaneously in increasing (or decreasing) intensity of the analyzed characteristics [25].

(2) Hedonic Test. Hedonic tests are designed to measure the degree of appreciation of a product. We use categories ranging from "likes a lot" to "do not like at all" through "neutral" with a variable number of intermediate categories. For each sample, the tasters select the category corresponding to their degree of appreciation. Compared to the classification test, it has the advantage of not requiring the simultaneous evaluation of all the products studied, but it supposes a good sensory memory of the scale of notation. Codified products are presented to a panel of 30 subjects. Each subject receives the samples in cups coded with three digits. The order of presentation of samples is random [25].

2.2.6. Statistic Study. The statistical analysis is carried out using the software Statistica 5.5. Analysis of variance was performed by ANOVA procedure with one factor for the physicochemical and antioxidant results. For the results of the sensory analysis, we applied the test of multiple comparisons in pairs of the sums of the rows. For the results of the hedonic analysis, the categories are converted into numerical notations ranging from 1 to 9, where 1 corresponds to "does not like at all" and 9 "loves a lot." The scores for each sample are presented in tabular form and analyzed by means of the variance analysis.

3. Results and Discussions

No data were available on the physicochemical parameters and the desamerisation of Bigarade jam, which makes comparison very difficult. So, all of the comparisons were made with results obtained on jam made from other fruits.

3.1. Physicochemical Analyzes. The physicochemical characteristics of the jams and parts of the fruit analyzed are illustrated in Table 1.

3.1.1. Hydrogen Potential (pH). pH is a parameter determining the suitability of food for conservation, and it is one of the main obstacles that microbial flora must overcome to ensure its proliferation. Thus, a pH of the order of 3 to 6 is very favorable to the development of yeasts and molds [26].

According to the results presented in Table 1, the pH varies significantly ($p \leq 0.05$) according to the parts of the fruit. The pH of the edible portion of the fruit is 2.51, which classifies the fruit as an acid fruit. Karadeniz [27] reported a pH value of Bigarade juice similar to ours (2.52).

The pH differs significantly ($p \leq 0.05$) between the bitter jam and the desamerized jams. The pH of jams varied from 2.80 to 2.87. These results are consistent with those required by the Codex Alimentarius (pH < 3.5). The pH of jam is an important factor to obtain an optimum gel condition [28]. Control of pH is critical to successful gel formation with pectins, particularly high methoxyl pectins. Low pH increases the percentage of unionized carboxyl groups, thus reducing electrostatic repulsion between adjacent pectin chains [29]. Our results are slightly higher than those found by Ellouze et al. [30] who worked on bitter orange marmalade. The latter yielded pH values between 2.3 and 2.6.

3.1.2. Titratable Acidity. The titratable acidity tells us the amount of organic acids present in the sample. Organic acids are, in general, intermediates of metabolic processes; they influence the growth of microorganisms and affect the quality of preservation of the products. They are directly involved in the growth, maturation, and senescence of the fruit. These acids also influence the sensory properties of fruits [31].

According to our results, the titratable acidity varies significantly ($p \leq 0.05$) depending on the parts of the fruit. The acidity of peels and edible part is 20.91 g/l and 48.00 g/l, respectively (Table 1). The results found for the edible portion are similar to those found by Ellouze et al. [30] (49.92 g/l) in Bigarade juice.

The bitter jam is considered to be significantly ($p \leq 0.05$) the most acidic (27.84 g/l). The acidity of the desamerized jams varies from 24.33 g/l to 24.96 g/l. The acidity is usually provided by the fruit which contains citric acid, tartaric acid, etc. [32].

3.1.3. Rate of Soluble Solids (Brix). From our results (Table 1), it is noted that the soluble solid contents of the peels is statistically similar ($p > 0.05$) to that of the edible portion.

The Brix level of the peels (11.67 ± 0.58) is slightly lower than the rate found by Moufida and Marzouk [33]. These authors have recorded a value of 12.24% for bitter orange. This difference in outcome can be explained by the influence of a few parameters such as climate, soil type, and fruit maturation process [9].

The Brix level of our jams is in the range of 59.67 to 65.33%. Sugar brings consistency and mass to the product and also promotes gelling [34].

3.1.4. Moisture Content. According to the results presented in Table 1, the moisture varies significantly ($p \leq 0.05$) from one jam to another depending on the parts of the fruit. The average moisture content of the fruit (peels and edible portion) is 78.34%. The moisture is highest in the edible part (89.69%) compared to that registered in peels (67%). These results are lower than those found by Lagha-Benamrouche and Madani [35]. The latter recorded a content of 75.82% for the peels and a value of 94.91% for the pulp. This variation in water content may also be due to different environmental conditions: exposure to different pedoclimatic conditions and geographical distribution [36].

By comparing the moisture content of our jams (33.82 to 39.54%) with those found by Mohd Naeem et al. [37] for fruit jams (strawberry, apricot, and blueberry (31.23 to 33.36%)) and Aina et al. [38] for pineapple jam (30%), we find that our jams are the moistest. Generally, the moisture content of foods can be used as an indicator of its shelf life. Low moisture content indicates that the jams have a long shelf life [39].

3.2. Chemical Composition of Jams and Fruit Parts. The chemical composition of the jams and the fruit parts studied are shown in Table 2. The spider diagram (Figure 1) allows better visualization of the effect of desamerization on the chemical composition of jams.

3.2.1. Ashes. From the obtained results (Table 2), we can observe that the ash content varies significantly ($p \leq 0.05$) according to the analyzed samples. The mineral content of the peels (0.67%) is higher compared to that registered in the edible part (0.33%). The data analysis shows that also the ash content of the peels is higher than the result reported by Aissou [40] for orange peel, lemon, and grapefruit. The latter are of the order of 0.30%, 0.32%, and 0.26%, respectively. The variation in the ash content of the fruit can be explained by the geographical origin, the climatic conditions, and the edaphic characteristics of the soils [41].

Concerning jams (Table 2), the ash content of the bitter jam is higher than the result reported by Aina et al. [38] for pineapple jam and Mohd Naeem et al. [37] for apricot jam. The latter are of the order of 0.05% and 0.25%, respectively. Minerals are essential for the proper functioning of tissues and act as second messengers in some biochemical cascade mechanisms [42]: average catalysts, mediate cell responses, control growth, and cell differentiation [43].

The results show that also the ash content proportionally increases with salt concentration in jams. This increase is due to the dissolution of NaCl in the medium giving Na^+ and Cl^-, which are mineral salts.

3.2.2. Pectins. Pectin is a polysaccharide present in plant cell walls, especially in fruits. Pectin is a component of soluble fiber with interesting technological applications in the gelling of a mixture of fruit and vegetable sugar.

According to our results, the peels are richer in pectin than the edible part (0.74% versus 0.11%, resp.) (Table 2). Comparing our results with those obtained by Sulieman et al. [44], for sweet oranges, it is found that our present fruit has much lower pectin contents (1 to 3.5% versus 0.1 to 0.74%, resp.). According to Kansci et al. [45], this difference may be due to the cultivation conditions and degree of ripeness of the fruit but also to the dosage method used. Technically, the low pectin content is an advantage in the production of clarified juices and syrups (clarification process) but a disadvantage for the preparation of jellies, jams, and marmalades.

The results obtained for the jams (Table 2) show that there are no significant differences ($p > 0.05$) in the pectin contents of desamerized jams and bitter jam; the desamerization does not seem to affect the pectin content of the jams.

3.2.3. Proteins. The results shown in Table 2 reveal that the Bigarade peels are richer in protein than the edible portion. The contents vary significantly ($p \leq 0.05$) from 1.679 to 0.076 g/100 g FM, respectively. The protein content of Bigarde peels (=1.7 g/100 g FM.) was comparable to the one reported for Thomson peels (1.8 g/100 g FM.) [46].

The protein content in the jams vary from 0.485 g/100 g FM to 0.98 g/100 g FM, comparing our results with those obtained by Mohd Naeem et al. [37] and Eke-Ejiofor and Owuno [47] for apricot (0.43 g/100 g FM.) and pineapple jams (0.46 g/100 g FM.), respectively. According to the jams' nutrition labelling, common ingredients are fruits, sugar, pectin, and citric acid. None of the ingredients used are an abundant source of protein; hence, this richness of protein is explained by the richness of our fruit, peels, in this compound. Bitter jam is the richest in protein followed by DJW. The results also show that desamerization decreases the protein content of the jams. Proteins are more soluble in solutions of ionic salts than in distilled water. At higher salt concentrations, protein solubility usually decreases, leading to precipitation; this effect is termed salting-out. Salts that reduce the solubility of proteins also tend to enhance the stability of the native conformation. In contrast, salting-in ions are usually denaturants [48].

3.2.4. Total Sugars. The results shown in Table 2 reveal that the total sugar contents of the peels and the edible part are in the range from 10.77 to 10.03 g GE/100 g FM, respectively. Our results show that our fruit contains total sugar content close to those found by Ellouze et al. [30]. The latter yields between 9.61 and 11.40 g/100 g FM.

This variation can be attributed to several factors such as plant age, maturation stage, and fruit physiological state during the analysis [49].

Table 2: Effect of desamerization on the chemical composition of jams and comparison with parts of the fruit.

	Bitter	DJW	DJ NaCl 2.50%	DJ NaCl 1.25%	DJ NaCl 0.625%	DJ NaCl 0.3125%	Peels	Edible portion
Ashes (g/100 g FM)	$0.328^{g} \pm 0.002$	$0.400^{f} \pm 0.002$	$0.916^{a} \pm 0.001$	$0.636^{c} \pm 0.002$	$0.594^{d} \pm 0.003$	$0.458^{e} \pm 0.003$	$0.670^{b} \pm 0.002$	$0.300^{gh} \pm 0.030$
Proteins (g BSA E/100 g FM)	$0.955^{c} \pm 0.003$	$0.98^{b} \pm 0.015$	$0.485^{g} \pm 0.022$	$0.568^{f} \pm 0.039$	$0.67^{e} \pm 0.007$	$0.738^{d} \pm 0.018$	$1.679^{a} \pm 0.017$	$0.076^{h} \pm 0.004$
Total sugars (g G E/100 g FM)	$57.5^{a} \pm 0.05$	$53.57^{bcde} \pm 0.764$	$53.47^{bcd} \pm 1.601$	$54.77^{b} \pm 1.626$	$54.57^{bc} \pm 0.929$	$53.5^{bcd} \pm 1.706$	$10.77^{f} \pm 0.416$	$10.03^{f} \pm 1.115$
Pectins (g/100 g FM)	$0.57^{DJ} \pm 0.01$	$0.56^{bcd} \pm 0.04$	$0.55^{b} \pm 0.06$	$0.55^{cd} \pm 0.03$	$0.59^{bc} \pm 0.01$	$0.56^{b} \pm 0.06$	$0.74^{a} \pm 0.04$	$0.11^{e} \pm 0.29$
Vitamin C (mg AAE/100 g FM)	$33.17^{c} \pm 2.29$	$29.33^{cd} \pm 2.31$	$17.19^{fg} \pm 2.29$	$21.29^{ef} \pm 2.3$	$23.95^{e} \pm 0$	$24.23^{de} \pm 4.48$	$56.99^{a} \pm 2.29$	$44.49^{b} \pm 2.27$
Reducing sugars (g GE/100 g FM)	$20.53^{f} \pm 0.14$	$21.43^{e} \pm 0.16$	$35.02^{a} \pm 0.78$	$23.34^{b} \pm 0.38$	$23.33^{bcd} \pm 0.11$	$23.34^{bc} \pm 0.22$	$2.42^{h} \pm 0.29$	$3.91^{g} \pm 0.08$
Nonreducing sugars (g/100 g FM)	$26.22^{a} \pm 1.65$	$15.74^{c} \pm 0.62$	$13.33^{de} \pm 0.12$	$18.36^{b} \pm 1.24$	$13.34^{d} \pm 0.3$	$13.39^{d} \pm 0.24$	$4.84^{g} \pm 0.77$	$6.63^{f} \pm 0.49$
Carotenoids (mg BCE/g FM)	$1.42^{b} \pm 0.03$	$1.26^{c} \pm 0.11$	$0.21^{e} \pm 0.07$	$0.25^{e} \pm 0.02$	$0.91^{d} \pm 0.21$	$1.27^{c} \pm 0.11$	$1.02^{d} \pm 0.11$	$2.18^{a} \pm 0.11$

AAE: ascorbic acid equivalents, GE: glucose equivalents, BSA E: bovin serum albumin equivalents, βCE: β carotene equivalents, FM: fresh matter, DJW: desamerized jam with water, and DJ: desamerized jams. Values with the same letter in each row show no significant difference ($p > 0.05$). Results are ranked in descending order: a > b > c > d > e > f > g > h.

For the jams, the results show that the total sugar levels differ significantly ($p \leq 0.05$) between the bitter jam and the desamerized jams. This can be explained by the phenomenon of diffusion of the matter (osmosis) and the training of the sugars with the washing waters of the mesocarpe during the desamerization.

The total sugar content of our jams (57.5–53.47 g GE/100 g FM) is lower than the bibliographic data. Aissou [40] recorded a rate of 68 g/100 g FM for a jam based on orange, lemon, and grapefruit pulp. The noticeable differences could be caused by the addition of sugar during the jam-making process [50]. According to Oakenfull [34], the sugar attracts water molecules, which concentrates the pectin molecules and promotes gelling. The addition of sugar is essential in order to preserve the jams satisfactorily. In comparison with apricot jam, Touati et al. [51] reported total sugars to be higher (64.88 g/100 g) than in the investigated jam. This difference may be related to the low soluble solid content of our fruit.

3.2.5. Reducing Sugars. The results shown in Table 2 indicate that the reducing sugar contents of the edible portion (3.91 ± 0.08 g/100 g FM) and the peels (2.42 ± 0.29 g/100 g FM) are included in the interval given by Aissou [40] for sweet oranges (2.25–3.83 g/100 g FM). According to Ayaz [52], the variation in the levels of reducing sugars can be attributed to various factors including the maturation stage, temperature, duration of exposure to the sun, and climatic conditions and also to genetic factors.

Reduced sugars varied significantly ($p \leq 0.05$) between jams with exception for DJ NaCl 0.3125%, DJ NaCl 1.25%, and DJ NaCl 0.625%. It is the bitter jam which is the poorest in reducing sugars (20.53 g/100 g FM), followed by DJW (21.43 g/100 g FM). The highest content was found for DJ NaCl 2.5% (35 g/100 g FM). This can be explained by the hydrolysis of sucrose contained in the jam during cooking. The acidity associated with a high temperature causes the inversion of 30–50% of added sucrose. The inversion of sucrose has its consequences:

(i) Increased sweetness: fructose is sweeter than sucrose (sweetening power = 1.14).

(ii) Obtaining a solution containing more dry matter: fructose and glucose which are more soluble than sucrose [53].

3.2.6. Nonreducing Sugars. Comparison of the nonreducing sugar content of the edible part of the fruit with that of the peels shows the richness of the edible part of these compounds. Values ranged from 6.63 to 4.84 g/100 g FM, respectively.

The nonreducing sugar content of the jams varied significantly ($p \leq 0.05$) between the jams except for DJ NaCl 0.625%, DJ NaCl 2.5%, and DJ NaCl 0.3125%. The contents range from 13.33% to 26.22%. Bitter jam contains more nonreducing sugars; the lowest content is found in the DJ NaCl 2.5%, DJ NaCl 0.625%, and DJ NaCl 0.3125%. This can be explained by the effect of cooking and salt used for desamerization. The presence of salt causes training of the sucrose with the washing waters of the mesocarpe during the desamerization. Cooking reduces sucrose content (which is a nonreducing sugar) following its hydrolysis into glucose and fructose.

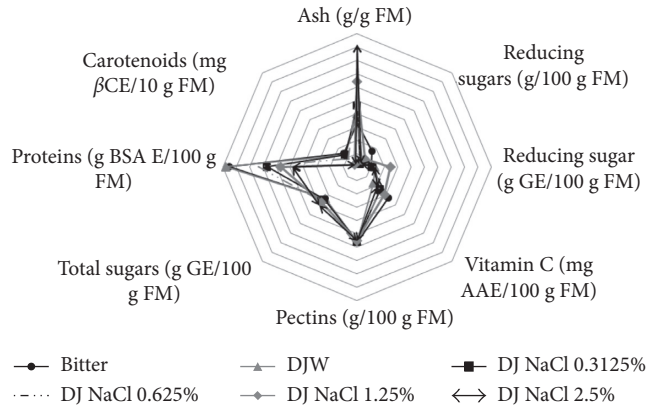

Figure 1: Comparison of effect of desamerization on the chemical composition of jams. AAE: ascorbic acid equivalents, GE: glucose equivalents, BSA E: bovin serum albumin equivalents, βCE β carotene equivalents, AAE: ascorbic acid equivalents, FM: fresh matter, DJW: desamerized jam with water, and DJ: desamerized jams.

3.2.7. Vitamin C. The results of the determination of vitamin C in different parts of the fruit show that peels contain more vitamin C compared to the edible part. The contents range from 56.99 to 44.49 mg/100 g FM, respectively. Our result corroborates those of Gorinstein et al. [54]. These authors have reported that citrus peels appear to contain more vitamin C than the edible part (pulp). It contains only 0.477 mg/g FM for orange, 0.479 mg/g FM for lemon, and 0.351 mg/g FM for grapefruit, compared to 0.596, 0.598, and 0.438 mg/g FM for the peels of the same varieties of citrus, respectively. These results also show that orange peel is the richest in ascorbic acid. The variability of the ascorbic acid content of fruits is influenced by the seasonal and annual variations in the degree of sunshine and humidity, the variety of the fruit, the position of the fruits on the tree, and their degrees of ripeness [55]. Other factors may also be involved, such as the sensitivity of ascorbic acid to oxidation by air and in an aqueous medium. Quantification is also influenced by the assay method, which is itself dependent on the complexity of the plant material [56].

For jams, the results show that the vitamin C levels decrease significantly ($p \leq 0.05$) with desamerization. The levels pass from 33.17 mg/100 g FM for bitter jam to 17.19 mg/100 g FM for DJ NaCl 2.5%. This tremendous decrease in vitamin C content in jam is due to the use of heat treatment in the desamerization processing and probably is mainly due to oxidation of vitamin C. The oxidation of vitamin C may also be due to the elimination of some bitter flavonoids. According to Dupaigne [57], degradation of naringine during desamerization is accompanied by loss of vitamin C.

The analyzed jams have vitamin C contents between 17.19 and 33.17 mg/100 g FM. The vitamin C content of our

Table 3: Effect of desamerization on the phenolic composition of jams and comparison with parts of the fruit.

	Bitter	DJW	DJ NaCl 2.50%	DJ NaCl 1.25%	DJ NaCl 0.625%	DJ NaCl 0.3125%	Peels	Edible portion
Total phenol (mg GAE/g FM)	$33.61^{a} \pm 0.25$	$31.01^{c} \pm 0.05$	$18.18^{f} \pm 1.56$	$22.43^{e} \pm 2.16$	$27.38^{d} \pm 1.72$	$32.09^{b} \pm 0.63$	$7.45^{g} \pm 0.20^{g}$	$31.96^{bc} \pm 0.03$
Flavonoids (mg QE/g FM)	$6.39^{b} \pm 0.20$	$6.29^{bc} \pm 0.10$	$4.83^{ef} \pm 0.77$	$5.51^{e} \pm 0.07$	$6.10^{d} \pm 0.01$	$6.24^{bc} \pm 0.12$	$1.68^{g} \pm 0.17$	$10.8^{a} \pm 0.04$
Flavonols (mg QE/g FM)	$1.75^{a} \pm 0.10$	$1.49^{bc} \pm 0.12$	$0.56^{fg} \pm 0.17$	$0.76^{f} \pm 0.06$	$1.14^{e} \pm 0.17$	$1.23^{cd} \pm 0.06$	$0.29^{h} \pm 0.02$	$1.59^{ab} \pm 0.11$
Proanthocyanidins (μg CE/g FM)	$40.91^{b} \pm 0.30$	$40.53^{c} \pm 0.05$	$36.36^{g} \pm 0.15$	$38.24^{ef} \pm 0.14$	$38.42^{e} \pm 0.25$	$40.47^{cd} \pm 0.09$	$0.07^{h} \pm 0.00$	$51.05^{a} \pm 0.29$

GAE: gallic acid equivalents, CE: catechin equivalents, QE: qercetin equivalents, FM: fresh matter, DJW: desamerized jam with water, and DJ: desamerized jams. Values with the same letter in each row show no significant difference ($p > 0.05$). Results are ranked in descending order: a > b > c > d > e > f > g > h.

jams is much higher than the results obtained by Tanwar et al. [58]. The authors have reported vitamin C levels of around 7.5 mg/100 g for the guava jam. We found that our jams are very rich in vitamin C. This richness of ascorbic acid is explained by the richness of our fruit, peels, in vitamin C [35]. The latter refers levels of vitamin C in the range of 9.12 mg AAE/g FM.

3.2.8. Carotenoids. According to our results, the peels are richer in carotenoids than the edible part (2.18 mg βCE/g FM, versus 1.02 mg βCE/g FM, resp.). Our results corroborate those of Wang et al. [59]. These authors have reported that orange peels are richer in carotenoids than the edible part of the fruit (445 μg Eβc/g FM for peels versus 5.17 μg Eβc/g FM for the edible part).

The results obtained for jams (Table 2) show that the carotenoid content varies significantly ($p \leq 0.05$) between jams except for (DJ NaCl 1.25%-DJ NaCl 2.5%) and (DJ 0.3125% NaCl-DJW). On the basis of carotenoid content, jams are classified in descending order: bitter jam (1.42 mg βCE/g) > DJW-DJ NaCl 0.3125% > DJ NaCl 0.625% > DJ NaCl 1.25%-DJ NaCl 2.5% (0.21 mg βCE/g). The results also show that bitter jam contains fewer carotenoids than fresh fruit. Since there are double bonds in the carbon chain, carotenoids are susceptible to some reactions such as oxidation and isomerisation (cis-trans) during food processing and storage, especially due to light, heat, acids, and oxygen, thus causing loss of color and reduction of biological activity [60].

3.2.9. Phenolic Contents of Sample. The results of the determination of phenolic content in different parts of the fruit show that the peels contain more total polyphenols, flavonoids, flavonols, and proanthocyanidins compared to the edible part Table 3. The total polyphenol content of the peels is four times higher than that of the edible part (31.96 mg GAE/g FM versus 7.45 mg GAE/g FM), while the flavonoid and flavonol contents are approximately six times higher (10.8 mg EQ/g FM versus 1.68 mg EQ/g FM and 1.59 mg EQ/g FM versus 0.29 mg EQ/g FM, resp.). The results also show that the proanthocyanidin contents of the edible part is negligible compared with that of the peels (0.07 mg EC/g FM versus mg EC/g FM). Our results corroborate those of Guimarães et al. [61]. These authors have reported that citrus peels appear to contain more phenolic content than the edible part.

For jams, the results show that the phenolic levels decrease significantly ($p \leq 0.05$) with desamerisation. The levels pass from 33.61 mg GAE/g for bitter jam to 18.18 mg GAE/g for DJ NaCl 2.5% concerning total phenol, from 6.39 mg QE/g to 4.83 mg QE/g for flavonoides, from 1.75 mg QE/g to 0.56 mg QE/g for flavonols, and from 40.91 mg CE/g to 36.36 mg CE/g, respectively. On the basis of the content of phenolic compounds, jams are classified in descending order: bitter jam > DJW-DJ NaCl 0.3125% > DJ NaCl 0.625% > DJ NaCl 1.25%-DJ NaCl 2.5%. The results show that the phenolic compound levels significantly decrease ($p \leq 0.05$) with desamerisation. This decrease can be explained by the effect of cooking and salt used for desamerization. The presence of salt causes training of the phenolic compounds, which are water soluble, with the washing waters of the mesocarpe during the desamerization. Other factors may also be involved, such as the sensitivity of phenolic compounds to oxidation by air and temperature and in an aqueous medium. Klopotek et al. [62] show that treating strawberry at 80°C for 15 minutes causes a 30% loss of phenolic compounds. Water is a source of degradation of phenolic compounds; in the presence of water, an enzymatic activity may quickly cause irreversible changes in antioxidants, such as oxidation which leads to their decomposition or polymerization [63]. The comparison of our results with historical data shows that Bigarade jam is very rich in total polyphenols and flavonoids compared to strawberry jam. According to Plessi et al. [64] and Danijela et al. [65], the contents of these compounds are 310–510 mg GAE/100 g and 0.7–0.75 mg/100 g, respectively.

3.3. Antioxidant Activity. The antioxidant potential of jams was estimated using the method of reduction of potassium ferricyanide. The presence of reducing agents in the extracts induced reduction of the ferric ions (Fe^{+3}) to ferrous ion (Fe^{+2}). This reduction is measured by the intensity of the blue-green color that results. It absorbs at a wavelength of 700 nm. An increase in absorbance indicates a high reducing power.

The analysis of the reducing power of jams at the concentration of 1 mg/ml resulted in absorbances between 0.805 and 0.556 (Figure 2). As can be seen, it is the bitter jam that has the highest absorbances and therefore the most pronounced reductive power. On the basis of the reducing capacity, the jams are classified in descending order as following: bitter jam > DJW-DJ NaCl 0.3125% > DJ NaCl 0.625% > DJ NaCl

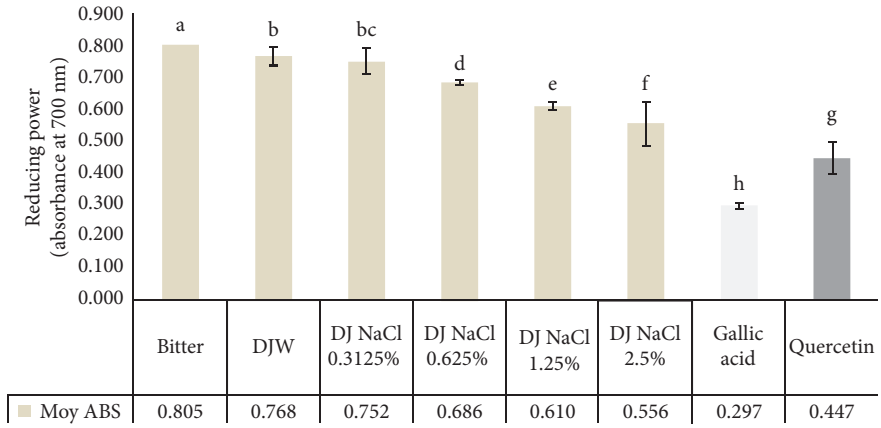

Figure 2: Reducing power of jams at 1 mg/ml, quercetin and gallic acid at 0.02 mg/ml. FM: fresh matter, DJW: desamerized jam with water, and DJ: desamerized jams.Values bearing the same letter showed no significant difference ($p \leq 0.05$). The results are sorted in decreasing order: a > b > c > d > e > f > g > h.

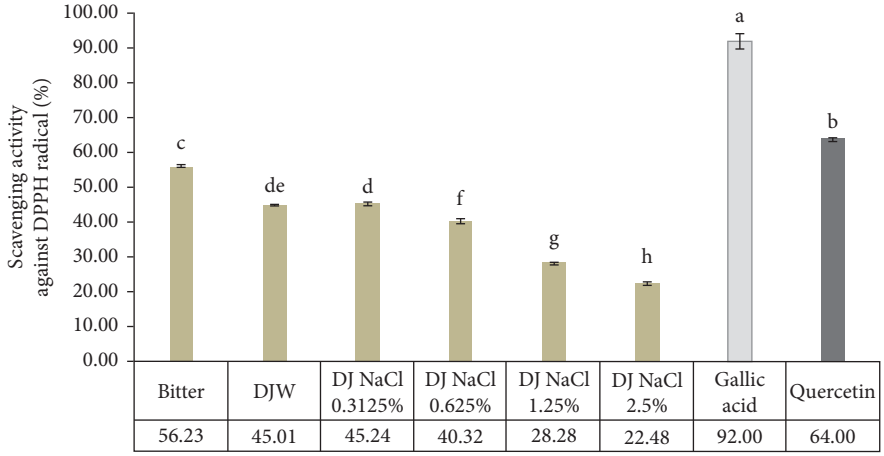

Figure 3: Mean antioxidant activity against DPPH radical of jams at 1 mg/ml and standards at 40 μg/ml. DJW: desamerized jam with water, DJ: desamerized jams, and DPPH: 1,1-diphenyl 1-2-picrylhydrazyl. Values bearing the same letter showed no significant difference ($p \leq 0.05$). The results are sorted in decreasing order: a > b > c > d > e > f > g > h.

1.25% > DJ NaCl 2.5%. These results also show that the reducing power of jams is significantly higher ($p \leq 0.05$) than that of standards tested at 0.02 mg/ml: quercetin and gallic acid (respective absorbances: 0447 and 0.29).

The scavenging model of DPPH radical is widely used as a method for assessing antioxidant activity in a period relatively short compared to other methods. As displayed in Figure 3, the antiradical activity values for investigated extracts (at a concentration of 1 mg/ml) varied between 56.23% and 22.48%. The bitter jam presents the highest antiradical activity. The latter is significantly lower ($p \leq 0.05$) than that of the standards tested. Based on the antiradical activity, the jams are classified in the following order: bitter jam> DJW-DJ NaCl 0.3125% > DJ NaCl 0.625% > DJ NaCl 1.25% > DJ NaCl 2.5%. These results show that the antioxidant potential is inversely proportional to the salt concentration used for the desamerisation. This can be explained by the loss of compounds with antioxidant potential as already observed during their quantification.

Comparing our results with the bibliographic data leads us to say that Bigarade jam exhibits a higher antioxidant activity than melon jam which was 4.95% according to Benmeziane et al. [63] and than cherry, apricot, and fig jam (10.06%, 9.95%, and 8.96%, resp.), according to Rababah et al. [66].

The coefficients of correlation between the antioxidant capacities of jams and the contents of bioactive compounds are illustrated in Table 4. Significant positive correlations ($p \leq 0.05$) were observed between the contents of bioactive compounds (polyphenols, flavonoids, flavonols, and carotenoids) and the antioxidant capacities evaluated by the methods of reducing power and inhibition of the radical DPPH ($R2 = 0.753–0.981$). Similar results have been observed by Lagha-Benamrouche and Madani [35]. However, low correlation coefficients were observed between vitamin C and proanthocyanidin levels and antioxidant activities ($R2 = 0.025–0.437$).

3.4. Results of Sensory Evaluation

3.4.1. Rankings by Degree of Bitterness. To process the data from the ranking tests in a first step, we calculated all the differences between the sums of the ranks of the products

Table 4: Correlation matrix between the levels of bioactive compounds and the antioxidant activities of jams.

	TP	F	FOL	PAC	VC	Car	RP	DPPH
TP	1	—	—	—	—	—	—	—
F	0.870*	1	—	—	—	—	—	—
FOL	0.829*	0.690*	1	—	—	—	—	—
PAC	0.519	0.816*	0.297	1	—	—	—	—
VC	0.033	0.003	0.045	0.657*	1	—	—	—
Car	0.956*	0.800*	0.898*	0.476	0.003	1	—	—
RR	0.981*	0.804*	0.954*	0.437	0.025	0.963*	1	—
DPPH	0.948*	0.753*	0.961*	0.341	0.086	0.931*	0.963*	1

TP: total polyphenol, F: flavonoids, FOL: flavonol, PAC: proanthocyanidins, VC: vitamin C, Car: carotenoids, RP: reducing power, and DPPH: 1.1-diphenyl 1-2-picrylhydrazyl. *Significant correlation ($p < 0.05$).

Table 5: Multiple comparisons by pair of the sums of rows for the degree of bitterness of the jams.

Samples	Effective (n)	Sums of ranks	Differences in the ranks between the possible pairs	Critical value for $p \leq 0.05$ and effective = 30 *number of products* = 6	Groups
Bitter (1)	30	30	(2)-(1) = 33 (3)-(1) = 86* (4)-(1) = 78* (5)-(1) = 107* (6)-(1) = 149*	42	A
DJW (2)	30	63	(3)-(2) = 53* (4)-(2) = 45* (5)-(2) = 74* (6)-(2) = 116*	42	A
DJ NaCl 0.312% (3)	30	116	(3)-(4) = 08 (5)-(3) = 21 (6)-(3) = 63*	42	B
DJ NaCl 0.625% (4)	30	108	(5)-(4) = 29 (6)-(4) = 61*	42	B
DJ NaCl 1.25% (5)	30	137	(6)-(5) = 42*	42	B
DJ NaCl 2.5% (6)	30	179	—	—	C

DJW: desamerized jam with water; DJ: desamerized jam. *Significant at the $p \leq 0.05$ level.

taken 2 to 2. Then, in a second step, we read the critical value at the intersection of the column (products) and line (subjects) on the table of Newell and MacFarlane corresponding to the risk $\alpha \leq 5\%$. Any calculated difference between the products, equal to or greater than this critical value, means that the corresponding products can be regarded as different (Table 5).

The calculated value for bitter jam and desamerized jam with water is less than the critical value. We conclude, therefore, that there is no discernible difference between the two jams, and the latter are classified in the same group (A) and possess the same degree of bitterness.

The multiple paired comparisons of the three desamerified jams with NaCl at the following percentages 0.3125%, 0.625%, and 1.25% showed no significant difference in the degree of desamerization between them; the tasters find that these jams are less bitter than the previous ones but do not differ between them, and they are therefore classified in another group (B). The calculated value for desamerified jams with NaCl at the percentages 1.25% and 2.5% is equal to the critical value. It is concluded that the difference in perceived bitterness between the two samples is significant, and the two jams are classified into two different groups.

3.4.2. Hedonic Test. After the evaluation of the five samples, the descriptive categories were converted into numerical notations. The results are tabulated and analyzed for variance (Table 6).

The coefficients F for treatment and tasters were calculated by dividing the respective AS (average square) values by the AS of the error. The calculated F coefficients must exceed the F coefficients in the F distribution table (significance at $p \leq 5\%$).

As the coefficient F calculated for the treatment (F 25.13) exceeded the coefficient of the table (F 2.6), it was concluded that there is a significant difference ($p \leq 0.05$) between the averages of the hedonic results for the five desamerized jams. The results indicate that also the calculated coefficient for the tasters (F 3.95) exceeds the coefficient of the table (F 2.07). Such results reveal, therefore, a significant effect attributable to the tasters.

Analysis of variance indicated that there were significant differences between the five desamerized jams. In order to determine which samples of jam differ significantly from each other, a multiple comparison test was conducted; Duncan's new multiple comparison test was conducted using the critical value tables (Q values) at a significance level of 5%. This test compares the differences between all pairs of

Table 6: Results by category of the hedonic test.

	Desamerized jams (processing)						
Tasters	DJW	DJ NaCl 2.5%	DJ NaCl 1.25%	DJ NaCl 0.625%	DJ NaCl 0.3125%	Total tasters	Average tasters
1	1	1	—	—	1	3	0.6
2	6	—	4	—	6	16	3.2
3	—	1	7	—	—	8	1.6
4	—	6	—	—	7	13	2.6
6	—	—	—	7	—	7	1.4
8	4	—	—	—	—	4	0.8
9	3	3	5	9	9	29	5.8
10	2	—	—	—	—	2	0.4
13	—	4	—	—	—	4	0.8
14	—	—	—	—	8	8	1.6
15	—	—	—	8	—	8	1.6
16	—	—	6	—	—	6	1.2
Total treatment	16	15	22	24	31		
Grand total						108	
Average treatment	3.2	5	5.5	8	6.2		

Highest score = 9 (likes enormously), lowest score = 1 (disliked). DJ: desamerized jam. DJW: desamerized jam with water; DJ: desamerized jam.

Table 7

Desamerized jams	DJW	DJ NaCl 2.5%	DJ NaCl 1.25%	DJ NaCl 0.3125%	DJ NaCl 0.625%
Average treatment	3.2	5	5.5	6.2	8

Table 8: Paired comparisons of average treatments for the degree of appreciation of jams.

Samples	Differences in means between the possible pairs	Deviation value at $p \leq 0.05$	Groups
DJ NaCl 0.625% (1)	(1)-(5) = 4.8*	1.59	A
	(1)-(4) = 3*	1.56	
	(1)-(3) = 2.5*	1.51	
	(1)-(2) = 1.8*	1.43	
DJ NaCl 0.3125% (2)	(2)-(5) = 3*	1.56	B
	(2)-(4) = 1.2	1.51	
	(2)-(3) = 0.7	1.43	
DJ NaCl 1.25% (3)	(3)-(5) = 2.3*	1.51	B
	(3)-(4) = 0.5	1.43	
DJW (4)	(4)-(5) = 1.8*	1.43	B
DJ NaCl 2.5% (5)	—	—	C

DJW: desamerized jam with water; DJ: desamerized jams. *Significant at $p \leq 0.05$. A, B, and C: the homogeneous groups.

averages to calculate the deviation values of each pair. If the difference between the pairs of averages is greater than the value of the calculated deviation, the difference between the averages is significant at the given significance level. The deviation values are calculated based on the number of averages between the two averages tested, when the averages are placed in the order of size.

To calculate the Duncan test, the processing averages were placed in descending order as shown in Table 7.

To compare the means of this example, the deviation values for a range of 5, 4, 3, and 2 mean were calculated with the following equation:

$$\text{deviation value} = Q\sqrt{\frac{AS(E)}{t}}. \quad (1)$$

where t is the number of individual responses used to calculate each average.

The values of Q are given from the table of critical values for the Duncan multiple comparisons test ($p \leq 0.05$). When the mean difference is greater than the deviation value, the difference between these two means is, therefore, significant. The significant differences between averages were presented using letters.

The results show that the tasters significantly preferred the DJ NaCl 0.625% to all the other samples and liked DJ NaCl 0.3125%, DJ NaCl 1.25%, and DJ NaCl 2.5% in comparison with DJW in the same way (Table 8).

4. Conclusion

The present work aims to study the influence of desamerization of the mesocarpe on the chemical composition and the sensorial quality of the bitter orange jam. Salt, heat, and water are considered as driving elements in the operation of

desamerization process. The results of the physicochemical parameters and the chemical composition of the jams show that desamerization decreases acidity, sugars, protein, and bioactive compound levels (vitamin C, carotenoids, and polyphenols) and increases the ash rate. The desamerized jams presented a low content of antioxidants compared to the bitter jam and fresh product. But, regardless of the degradation of total phenolics and some antioxidants, the present results suggest that desamerized jam made from the fruit Bigarade still remain good sources of bioactive compounds with antioxidant potential in the diet.

Regarding the sensory analysis of the jams, the results show that the DJW presents the same bitterness as the bitter jam and that the salt significantly reduces the bitterness of the jams. The hedonic analysis shows that the tasters preferred significantly the DJ NaCl 0.625% to all the other samples.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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Research Article

Sea Buckthorn (*Hippophae rhamnoides* L.) and Quince (*Cydonia oblonga* L.) Juices and Their By-Products as Ingredients Showing Antimicrobial and Antioxidant Properties for Chewing Candy: Nutraceutical Formulations

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Sustainable and environmentally friendly approaches to the production of health foods have become very popular. The concept of this study was to develop chewing candy (CC)—nutraceutical formulations based on sea buckthorn (*Hippophae rhamnoides* L.) and quince (*Cydonia oblonga* L.) juice and juice by-products (BuJ, QuJ, BuBP, and QuBP, resp.), as ingredients showing antimicrobial properties against *Klebsiella pneumoniae*, *Salmonella enterica*, *Pseudomonas aeruginosa*, *Acinetobacter baumannii*, *Proteus mirabilis*, methicillin-resistant *Staphylococcus aureus* (MRSA), *Enterococcus faecalis*, *E. faecium*, and *Bacillus cereus*. Two texture-forming agents (agar and gelatin) were tested for CC formulation. BuJ, QuJ, BuBP, and QuBP showed antimicrobial activity against all the pathogens tested, and the largest inhibition zones against *Bacillus* and *Proteus mirabilis* were observed for BuJ and QuJ, respectively. Agar and/or gelatin selection has a significant influence on CC texture ($p = 0.0001$), and interactions of agar and/or gelatin selection × juice or juice by-products and sea buckthorn or quince × juice or juice by-products were also significant ($p = 0.0001$). The best acceptability was shown for CC prepared with agar and BuBP (131.7) and with gelatin and QuJ (132.0). The addition of BuJ, QuJ, BuBP, and QuBP increases the antioxidant activity of CC by five times. Finally, not just juice, but also juice by-products, have great potential as desirable antimicrobial ingredients for the food industry.

1. Introduction

Many efforts have been made to cope with the agroindustrial waste produced by food manufactories. Nowadays, many by-products (BP) are transformed into useful ingredients, and some of these ingredients have been commercialized and widely used in food, nutraceutical, cosmetic, and other industries [1]. However, vegetables and some fruits yield between 25% and 30% of nonedible products, and the BP of fruits and vegetables are made up of skins and seeds of different shapes and sizes that normally have no further usage and are commonly wasted or discarded [2]. Therefore, development of sustainable technologies is still relevant because many BP have until now not been recovered [3]. The very high content of BP after juice production remains. As juice BP have many biologically active compounds which are healthy for consumers, they can be used for preparation of higher value foods and/or supplements. Higher value food should be attractive for consumers, and juice BP can be incorporated into chewing candy (CC) formulations. In some European and Asian countries, sea buckthorn (Bu) and quince (Qu) fruits are very popular as they have a high content of desirable compounds and show good sensory characteristics [4]. Bu is a good source of mineral acids, vitamins, carbohydrates, amino acids, and natural antioxidants, including phenolics, flavonoids, ascorbic acid,

Table 1: Chewing candy formulas.

	GC	Sugar, g	Water, mL	Citric acid, g	A, g	G, g	Bu J, mL	Bu BP, g	Qu J, mL	Qu BP, g
	AC	30	20	0.9	10	—	—	—	—	—
	A + Bu J		—	—			20	—	—	—
A	A + Bu BP		20				—	20	—	—
	A + Qu J		—				—	—	20	—
	A + Qu BP		20				—	—	—	20
	GC		20	0.9	—	10	—	—	—	—
	G + Bu J		—	—			20	—	—	—
G	G + Bu BP		20				—	20	—	—
	G + Qu J		—				—	—	20	—
	G + Qu BP		20				—	—	—	20

GC: gummy candies; A: agar; C: control; G: gelatin; J: juice; BP: juice by-products; Bu: sea buckthorn; Qu: quince.

tocopherols, fatty acids, carotenoids, and organic acids, which possess various biological activities [5]. Qu is described as a fruit with high economic and medicinal potential [6]. Also, Bu and Qu are good candidates as antimicrobial agents. The main components responsible for the antimicrobial properties of plants are polyphenolic compounds, and it has been shown that the oils from different parts of Bu inhibit *Staphylococcus aureus*, *Escherichia coli*, *Bacillus subtilis*, and *B. coagulans* [7]. However, literature about the antimicrobial activity of Bu and Qu BP is scarce. Anyway, BP of Bu and Qu juice are not used efficiently enough and taking into account that the botanical dietary supplement segment is anticipated to witness considerable growth over the forecast period on account of the increasing popularity of nutraceuticals with natural ingredients, the development of new products has become very relevant [8]. Nutraceuticals and/or supplements are available in numerous forms including dry, solid or liquid extracts, tablets, capsules, powders, and so on; however, consumers prefer to choose food in traditional form, and incorporation of high value botanical ingredients in CC formulations has become very attractive.

The concept of this study was to develop chewing candy (CC)— nutraceutical formulations based on sea buckthorn (*Hippophae rhamnoides* L.) and quince (*Cydonia oblonga* L.) juice and juice by-products (BuJ, QuJ, BuBP, and QuBP, resp.), as ingredients showing antimicrobial properties against *Klebsiella pneumoniae*, *Salmonella enterica*, *Pseudomonas aeruginosa*, *Acinetobacter baumannii*, *Proteus mirabilis*, methicillin-resistant *Staphylococcus aureus* (MRSA), *Enterococcus faecalis*, *E. faecium*, and *Bacillus cereus*. Two texture-forming agents (agar and gelatin) were tested for CC formulation.

2. Materials and Methods

2.1. Materials Used for Chewing Candy Preparation. Bu (*Hippophae rhamnoides* L., variety "Щербинка") and Qu (*Cydonia oblonga* L., variety "Rondo") were purchased from local farms (from Klaipeda and Alytus districts, resp., Lithuania). Bu and Qu juice (BuJ and QuJ, resp.) prepared from fresh fruits and juice by-products (BuBP and QuBP, resp.), the dry part after juice preparation, were prepared at laboratory scale by using a "KENWOOD JE850" juicer at the Lithuanian University of Health Sciences (Kaunas, Lithuania). Juice and by-products were used fresh without pasteurisation and drying, as well as by-products were used with seeds. Agar powder (*Gelidium sesquipedale* algae, Rapunzel, Germany) was used as a polymer with mucoadhesive properties for CC formation. Gelatin (Klingai, Kaunas, Lithuania) was also tested. Sugar was obtained from Nordic Sugar (Kedainiai, Lithuania), xylitol from Natur Hurtig (Nuremberg, Germany), and citric acid from Sanitex (Kaunas, Lithuania).

2.2. Evaluation of the Acidity Parameters of Sea Buckthorn and Quince Products. The pH values of BuJ, QuJ, BuBP, and QuBP were measured and recorded using a pH electrode (PP-15; Sartorius, Goettingen, Germany). The total titratable acidity (TTA) was determined for a 10 g sample of BP and/or juice homogenized with 90 mL of distilled water and expressed as millilitres of 0.1 mol·L^{-1}·NaOH required to achieve a pH of 8.2.

2.3. Chewing Candy Formulas. The control CC formula consisted of sugar (30 g), water (20 g), citric acid (0.90 g), agar (10.0 g), and/or gelatin (10.0 g) (Table 1). Chewing candies were prepared by addition to the main formula of Bu (*Rhamnus*, family *Rhamnaceae*) and/or Qu (*C. oblonga*, family *Rosaceae*) juice or BP. It should be mentioned that candies with juice and/or BP were prepared without the addition of citric acid. For preparation of candies with agar, firstly, agar powder was soaked in water for 30 min and then melted by heating for 5 min; then, sugar and/or xylitol was added and dissolved under boiling conditions. The mixture obtained was further heated to 103 ± 2°C under stirring. Citric acid was incorporated into the control CC mass at the end of the process. For preparation of CC with gelatin, firstly, gelatin powder was soaked in water for 30 min, and then melted at 80 ± 2°C; then, sugar and/or xylitol and citric acid were added and dissolved. After mixing, the mass obtained (both that prepared with agar and prepared with gelatin) was poured into a mould, and CC were dried at 22–24°C for 24 h to get a gel-hard form.

2.4. Evaluation of Antimicrobial Activity of Sea Buckthorn and Quince Juices and Their By-Products. An agar well diffusion assay was used for testing the antimicrobial activity of BuJ,

QuJ, BuBP, and QuBP. For this purpose, a 0.5 McFarland unit density suspension of each pathogenic bacteria strain was inoculated onto the surface of cooled Mueller–Hinton agar (Oxoid, UK) using sterile cotton swabs. Wells 6 mm in diameter were punched in the agar and filled separately with BuJ, QuJ, BuBP, and/or QuBP. Juice and juice production by-products were used fresh without pasteurisation and drying. The antimicrobial activity against tested bacteria was determined by measuring the diameter of inhibition zones (mm). The experiments were repeated three times, and the average of inhibition zones was calculated.

2.5. Determination of Total Phenolic Compound (TPC) Content, Antioxidant Activity, and Colour Characteristics of Chewing Candies. The TPC in juice and BP samples was determined by a spectrophotometric method, as reported elsewhere [9]. The absorbance of samples was measured at 765 nm using a J.P. Selecta S.A. V-1100D spectrophotometer (Barcelona, Spain). Antioxidant activity of the samples was evaluated according to the method reported by Zhu et al. [10]. Colour characteristics were evaluated using a CIE $L^*a^*b^*$ system (Croma Meter CR-400, Konica Minolta, Japan) [11].

2.6. Evaluation of Chewing Candy Texture. The hardness of CC was evaluated by using a TA.XT2 texture analyser (Stable Micro Systems Ltd., Godalming, UK) (compression force 0.5 N, test speed 0.5 mm·s^{-1}, posttest speed 2 mm·s^{-1}, distance 6 mm).

2.7. Evaluation of Overall Acceptability of Chewing Candies. Overall acceptability of CC was evaluated according to ISO method 8586-1 [12] for preliminary sensory acceptability, using a 150 mm hedonic line scale ranging from 150 (extremely like) to 0 (extremely dislike).

2.8. Statistical Analysis. The results were expressed as the mean value of at least three measurements ± standard deviation. In order to evaluate the effects of the different formula components on CC quality parameters, data were analysed by analysis of variance (IBM SPSS Statistics, ver. 22). Results were recognized as statistically significant at $p \leq 0.05$.

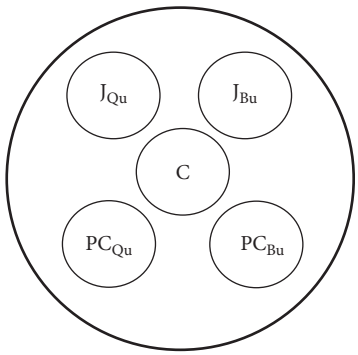
3. Results and Discussion

3.1. Antimicrobial Activity of Sea Buckthorn and Quince Juices and Their By-Products. The antimicrobial activity of BuJ, QuJ, BuBP, and QuBP is shown in Table 2. BuJ, QuJ, BuBP, and QuBP showed antimicrobial activity against all the pathogenic bacteria tested; comparing the antimicrobial activity of juice, the largest inhibition zones for BuJ and QuJ were observed against *B. cereus* (29.0 ± 0.5 and 30.0 ± 0.7 mm, resp.) and *Proteus mirabilis* (25.0 ± 0.6 and 26.0 ± 0.4 mm, resp.). In most cases, BuBP and QuBP showed weaker antimicrobial activity than the juice (on average, BuBP inhibition zones were 0 to 7 mm smaller, and QuBP zones were 0 to 5 mm smaller). The antimicrobial activity of BuBP ranged from 8.0 ± 0.4 to 25.0 ± 0.5 mm (for *K. pneumoniae* and *B. cereus* inhibition, resp.) and of QuBP from 8.0 ± 0.4 to 26.0 ± 0.5 mm (for *S. enterica* and *B. cereus* inhibition, resp.). The inhibition zones for juice and BP were found to be the same for *S. enterica* (11.0 and 8.0 mm for Bu and Qu products, resp.). Antibiotic resistance is one of the biggest threats to global health, food security, and development today [13], and natural botanical sources showing antimicrobial activity against antibiotic-resistant pathogens can be very important. It was published that n-hexane and chloroform extracts of Bu berries and n-hexane extract of Bu leaves show significant ($p < 0.05$) antibacterial activity comparable with vancomycin, and it was concluded that extracts of Bu berries and leaves have antibacterial activity against MRSA [14]. According to Fattouch et al. [15], polyphenolic extract of Qu peel shows antimicrobial activity against Gram-positive (*S. aureus*) and Gram-negative bacteria (*E. coli* and *P. aeruginosa*) and yeast (*Candida albicans*). The MIC of ethanolic extract of Qu peel ranges between 0.1 and 5.0 mg·polyphenol·mL^{-1} [16]. Both Bu and Qu have a high phenolic compound content, and recent studies have shown that phenolic compounds mostly modulate the composition of gut microbial communities through the inhibition of pathogenic bacteria and stimulation of beneficial bacteria; in the latter, phenolic compounds may exert a prebiotic function and increase the population of beneficial bacteria, including probiotics, suggesting a mutual relationship between phenolic compounds and probiotics [17–19]. The main components responsible for the antimicrobial properties of plants are polyphenolic compounds; they exhibit anti-inflammatory activity *in vitro* and *in vivo*, and their mechanism of action is the inhibition of enzymes (phospholipase oxygenase), that is, by binding with hydrosulphide groups and inactivating bacterial proteins [20]. According to our results, not just the juice, but also BuBP and QuBP have great potential as antimicrobial ingredients for the food industry.

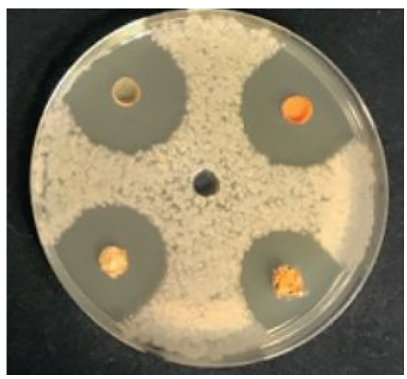
3.2. Acidity Parameters and Colour Coordinates of Sea Buckthorn and Quince Juices and Their By-Products. Acidity parameters and colour coordinates of BuJ, QuJ, BuBP, and QuBP are shown in Figures 1(a) and 1(b). Comparing juice and BP, lower pH values were obtained for the juice (compared to BP, BuJ pH was lower by 24.0% and QuJ pH was lower by 10.4%) (Figure 1(a)). Opposite to pH values, BuJ TTA was two times lower than that of BP; however, QuJ TTA was 1.9 times higher than that of BP. BuBP and QuBP showed higher lightness (L^*) values than juice; L^* coordinates ranged from 29.82 ± 0.96 to 35.84 ± 1.27 for BuJ and BuBP, respectively, and from 36.23 ± 1.63 to 48.53 ± 1.08 for QuJ and QuBP, respectively (Figure 1(b)). Different tendencies of Bu and Qu redness (a^*) were observed when comparing juice and BP. Coordinates of a^* for BuJ were 49.9% lower than those for BP; however, a^* coordinates for QuJ were 30.7% higher than those for BP. In all cases, higher yellowness (b^*) coordinates were found for BP than for juice (BuBP b^* coordinates 155.2% higher, QuBP 56.2% higher).

Table 2: Antimicrobial activity of buckthorn and quince juices and their 363 by-products.

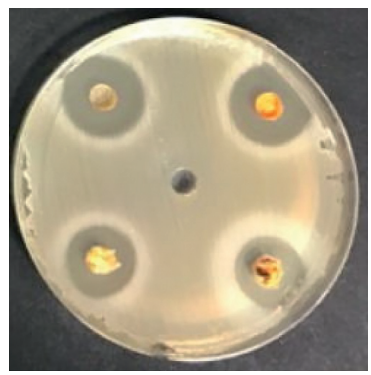
Microorganisms	J		BP	
	Bu	Qu	Bu	Qu
	Inhibition zones, mm			
Klebsiella pneumoniae	14.0 ± 0.3^{d}	13.0 ± 0.3^{c}	8.0 ± 0.4^{a}	10.0 ± 0.3^{b}
Salmonella enterica	11.0 ± 0.2^{b}	8.0 ± 0.2^{a}	11.0 ± 0.3^{b}	8.0 ± 0.4^{a}
Pseudomonas aeruginosa	23.0 ± 0.4^{b}	23.0 ± 0.3^{b}	20.0 ± 0.2^{a}	20.0 ± 0.2^{a}
Acinetobacter baumannii	20.0 ± 0.5^{c}	22.0 ± 0.3^{d}	16.0 ± 0.1^{a}	19.0 ± 0.3^{b}
Proteus mirabilis	25.0 ± 0.6^{c}	$26.0 \pm 0.4^{c,d}$	20.0 ± 0.2^{a}	23.0 ± 0.4^{b}
MRSA	22.0 ± 0.3^{c}	22.0 ± 0.2^{c}	17.0 ± 0.2^{a}	18.0 ± 0.2^{b}
Enterococcus faecalis	25.0 ± 0.4^{c}	23.0 ± 0.3^{b}	18.0 ± 0.3^{a}	18.0 ± 0.2^{a}
Enterococcus faecium	20.0 ± 0.3^{b}	22.0 ± 0.3^{c}	18.0 ± 0.2^{a}	22.0 ± 0.4^{c}
Bacillus cereus	29.0 ± 0.5^{c}	30.0 ± 0.7^{d}	25.0 ± 0.5^{a}	$26.0 \pm 0.5^{a,b}$



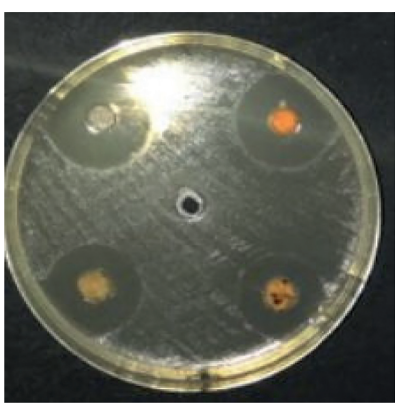
Experimental design



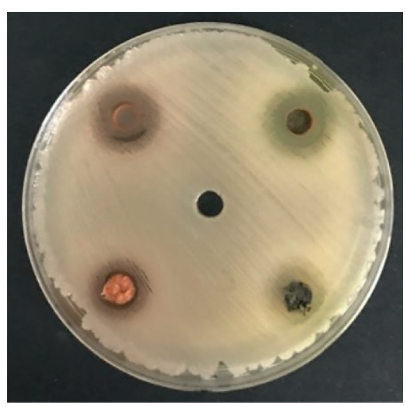
Bacillus cereus



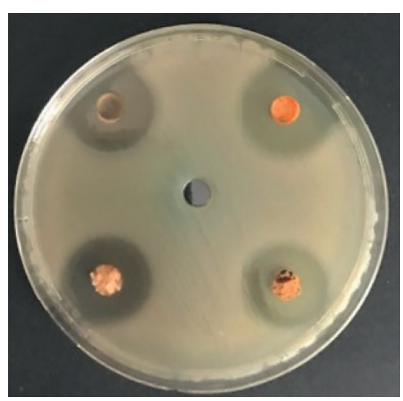
MRSA



Proteus mirabilis



Klebsiella pneumoniae



Pseudomonas aeruginosa

J: juice; BP: juice by-products; Bu: sea buckthorn; Qu: quince; C: control; MRSA: methicillin-resistant *Staphylococcus aureus*. Data expressed as means ($n = 3$) ± standard deviation (SD). $^{a-d}$Mean values with different letters are significantly different when $p \leq 0.05$.

The main components of Bu identified are ascorbic acid, carotenoids, and various phenolics, including proanthocyanidins, gallic acid, ursolic acid, caffeic acid, coumaric acid, ferulic acid, catechin and epicatechin derivatives, quercetin, kaempferol, and isorhamnetin glycoside derivatives [21]. However, colour is influenced by carotenoids, and the main carotenoids of Bu are α-, β-, and δ-carotene and lycopene [22]. It was published that the edible parts of Qu can have 16.8 mg·100·g^{-1} ascorbic acid; this is the main compound which influences the sour taste of these fruits [6].

3.3. Texture, Colour Characteristics, and Overall Acceptability of Chewing Candies. CC texture, colour characteristics, and overall acceptability are shown in Table 3. Comparing the CC group prepared with agar (A), the hardest texture was obtained for the CC prepared with BuBP (hardness of A + BuBP samples was 1.00 ± 0.01 mJ). Compared with control CC (AC), CC prepared with the addition of juice and/or BP were harder in all cases (from 35% to 400% for CC prepared with BuJ and BuBP, resp.). Comparing the CC group prepared with gelatin (G), the hardest texture was obtained for the CC with BuJ (hardness of G + BuJ samples was 3.13 ± 0.12 mJ); in contrast, CC prepared with the addition of BuBP had the softest texture (37.1% softer). In all cases, CC prepared with juice were harder than the candies prepared with the addition of BP. Selection of A and/or G

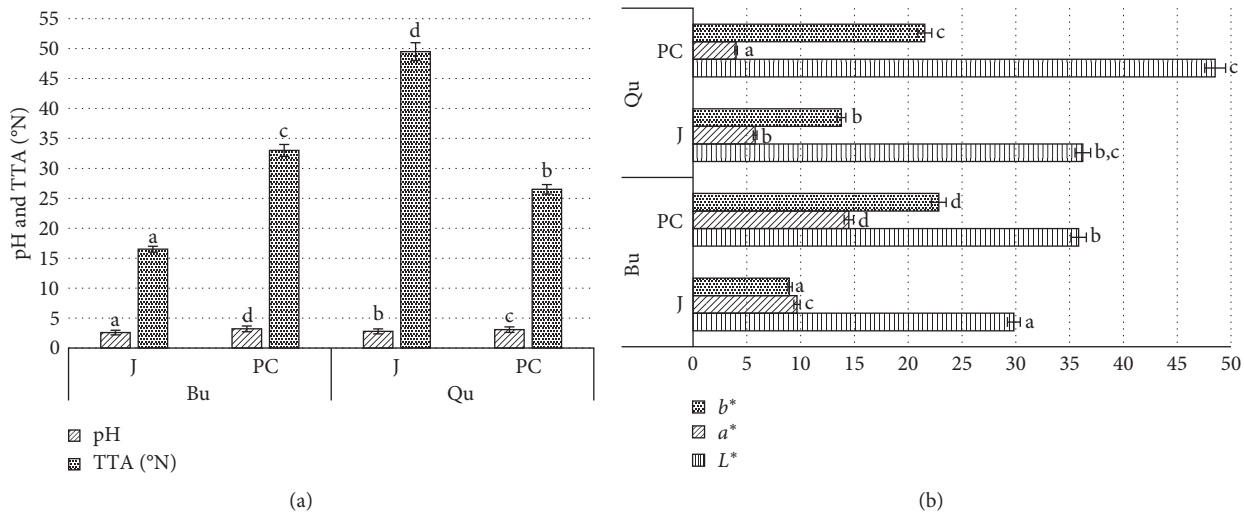


Figure 1: Acidity parameters (a) and colour coordinates (b) of sea buckthorn (Bu) and quince (Qu) juice (J) and juice by-products (PC) (BuJ, QuJ, BuBP, and QuBP, resp.).

had a significant influence on CC texture ($p = 0.0001$), and interactions of A and/or G selection × juice or BP and Bu or Qu × juice or BP were also significant ($p = 0.0001$).

In all cases, higher lightness (L^*) coordinates were obtained for CC prepared with A (on average 48.3% higher compared to CC prepared with G). Most of the factors analysed had a significant influence on CC L^* coordinates (except A and/or G selection). Comparing control CC prepared with A and G, no significant changes were found for redness (a^*) coordinates; however, comparing a^* coordinates of CC prepared with the addition of Bu and Qu products (juice and/or BP), a different influence of A and/or G selection on a^* coordinates was established. Samples prepared with BuBP and A had 60.5% higher redness than samples prepared with G; in contrast, CC prepared with G and QuJ and BP had 143.6% and 35.4% higher redness, respectively, than samples prepared with agar. Comparing yellowness (b^*), CC prepared with G had b^* coordinates 44.6% lower than those of the control CC. However, b^* coordinates of CC were more influenced by the addition of Bu and Qu products (juice and/or BP), and the highest b^* was obtained for the CC prepared with G and BuJ (27.44 ± 2.15). Bu or Qu selection ($p = 0.0001$) and juice or BP selection ($p = 0.0001$) had a significant influence on CC a^* and b^* coordinates, and interactions of A and/or G × Bu or Qu, A and/or G × juice or BP, and Bu or Qu × juice or BP were also significant.

No significant differences were obtained for overall acceptability between the control CC samples prepared with A and G; however, most of the CC prepared with the addition of Bu and Qu products (juice and/or BP) were more acceptable than controls. The best acceptability was obtained for CC prepared with A and BuBP (131.7 ± 10.7) and CC prepared with G and QuJ (132.0 ± 7.8). CC prepared with A and BuBP was also hardest; however, no correlation was found between hardness and overall acceptability of CC. A significant influence of the interaction of analysed factors A and/or G selection × juice or BP on overall acceptability was established ($p = 0.001$).

Pressing of juice produces large amounts of pomace, which are currently discarded as waste or utilized rather inefficiently; therefore, considerable amounts of nutrients are lost annually [23]. Finally, development of new products in which the whole BP can be used is very important; it leads to reduced production costs because extracts should not be prepared. Further, in our experiment, the best acceptability was found for CC formulas improved by changing saccharose to xylitol (1 : 1). In comparing the parameters of CC samples with saccharose and xylitol, no significant changes were observed, except in overall acceptability which was significantly higher for the CC with xylitol.

3.4. Total Phenolic Compound Content and Antioxidant Activity of Chewing Candies. The TPC content and antioxidant activity of the CC are presented in Table 4. Comparing CC groups prepared with A and G, in all cases, control CC samples had a lower TPC content (in the CC group prepared with A, from 132.7% to 312.5% lower, for CC with BuBP and QuJ, resp.; in the group prepared with G, from 44.5% to 160.9% lower, for CC with QuJ and BuBP, resp.) than CC prepared with the addition of Bu and Qu products (juice and/or BP). Also, comparing CC control groups, a 45.0% higher TPC content was obtained for the CC group prepared with G; however, no significant differences in antioxidant activity of control CC groups were established. In all cases (in both CC groups prepared with A and with G), stronger antioxidant activity was established for the CC prepared with Qu and Bu products (juice and/or BP): in the CC group prepared with A, from 130.7% to 563.7% higher (in CC prepared with the addition of BuJ and QuBP, resp.), and in the group prepared with G, from 95.2% to 325.8% higher (in CC prepared with the addition of BuJ and QuBP, resp.). A significant influence of the parameters analysed (A or G selection; Bu or Qu; juice or BP) on TPC content in CC ($p = 0.0001$) and their antioxidant activity ($p = 0.0004$, $p = 0.0001$ and $p = 0.0001$, resp.) was observed. Interactions of Bu or Qu × juice or BP were also significant for the antioxidant properties of CC

Table 3: Hardness, colour coordinates, and overall acceptability of gummy candies.

GC		Texture, mJ	Colour coordinates			Overall acceptability
			L^*	a^*	b^*	
A	AC	0.20 ± 0.01^{a}	46.05 ± 4.32^{d}	1.57 ± 0.28^{c}	13.63 ± 1.44^{e}	85.3 ± 4.2^{a}
	A + Bu J	0.27 ± 0.05^{a}	28.42 ± 4.55^{a}	3.72 ± 0.21^{e}	13.30 ± 2.18^{e}	114.7 ± 9.5^{d}
	A + Bu BP	1.00 ± 0.01^{d}	27.27 ± 1.49^{a}	2.38 ± 0.23^{d}	13.20 ± 1.35^{e}	131.7 ± 10.7^{f}
	A + Qu J	0.43 ± 0.06^{b}	31.39 ± 1.98^{b}	0.94 ± 0.16^{a}	10.88 ± 1.05^{c}	107.0 ± 12.1^{d}
	A + Qu BP	0.80 ± 0.01^{c}	27.40 ± 0.81^{a}	0.96 ± 0.06^{a}	10.08 ± 0.39^{b}	124.0 ± 10.4^{e}
	A + Bu BP + Xy	1.00 ± 0.01^{d}	28.30 ± 1.23^{a}	2.34 ± 0.17^{d}	13.04 ± 1.14^{e}	147.7 ± 2.3^{g}
G	GC	2.07 ± 0.15^{e}	48.45 ± 1.46^{e}	1.56 ± 0.32^{c}	7.55 ± 1.52^{a}	90.4 ± 5.1^{b}
	G + Bu J	3.13 ± 0.12^{h}	49.44 ± 0.16^{e}	3.93 ± 0.39^{e}	27.44 ± 2.15^{f}	108.7 ± 22.9^{d}
	G + Bu BP	1.97 ± 0.25^{e}	34.60 ± 0.88^{c}	0.94 ± 0.11^{a}	12.49 ± 0.21^{d}	$99.3 \pm 17.6^{b,c}$
	G + Qu J	2.70 ± 0.11^{g}	58.81 ± 4.53^{f}	2.29 ± 0.17^{d}	12.74 ± 1.04^{d}	132.0 ± 7.8^{f}
	G + Qu BP	2.53 ± 0.01^{f}	46.60 ± 0.96^{d}	1.30 ± 0.14^{b}	7.54 ± 0.32^{a}	96.3 ± 6.7^{b}
	G + Qu J + Xy	2.67 ± 0.09^{f}	58.62 ± 3.21^{f}	2.26 ± 0.12^{d}	12.68 ± 0.93^{d}	142.0 ± 3.6^{g}

Influence of different texture-forming agents (A or G), different plants (Bu or Qu), and J or BP C of plants on gummy candy parameters

Source	*Dependent variable*		*Mean square*	*F*	*p*
A or G	Texture		26.520	2501.853	0.0001
	Colour coordinates	L^*	1433.441	252.122	0.0001
		a^*	0.043	0.837	0.371
		b^*	1.222	0.708	0.410
	Overall acceptability		199.180	1.217	0.283
Bu or Qu	Texture		0.003	0.287	0.598
	Colour coordinates	L^*	253.110	44.519	0.0001
		a^*	11.261	216.690	0.0001
		b^*	216.300	125.212	0.0001
	Overall acceptability		21.094	0.129	0.723
J or C	Texture		0.020	1.871	0.186
	Colour coordinates	L^*	425.884	74.884	0.0001
		a^*	10.534	202.689	0.0001
		b^*	158.672	91.852	0.0001
	Overall acceptability		70.384	0.430	0.519
A or G × Bu or Qu	Texture		0.011	1.022	0.324
	Colour coordinates	L^*	146.718	25.806	0.0001
		a^*	3.197	61.524	0.0001
		b^*	77.508	44.868	0.0001
	Overall acceptability		550.084	3.362	0.082
A or G × J or C	Texture		2.214	208.900	0.0001
	Colour coordinates	L^*	205.686	36.177	0.0001
		a^*	2.653	51.055	0.0001
		b^*	153.167	88.665	0.0001
	Overall acceptability		2503.084	15.297	0.001
Bu or Qu × J or C	Texture		0.149	14.041	0.001
	Colour coordinates	L^*	1.109	0.195	0.663
		a^*	4.234	81.462	0.0001
		b^*	34.058	19.715	0.0001
	Overall acceptability		313.204	1.914	0.182
A or G × Bu or Qu × J or C	Texture		0.683	64.475	0.0001
	Colour coordinates	L^*	5.881	1.034	0.321
		a^*	0.154	2.956	0.101
		b^*	33.773	19.550	0.0001
	Overall acceptability		313.204	1.914	0.182

GC: gummy candies; A: agar; C: control; G: gelatin; J: juice; BP: juice by-products; Bu: sea buckthorn; Qu: quince; Xy: xylitol. L^*: lightness; a^*: redness ($-a^*$: greenness); b^*: yellowness ($-b^*$: blueness). Data are represented as means ($n = 3$) ± SD. $^{a-g}$Mean values with different letters are significantly different when $p \leq 0.05$.

($p = 0.0001$), and interactions of A or G selection × juice or BP and Bu or Qu × juice BP selection were significant for TPC content in CC. The main phytochemicals present in Qu (quercetin, 5-O-caffeoylquinic acid, rutin, β-carotene, lycopene, β-cryptoxanthin, lutein, plus zeaxanthin) have received attention due to their high antioxidant potential and capability of preventing and treating pathologies [24]. Total antioxidant activity in Bu is significantly associated with total phenolics, isorhamnetin-3-rutinoside, and isorhamnetin-3-glucoside [25]. Therefore, strong antioxidant activity of Bu has been reported by several authors [26]. Finally, BuJ, QuJ, BuBP, and QuBP increased the TPC content and antioxidant activity of the CC, and

Table 4: Total phenolic compounds (TPC) content and antioxidant activity of gummy candies.

GC		TPC content, RE mg·100·g^{-1}	Antioxidant activity, %
A	AC	47.1 ± 3.1^{a}	12.4 ± 1.9^{a}
	A + Bu J	153.5 ± 6.4^{f}	28.6 ± 1.7^{c}
	A + Bu BP	194.3 ± 7.2^{i}	35.2 ± 2.8^{f}
	A + Qu J	109.6 ± 4.3^{d}	41.8 ± 3.4^{g}
	A + Qu BP	162.1 ± 4.7^{g}	57.5 ± 3.1^{i}
	A + Bu BP + Xy	186.1 ± 4.5^{i}	36.4 ± 1.8^{f}
G	GC	68.3 ± 3.4^{b}	12.4 ± 0.7^{a}
	G + Bu J	149.4 ± 5.6^{e}	24.2 ± 1.6^{b}
	G + Bu BP	178.2 ± 7.0^{h}	31.6 ± 2.3^{d}
	G + Qu J	98.7 ± 4.3^{c}	37.3 ± 2.9^{f}
	G + Qu BP	143.5 ± 6.2^{e}	52.8 ± 3.4^{h}
	G + Qu J + Xy	100.3 ± 3.9^{c}	34.1 ± 1.6^{e}

Influence of different texture-forming agents (A or G), different plants (Bu or Qu), and J or BP of plants on gummy candy parameters

Source	*Dependent variable*	*Mean square*	*F*	*p*
A or G	TPC content	45.679	1.565	0.225
	Antioxidant activity	65.473	10.259	0.004
Bu or Qu	TPC content	9780.844	335.144	0.0001
	Antioxidant activity	1827.015	286.276	0.0001
J or C	TPC content	10445.854	357.931	0.0001
	Antioxidant activity	766.140	120.047	0.0001
A or G × Bu or Qu	TPC content	32.434	1.111	0.304
	Antioxidant activity	0.540	0.085	0.774
A or G × J or C	TPC content	145.534	4.987	0.037
	Antioxidant activity	0.135	0.021	0.886
Bu or Qu × J or C	TPC content	287.734	9.859	0.005
	Antioxidant activity	110.940	17.383	0.0001
A or G × Bu or Qu × J or C	TPC content	6.934	0.238	0.631
	Antioxidant activity	0.375	0.059	0.811

GC: gummy candies; A: agar; C: control; G: gelatin; J: juice; BP: juice by-products; Bu: sea buckthorn; Qu: quince; TPC: total phenolic compounds; RE: rutin equivalents. L^*: lightness; a^*: redness ($-a^*$: greenness); b^*: yellowness ($-b^*$: blueness). Data are represented as means ($n = 3$) ± SD. $^{a-i}$Mean values with different letters are significantly different when $p \leq 0.05$.

incorporation of these ingredients into the main formula leads to the preparation of products with better functionality.

4. Conclusions

BuJ, QuJ, BuBP, and QuBP showed antimicrobial activity against all the pathogenic bacteria tested; according to the results obtained, not just the juice, but also BP have great potential as desirable antimicrobial ingredients for the food industry. In all cases, BuJ, QuJ, BuBP, and QuBP increase the TPC content and antioxidant activity of the CC. However, A and/or G selection ($p = 0.0001$) and interactions of A and/or G selection × juice or BP and Bu or Qu × juice or BP ($p = 0.0001$) have a significant influence on CC texture. Combination of A and BuBP and of G and QuJ leads to the preparation of products with a high degree of overall acceptability containing desirable antimicrobials.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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Research Article

A Combined System of Ground Well and Composted Olive Cake for Hot Water Production at Olive Mills

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Building a system that consists of a combination of geothermal component (water well (pit)) and heat recovery from aerobic biological fermentation of olive cake for hot water production at an olive mill is examined in this work. Hot water is essential for mill operation and constitutes a main operational cost, and in many countries, including Jordan, it is normally produced using diesel fuel. In this process, treated and untreated olive cake was characterized. Results show that olive cake is rich in crude fiber and NFE, contains moderate amounts of crude protein and fat, and a good amount of ash. The as-received moisture content ranged from 33.3 to 35.6%, while water activity was between 0.93 and 0.96. The total counts, thermophilic bacteria, and the total mold count of fermentation ranged, respectively, from 2.1×10^8 to 2.4×10^8, 1.7×10^4 to 1.9×10^4, and 1.5×10^2 to 1.7×10^2. The temperature results showed that the well and the covered tank led to a rise in water temperature before entering the boiler in the range of 7 to 13°C. The system effected significant raises in water temperature entering the boiler ranging from 19°C up to 25°C, which holds a promising potential for the system to satisfy much of the mills needs at this range of temperature before entering the boiler provided a large enough pile (pile scale up) is used to handle larger flow rates. The exhausted cake may well be utilized as a soil organic fertilizer.

1. Introduction

In most Mediterranean countries including Jordan, olive tree culture goes back to many centuries, and olive trees in the region enjoy a special cultural and economic significance. Due to various social and economic transformations, the region has witnessed substantial expansion in olive trees in the last several decades. In fact, in Jordan alone, there are more than 110 olive mills that process olive fruits for olive oil production. The latter enjoys several proven health advantages [1, 2].

In addition to olive oil, two other materials are produced by olive processing mills, namely, the solid residue and wastewater as by-products, both of which constitute a serious environmental risk if not adequately disposed of or properly utilized [3]. Thus, numerous researches have investigated the olive oil industry by-products and wastes. In particular, the solid waste has attracted attention for its potential as an energy source [4, 5], oil source, and animal feed [6, 7], among other purposes. The wastewater by-product has also been investigated where several researches attempted to improve the quality of wastewater for better utilization in many areas such as crop irrigation and crop fertilization [8, 9].

Recently, a strong correlation was observed between olive oil production and environmental pollution, and researchers became concerned with the basic issue of the wastes produced after extracting oil from the fruits. Aqueous sludge, which makes about 45% to 54% of the total waste, is treated by using different methods such as chemical, biochemical, and electrochemical treatments, supercritical extraction, and separation processes based on membrane technology [10–12]. Olive cake, which is also considered a major solid pollutant, constitutes more than 80% of the

consumed olives and depends on olive varieties and the extraction process. Generally, olive cake is utilized as a fuel due to its relatively high energy content [5] and as a raw material for soap making due to its high-quality oil content of about 5% to 8% [13].

However, these application areas require drying of the olive cake from a moisture content of 20% to 45% to approximately 5% to 6%; this process is regarded as energy intensive [14, 15].

During olive oil production, mills need large amounts of hot water that are normally obtained from boilers that operate on diesel fuel. In Jordan, that imports more than 97% of its energy, the annual energy bill averages about JOD 10,000 per mill (equivalent to US$15,000) and is projected to be doubled with the continuous increase in diesel fuel prices. Mills use large amounts of hot water at about 60 to 70°C for about four to five months, which is the length of the annual pressing season. In addition, some mills have on-site olive oil storage facilities for filling and exporting purposes. These storage facilities also require additional large volumes of hot water at 30 to 40°C for oil processing.

Olive processing yields enormous quantities of solid waste after the separation of the aqueous phase. This residue contains 4% to 9% of olive oil, depending on the extraction system used: continuous or discontinuous pressure [16]. Chemically speaking, this solid waste is a lignocellulosic organic material [17]. In different countries, this by-product is burned for heat generation. However, since the enactment of international regulations limits the emission of CO_2, this practice has been prohibited, thus requiring alternative practices.

The current practice regarding hot water production for the milling process at the mill under consideration, as well as in most other mills, involves transporting water from nearby springs and storing them in bear (uninsulated) metal tanks on the rooftop of the mill and then feeding this water to the diesel-fired boiler. Given that most of the milling season coincides with the cold winter season when ambient temperatures in most regions including the mill under study are very low and even get close to zero during part of the milling season, large sensible heat is needed to raise the water temperature up to 60 to 70°C needed for olive processing. This fact is reflected in substantial diesel fuel requirements that under current price trends present a heavy toll and a major operational expense of the mill. Based on the above, it may be obvious that the energy bill especially that pertains to hot water makes a major operational cost, and any reductions in energy expenses will effect significant savings in the running costs of operating the mill.

In light of the facts that many families depend on olive oil as an important source of living, the tighter competition among oil producers in available markets, and that Jordan imports almost all its energy needs, this study was set out to explore the possibility of building an alternative novel the on-site system for hot water production. The proposed system which was erected on the mill premises consisted of a combination of geothermal component (water well or pit) and heat recovery from the process heat of the biological fermentation of olive cake that is produced in large quantities at the mill in an attempt to reduce/supplement the diesel fuel consumption at the mill. In fact, heat recovery from the aerobic treatment of organic wastes was investigated although in very limited instances [18].

2. Materials and Methods

2.1. Proximate Chemical Analysis. Proximate analysis was carried out according to the procedures outlined by the AOAC [19].

2.2. Determinations of pH. The regular pH meter was used to determine the pH of fermented and unfermented olive cake. Six measurements were taken for each sample, and the final reported reading was the arithmetic average of the readings.

2.3. Water Activity. The regular water activity instrument was used to determine the water activity of fermented and unfermented olive cake. Three determinations (about 25 grams each) were taken for each measurement, and the final reported reading was the arithmetic average of all readings.

2.4. Microorganism Examinations

2.4.1. Sample Preparation (Dilution Preparation). Twenty-five-gram samples were taken from fermented and unfermented olive cake. Each sample was then added to 250 ml of peptone water to a dilution of 1 : 10.

2.4.2. Total Count (TPC) and Total Thermophilic Bacteria (TTB). From the previous solution (Section 2.4.1), 0.1 and 1 ml were put in Petri dishes and plate count agar that was prepared in standard methods relevant to TPC and TTB was poured onto them. After that, the dishes were put in an incubator for 24 to 48 hrs at 37°C. The colony was counted after the growth.

2.4.3. Total Mold Counts. From the previous solution, 0.1 and 1 ml were put in Petri dishes, and potato dextrose agar that was prepared by standard methods was poured on them. After that, the dishes were put in an incubator for 3 to 5 days at 25°C. The colony was counted after the growth.

2.5. The Hot Water Production System. The system in this work utilized a combination of renewable energy sources for hot water production. The sources included geothermal energy by digging a ground pit (well) with a total capacity of 20 m^3 of water. The well (first station) receives water from tankers that bring water from nearby springs. The water is then pumped to the partially buried tank (second station) that is covered with olive cake for merely thermal insulation purposes and utilizing the possible uncontrolled

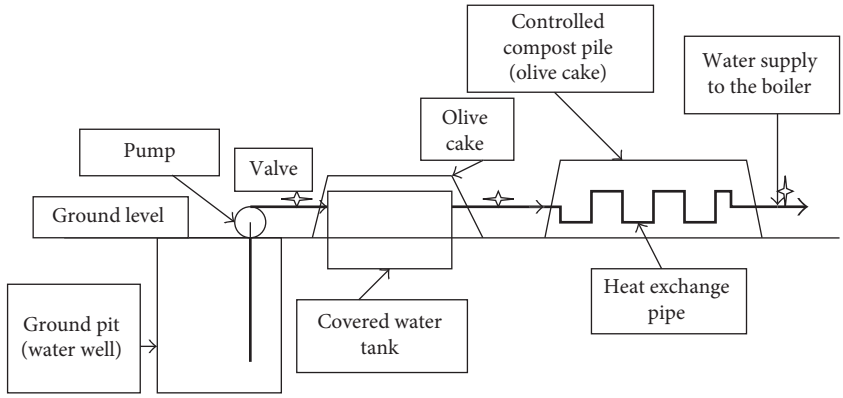

Figure 1: A schematic diagram of the combined system used in this work for hot water production at the olive mill.

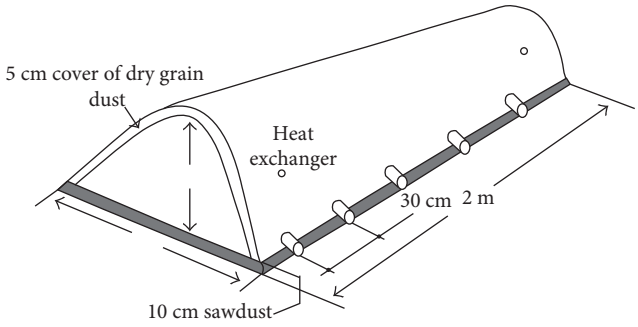

Figure 2: A schematic of the compost pile of olive cake used in this work [20].

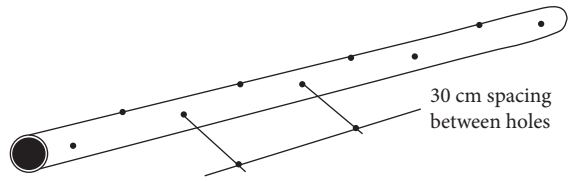

Figure 3: A sketch of the perforated pipes used for aerating the pipe [20].

compost process heat to further heat the water. Water then leaves the tank to a heat exchanger that is completely inserted into a controlled olive cake compost pile (third station) where temperature reaches 60°C to 70°C due to the biological activity. A schematic of the system is shown in Figure 1 [20].

A 100-meter long heat exchanger made of aluminum tubes with an internal diameter of 18 mm was used. The tubes were wound at the Engineering Workshops of Jordan University of Science and Technology to fit within a 13-meter long pile of olive cake. The water flow rate into and out of the heat exchanger at steady-state conditions was measured by a calibrated (graduated) beaker with a stop watch from which the hot water volumetric flow rate was calculated.

As shown in Figure 2, the pile was of the passively aerated type fitted with 12-inch (30 cm) diameter PVC perforated pipes for efficient aeration and air circulation as illustrated in Figure 3. A total of 12 PVC pipes were used. It should be noted that, on one hand, the pile was located in open air next to the mill premises, and on the other hand, the pile management requirements were minimal. Consequently, pile emissions would be substantially diluted, and human exposure to pile emissions would be marginal. As such, no measures of pile emission control were implemented.

A valve was fitted at the exit point of each of the three stations, and numerous measurements of water temperatures were made at these points in addition to the ambient temperature using thermocouples along with a digital multimeter. The water temperature data were averaged for every two weeks (15 days) and reported as a single value. Water temperature measurements were started at the end of January and extended over the following three consecutive months (till the end of April).

2.6. Statistical Analysis. Data are presented as means of three determinations and analyzed using the general linear model procedure with SAS Version 8.2 software package [21]. The LSD analysis was used to compare means. Significant differences were defined at the $p < 0.05$ level.

3. Results and Discussion

3.1. Chemical Analysis. The chemical composition of fermentation periods of olive cake during 90 days of fermentation is shown in Table 1. As may be seen from the results, the unfermented (0 day) and fermented olive cake is rich in crude fiber and NFE and contains moderate amount of crude protein and fat. Also, the results in Table 1 show that olive cake contains a good amount of ash. Table 1 also indicates that the olive oil is rich in the nutrients which helped microorganisms to grow for longer periods of times. That is, these nutrients assist microorganisms to produce heat for longer periods of time, thus enhancing the feasibility of the hot water system. In addition, Table 1 shows the fermentation time for three months. It is shown that no significant differences were found during fermentation. These results indicted that olive cake can be used for long time as a source of fermentation.

Table 1: Chemical composition of olive cake during 90 days of fermentation.*

Fermentation period (day)	Dry matter	% of dry matter				
		Ash	Crude protein	Crude fiber	Ether extract	NFE**
0	65.3 ± 4.4	4.4 ± 0.2	7.2 ± 0.5	43.6 ± 3.3	11.4 ± 0.8	32.3 ± 2.5
30	66.7 ± 4.5	4.6 ± 0.3	7.1 ± 0.4	44.0 ± 3.8	11.3 ± 0.7	32.3 ± 2.7
60	65.9 ± 4.1	4.2 ± 0.2	7.4 ± 0.5	44.5 ± 3.2	11.5 ± 0.8	32.0 ± 2.4
90	65.3 ± 4.4	4.3 ± 0.3	7.1 ± 0.4	45.5 ± 3.9	11.4 ± 0.7	32.7 ± 2.3

*Means ± SD; **nitrogen-free extract.

Table 2: Moisture contents and water activity of olive cake during 90 days of fermentation.

Fermentation period (day)	Moisture contents (%)	Water activity*
0	34.7 ± 2.2	0.95 ± 0.03
30	33.3 ± 2.1	0.93 ± 0.04
60	34.1 ± 2.5	0.94 ± 0.05
90	35.6 ± 2.1	0.96 ± 0.04

*Means ± SD.

3.2. Water Activity and Moisture Content. Moisture content and water activity values of olive cake over 90 days of fermentation are shown in Table 2. It may be noted from the table that moisture contents of the unfermented (0 day) and fermented olive cake ranged from 33.3% to 35.6%, while the corresponding values for water activity were 0.93 and 0.96, respectively. In either case, no statistically significant difference was found during the 90 days of fermentation. The numerically higher water activity could be due to the fat contents that let the water molecules to move easily in the media (olive cake). Also, these results agreed with the microorganism determinations.

3.3. Microorganism Examinations. Figure 4 reports the total counts, the thermophilic bacteria, and the total mold count of olive cake over 90 fermentation days. Figure 4 shows that the total plate count of the unfermented (0 day) and fermented olive cake (during 90 days of fermentation) ranged from 2.1×10^8 to 2.4×10^8, and the corresponding values for the bacteria were 1.7×10^4 to 1.9×10^4, respectively. In either case, no significant difference was found. As for the mold count, Figure 4 indicates that the total mold count of the unfermented (0 day) and fermented olive cake (during 90 days of fermentation) ranged from 1.5×10^2 to 1.7×10^2 with no statistically significant difference. These high values of growth of both types of bacteria could be due to high available nutrients and high amounts of water activity. Also, the type of dominant mold was identified, and the *Aspergillus* spp. was found during the fermentation periods.

3.4. Temperature Measurements. The data that pertain to temperature over the course of the study are reported in Figure 5. Noting that the ambient temperature is the baseline (reference value) that represents the current practice (without the system), the data in Figure 5 indicate that the well alone effected a rise in water temperature in the range of

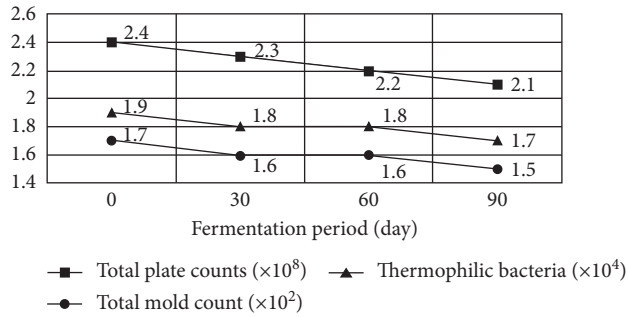

Figure 4: The total counts, the thermophilic bacteria, and the total mold count of olive cake during 90 days of fermentation.

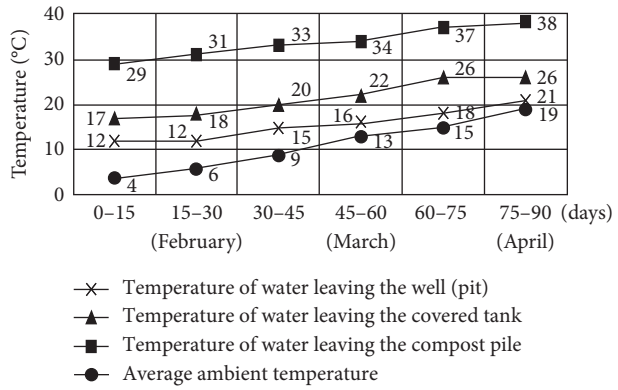

Figure 5: Water temperature data for three months.

2 to 8°C, with the lower values being in the warmer weather and the higher values for the cooler period that is more characteristic of the olive milling season.

It should be emphasized here that the well can provide this boost in water temperature at a steady basis with no regard to flow rate. That is, the well can provide the total water requirements of the mill while maintaining this temperature boost. Also, the data indicate that the combination of the well and the covered tank lead to a rise in water temperature before entering the boiler in the range of 7 to 13°C. This rise in water temperature is apparently better (higher) than that by the well alone and could lead to significant fuel savings. It should be emphasized here that, given ample time, the tank, like the well, can provide this boost in water temperature at a steady basis with no regard to flow rate. That is, the tank can provide the total water requirements of the mill while maintaining this temperature boost.

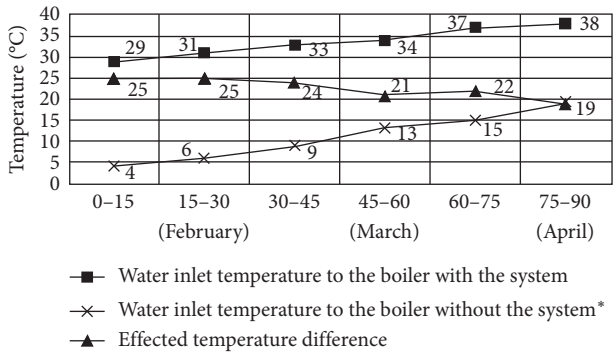

Figure 6: Water temperature entering the boiler with and without the system.

As for the whole system (the three stations), the rise in water temperature relative to the ambient is reported in the last column of Figure 6. It may be readily seen from Figure 6 that, under the flow rate through the heat exchanger in this work (0.4 m^3/h), which is mainly due to limitations of exchanger pipe size and flow speed, the system effected significant raises in water temperature entering the boiler ranging from 19°C up to 25°C, which holds a promising potential for the system to satisfy much of the mill needs at this range of temperature before entering the boiler provided a large enough pile (pile scale up) is used to handle larger flow rates. Note that under steady-state conditions, the temperature of water entering the boiler from the rooftop tanks (without the system) is equal to the ambient.

Given that 1.0 ton of olive fruit requires 200 kg of hot water (a ratio of 5 : 1) and should a scale up system be considered, the current system is sufficient. The 20 m^3 well can process 100 tons of olive fruits, and the 0.4 m^3/h flow rate from the pile is sufficient for 2 tons of olives/h, which is more than the capacity of almost all existing mills. The scenario may involve filling the well in the mill's off time each night which is equivalent to a 4-hour residence time during which water temperature rises by 2 to 8°C above the ambient as was demonstrated during our work.

4. Conclusions

Based on the findings reported herein and the fact that olive milling season in the Mediterranean region coincides with cold winter times, it may be concluded that combining geothermal (well) and on-site aerobic biological treatment of the milling solid by-product (olive cake) holds a promising potential as a simple, low-cost energy source for the needed hot water. This is evidenced by the fact that the system effected significant raises in water temperature entering the boiler to satisfy much of the mill needs at this range of temperature before entering the boiler provided a large enough pile (pile scale up) is used to handle larger flow rates. Consequently, significant savings are possible by implementing the system.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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Research Article

Organic Acids, Antioxidants, and Dietary Fiber of Mexican Blackberry *(Rubus fruticosus)* Residues cv. Tupy

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Blackberry fruit processing generates residues comprised of peel, seeds, and pulp that are abundant in flavonoids, colorants, and organic acids. The objective of this study was to determine the organic acids, antioxidants, and dietary fiber content of blackberry residues and compare antioxidants and dietary fiber content of a prune-based commercial product. The ABTS, DPPH, and FRAP methodologies were used for antioxidant capacity. The blackberry residues exhibited a high amount of malic acid (5706.37 mg/100 g db), phenols (4016.43 mg GAE/100 g db), and anthocyanins content (364.53 mg/100 g db) compared with the commercial product. These compounds contributed to the antioxidant capacity (by ABTS) of both products but were 20 times higher in blackberry residues. The fruit residues were able to reduce iron (by FRAP) 4.4 times compared to the prune-based product. Total dietary fiber (44.26%) and functional properties as water retention capacity (2.94 g/g), swelling capacity (5.00 mL/g), and fat absorption capacity (1.98 mL/g) of blackberry residues were significantly higher than those of the commercial sample. The results demonstrated that, due to its antioxidant compounds and functional properties, the blackberry residue can be considered a source of components with potential benefit to human health.

1. Introduction

Berries are mainly consumed fresh but are also used to manufacture products such as juices and concentrates [1]. The residues generated after separation of peels, seeds (4.4% to 12.2%), and pulp are commonly discarded by the industry but still have flavonoids, colorants, pectins, and organic acids [2–4] that can be used or recovered.

Organic acids and sugars are soluble constituents in berries that are responsible for taste and serve as an index of fruit ripeness; both factors affect consumer acceptability [5]. Previous research has reported that different species of whole berries contain organic acids such as citric, malic, tartaric, fumaric, and shikimic acids in total content ranges of 21.5 to 235 mmol/kg, specifically of 45.1 mmol/kg for the *fruticosus* species [6]. Published literature about the content of these individual compounds in blackberry *(Rubus fruticosus)* residues was not found.

Blackberry *(Rubus fruticosus)* fruits also contain phenolic compounds as anthocyanins, flavonols, chlorogenic acid, and procyanidins, which can have beneficial effects on human health [7]. Phenols are considered compounds with potent

antioxidant and free-radical scavenging properties that protect important biomolecules against oxidative damage [8]. Several studies have evaluated these compounds in blackberries *(Rubus)* of different species and varieties (*R.* sp. hyb Marion, *R. laciniatus* Evergreen, *R.* spp. Tupy, and *R. fruticosus*) as a whole fruit and in pulp or seeds, including the use of several extraction technologies (supercritical carbon dioxide extraction, ultrasound assisted extraction, pressurized liquid extraction, etc.) to recover phenols, anthocyanins, fatty acids, phytosterols, and tocopherols, compounds responsible for antioxidant activity [9–14]. Fewer studies have been performed on the evaluation of antioxidants in the bagasse or residues generated by the blackberry fruit processing [11, 13, 15]. Phenolic compounds and dietary fiber are generally studied separately, probably because of the differences in their chemical structures, physicochemical and biological properties, and metabolic pathways [16]. However, these are plant food constituents that are associated with many health benefits and have been demonstrated to reduce the risk of developing cancer and some chronic diseases [17, 18]. Dietary fiber has an essential role in intestinal health and appears to be significantly associated with a reduction of cholesterolemia and modification of the glycemic response [19].

Considering that most studies evaluate the whole fruit but not the residues that may have a great potential as a source of bioactive compounds (dietary fiber, antioxidants, fatty acids, etc.), the objective of this study was to determine the organic acids, antioxidants, and dietary fiber content of blackberry *(Rubus fruticosus)* cv. Tupy residues (comprised of seeds, peel, and pulp) and compare antioxidants and dietary fiber values with a commercial product.

2. Materials and Methods

2.1. Sample Preparation. Blackberries (*Rubus fruticosus* cv. Tupy) were collected from Atotonilco, Hidalgo, Mexico, in January 2016. Fruits without external injuries were selected and washed. Blackberries were processed into juice and the resulting residues (seeds, peel, and pulp) were collected as follows: fruit was stirred using an industrial blender (38BL52 LBC10, Waring Commercial, Torrington, CT, USA) and then passed through a conventional strainer; the retained bagasse (seeds and peel) was collected and mixed with the pulp (precipitate) obtained after juice was clarified by centrifugation (Allegra 25™, Beckman Coulter, Palo Alto, CA, USA) at 15,300g for 30 min at 4°C. All blackberry residues (BR) (bagasse plus pulp) were lyophilized (7753020, Labconco, Kansas City, MO, USA), milled, and sieved together to obtain a particle size of 500 μm. The samples were stored in sealed plastic bags at −30°C until further analysis. A prune *(Prunus domestica)*-based commercial product (CP) present on the market as tablets was milled and sieved under the same conditions described for blackberry residues. The CP was included in the present study only for comparison of antioxidants, dietary fiber, and color.

2.2. Chemical Composition. The chemical composition of the BR was determined according to the AOAC [20] for moisture (Official Methods 925.09), ash (Official Methods 923.03), protein (Official Methods 991.20, conversion factor 6.25), ethereal extract (Official Methods 930.39), and AOAC [21] for total, soluble, and insoluble dietary fiber (TDF, SDF, and IDF, resp.). Total carbohydrates were calculated by difference of moisture, ash, protein, ethereal extract, and total dietary fiber values [22].

2.3. Quantitative Analysis of Mineral Composition. A field emission type scanning electron microscope (FE-SEM) (JEOL SSM 6300, Jeol de Mexico, CDMX, Mexico) with a 300,000 magnification range and a resolution of 30 kv was used. The BR sample was placed on a double-sided graphite tape and was then coated with a thin layer of gold (1 mm) using an ionizing machine (Denton Vacuum LLC, Moorestown, NJ, USA) at a pressure of 20 millitorr and a current of 20 mA for 4 min. Quantitative analysis of mineral composition was obtained by means of a spectrum that forms part of the scanning electron microscopy.

2.4. Quantification of Organic Acids. Individual organic acids (oxalic, malic, citric, fumaric, and ascorbic) were quantified by high-performance liquid chromatography (HPLC). Briefly, 20 mL of metaphosphoric acid at 4.5% (w/v) was added to 500 mg of the sample and the mixture was stirred with magnetic shaking (P-Selecta, Asincro, Spain) for 15 min under dark conditions. After the mixture was centrifuged (Allegra 25, Beckman Coulter, Palo Alto, CA, USA) at 9000 rpm for 15 min, the supernatant was diluted to a final volume of 25 mL with metaphosphoric acid 4.5%. Prior to injection into the HPLC, extracts were filtered through filter paper no. 1242 and then a 0.45 μm Millipore PVDF membrane [23]. The HPLC was equipped with an isocratic pump (model PU-II, Micron Analitica, Madrid, Spain), an AS-1555 automatic injector (Jasco, Tokyo, Japan), a Sphereclone ODS(2)250×4.60 mm, 5 μm Phenomenex column (Torrance, CA, USA), and a UV-visible detector (Thermo Separation Spectra Series UV100, San Jose, CA, USA). The mobile phase was 1.8 mM H_2SO_4 at pH 2.6. UV detection at 215 nm and a flow rate of 0.4 mL/min were used for the analysis of organic acids, while a flow rate of 0.9 mL/min and UV detection at 245 nm were set for ascorbic acid. Linear calibration curves were obtained for quantification from stock solutions of all the identified compounds (oxalic, malic, citric, fumaric, and ascorbic acids). Data were analyzed using the Biochrom 2000 (version 3.0) software. Identification was made by comparing retention times of commercial pure standards, and quantification was based on the UV signal response, and the resultant peak areas in the chromatograms were plotted against concentrations obtained from standards (1.34, 1.40, 1.39, 1.45, and 25–100 mg/mL for oxalic, malic, citric, fumaric, and ascorbic acids, resp., in metaphosphoric acid 4.5%). Organic acid contents in blackberry residues were expressed in mg/100 g of dry basis (db).

2.5. Fatty Acid Profile. Blackberry residues were washed according to previous studies by Zafra-Rojas et al. [24] and Siriwoharn et al. [1] with minor modifications to simulate the industrial process. Briefly, 160 mL of deionized water was added to 10 g of sample and centrifuged at 10,000 rpm for

10 min; the supernatant was discarded and centrifugation repeated with 80 mL of deionized water; the resulting precipitate was washed under the same centrifuging conditions with methanol up to six washes until the supernatant was no longer red or was colorless.

Following the procedure suggested by Añorve-Morga et al. [25], 110 mL of chloroform-methanol ($CHCl_3$: MeOH, 2 : 1 vol/vol) was added to the washed samples and vortexed for 5 minutes and stored at 4°C for 48 h. Subsequently, 500 μL of the aqueous phase containing $CHCl_3$: MeOH was transferred to a tube and 1 mL of boron trifluoride-methanol (BF_3 : MeOH) (12.5 : 100, vol/vol) was added prior to heating the sample at 95°C in a water bath for 6 min [26]. After the mixture was allowed to cool and 1 mL of hexane plus 1 mL of hexane-saturated water was added, it was stirred and the aqueous phase was discarded. Two milliliters of hexane-saturated water was added, stirred, and reserved in a freezer until use. Prior to analysis, the organic phase was evaporated and the fatty acids were taken up in hexane. FAME (methyl esters of fatty acids) were analyzed and identified by comparing their retention times to standards of known amounts (FAME Mix C4-C24 Supelco® by Sigma-Aldrich, St. Louis, MO, USA).

For analysis of the fatty acids, a gas chromatograph (PerkinElmer, Australia) was used: AutoSystem XL equipped with a flame ionization detector (FID) and a Supelco® SP TM-2560 capillary column (75 m × 0.18 mm × 0.14 μm) using the splitless injection mode (1 μL). The initial column temperature was set at 150°C, increased at a rate of 4°C/min to 214°C, held for 2 min, and then increased to 244°C at 2.5°C/min and held for 5 min. The injector and detector temperatures were 230°C and 250°C, respectively. The ramp split was approximately 1 μL and nitrogen was used as a carrier gas at a constant flow of 1 mL/min.

2.6. Antioxidants Extraction. Two hundred milligrams of sample (BR and CP) was placed in a capped centrifuge tube; 10 mL of acidic methanol/water (50 : 50, v/v) was added and the tube was thoroughly shaken at room temperature for 1 h. The mixture was centrifuged at 3400 rpm for 20 min and the supernatant was recovered. Ten milliliters of acetone/water (70 : 30, v/v) was added to the residue and the shaking and centrifugation were repeated. Methanolic and acetone extracts were combined and used to determine the antioxidant capacity associated with extractable antioxidants [27].

2.6.1. Determination of Total Phenolic Content (TPC). Total phenolic content was determined by the Folin–Ciocalteu procedure [28]. Briefly, 100 μL of the sample was mixed with 500 μL of 1 : 10 diluted Folin–Ciocalteu reagent. Then, 400 μL (7.5%) of sodium carbonate was added and the mixture was incubated for 30 min at room temperature. The absorbance of the mixture was measured at 765 nm in the microplate reader (Power Wave XS UV-Biotek, software Gen5 2.09, Winooski, VT, USA). The standard curve was developed with concentrations of 0, 100, 200, and 300 mg of gallic acid/L. Gallic acid was used as reference standard and the results were expressed as mg of gallic acid equivalents per 100 grams of dry basis (mg GAE/100 g db) of blackberry residues and commercial product.

2.6.2. Determination of Anthocyanins. The anthocyanin content was determined through the differential pH method [29]. Two buffer solutions were prepared: 0.025 M potassium chloride at pH 1.0 and 0.4 M sodium acetate at pH 4.5. The pH was adjusted by adding concentrated HCl. The extracts of blackberry residues and prune (commercial product) were diluted in these two buffer solutions. After 15 min under dark conditions at room temperature, absorbance was measured at 510 and 700 nm using a microplate reader (Power Wave XS UV-Biotek, software Gen5 2.09, Winooski, VT, USA). The absorbance of the anthocyanins was calculated according to

$$\begin{aligned}\text{Abs} = {} & \left(\text{Abs}_{510} - \text{Abs}_{700}\right) \text{pH}_{1.0} \\ & - \left(\text{Abs}_{510} - \text{Abs}_{700}\right) \text{pH}_{4.5}.\end{aligned} \quad (1)$$

The concentration of anthocyanins in the extracts was calculated according to

$$\text{Anthocyanins } (\text{mg/L}) = \frac{(\text{Abs} \times \text{MW} \times \text{DF} \times 1000)}{\varepsilon \times 0.52}, \quad (2)$$

where Abs is the absorbance, MW is the molecular weight, DF is the dilution factor, and ε is the molar absorptivity, considering the molar absorptivity (ε) of 26,900 L mol^{-1} cm^{-1}, the molecular weight of 449.2 g/mol for cyanidin-3-glucoside, and the path length (0.52 cm) of the well. The results were expressed as mg of cyanidin-3-glucoside equivalent/100 grams on a dry basis (mg cy-3-gl/100 g db).

2.7. Antioxidant Capacity

2.7.1. ABTS Method. The radical cation ($ABTS^{\bullet+}$) was produced by reacting 7 mM ABTS stock solution with 2.45 mM potassium persulfate under dark conditions at room temperature for 16 h before use. The ABTS solution was diluted with deionized water until an absorbance of 0.70 ± 0.10 at 754 nm. An aliquot of 20 μL of the extract was added to 980 μL of diluted ABTS solution, and absorbance readings were taken after incubation for 7 min at room temperature. The absorbance of the mixture was measured at 754 nm in a microplate reader (Power Wave XS UV-Biotek, software Gen5 2.09, Winooski, VT, USA). The standard curve was linear with concentrations of 0, 10, 20, 30, 40, and 50 mg of ascorbic acid/L [30]. The antioxidant capacity of blackberry residues and commercial product (prune) was expressed as milligrams of ascorbic acid equivalents per 100 grams of dry basis (mg AAE/100 g db).

2.7.2. DPPH Method. Antiradical activity was measured with 1,1-diphenyl-2-picrylhydrazyl radical (DPPH) [31]. An ethanolic solution (7.4 mg/100 mL) of the stable DPPH radical was prepared. Then, 100 μL of the extract was taken into vials and 500 μL of the DPPH solution was added, and the mixture was left to stand for 1 h at room temperature. Finally, absorbance was measured at 520 nm using a microplate reader (Power Wave XS UV-Biotek, software Gen5 2.09,

Winooski, VT, USA). The standard curve was developed with concentrations of 0, 50, 100, 200, and 300 μmol of Trolox. Free-radical scavenging activity was expressed as μmol of Trolox equivalents per 100 g of dry basis (μmol TE/100 g db) of blackberry residues and commercial product.

2.7.3. Ferric Reducing Antioxidant Power (FRAP). The FRAP was assayed according to Benzie and Strain [32] with some modifications. The stock solutions included 300 mM acetate buffer (3.1 g $C_2H_3NaO_2.3H_2O$ and 16 mL $C_2H_4O_2$) (pH 3.6), 10 mM TPTZ (2,4,6-tripyridyl-s-triazine) solution in 40 mM HCl, and 20 mM $FeCl_3.6H_2O$ solution. The fresh solution prepared by mixing 25 mL acetate buffer, 2.5 mL TPTZ solution, and 2.5 mL $FeCl_3.6H_2O$ solution was warmed at 37°C before use. The sample (30 μL) was mixed with 90 μL of distilled water and 900 μL of the FRAP solution for 10 min in the dark. Absorbance was measured at 593 nm using a microplate reader (Power Wave XS UV-Biotek, software Gen5 2.09, Winooski, VT, USA). The standard curve was developed with concentrations of 0, 200, 400, 600, 800, and 1000 μmol ferrous sulfate ($FeSO_4$) 5 M, and the results were expressed as μmol Fe(II) per 100 g dry basis (μmol Fe(II)/100 g db) of blackberry residues and commercial product.

2.8. Properties of Dietary Fiber

2.8.1. Water Retention Capacity (WRC). An aliquot (0.5 g) of BR and CP was placed in 15 mL centrifuge tubes and 10 mL of distilled water was added. The tubes were shaken manually for 10 minutes and then allowed to stand for 24 h at room temperature prior to centrifugation (Hamilton Bell VanGuard V6500, New Jersey, USA) at 3400 rpm for 20 minutes. Immediately, the supernatant was removed and the pellet weighed. The water retention capacity was measured as the amount of water retained by the sample (g/g dry basis) [33, 34] and was calculated by

$$\mathrm{WRC}\,(\mathrm{g/g}) = \frac{(\mathrm{WFS} - \mathrm{WDS})}{\mathrm{WDS}}, \tag{3}$$

where WFS is the weight of the fresh sample (g) and WDS is the weight of the dry sample (g).

2.8.2. Swelling Capacity (SC). The lyophilized samples (200 mg) were placed in a graduated cylinder (0.1 mL) and the volume occupied (V_0) (mL) was measured. Then, 10 mL of distilled water was added and the mixture was stirred manually for 5 min and allowed to stand for 24 hours at room temperature. The final volume of the samples (V_1) (mL) was measured [33, 35] and the swelling capacity of BR and CP (mL/g) was calculated using

$$\mathrm{SC}\,(\mathrm{mL/g}) = \frac{(V_1 - V_0)}{\mathrm{sw}}, \tag{4}$$

where V_1 is the final volume (mL), V_0 is the volume occupied by the lyophilized sample (mL), and sw is the sample weight (g).

2.8.3. Fat Absorption Capacity (FAC). Five hundred milligrams (WDS) of BR and CP was placed in separated 15 mL centrifuge tubes and 10 mL of sunflower seed oil was added. The mixtures were shaken manually for 10 minutes and then allowed to stand for 24 h at room temperature. Samples were centrifuged at 3400 rpm (Hamilton Bell VanGuard V6500, New Jersey, USA) for 20 minutes and immediately the supernatant was removed and the pellet weighed (WFS). The fat absorption capacity was measured as the amount of fat retained by the sample (mL/g dry basis) [33, 34] and was calculated using

$$\mathrm{FAC}\,(\mathrm{mL/g}) = \frac{(\mathrm{WFS} - \mathrm{WDS})}{\mathrm{WDS}}, \tag{5}$$

where WFS is the weight of the fresh sample (g) and WDS is the weight of the dry sample (g).

2.9. Color Measurement. Color of BR and CP was measured using a colorimeter (Konica Minolta CM-608d, Sensing, Inc., Japan) set with the D65 illuminant and 10° observer angle. The CIE-Lab values were recorded, where L^* indicates lightness ($L = 0$ or 100 indicates black or white, resp.); a^* is the axis of chromaticity between green (−) and red (+), and b^* is the axis between blue (−) and yellow (+). Numerical values of L^*, a^*, and b^* were used to obtain chroma ($C = [a^2 + b^2]^{1/2}$) and *Hue* angle (h°) ($h^\circ = tg^{-1}(b/a)$) [36].

2.10. Statistical Analysis. All experiments were carried out in triplicate and expressed as mean ± standard deviation (SD). The variables used to compare the blackberry residues with commercial product were analyzed by a t-test and a confidence level of 95% using the SPSS® System for WIN™ version 15.0.

3. Results and Discussion

3.1. Chemical Composition, Minerals, and Organic Acids. Table 1 shows the chemical composition, mineral content, and organic acids of blackberry residues. The BR had a moisture content of 74.83 ± 0.76% and could be described as a semisolid fibrous product from which the liquid has been extracted during juice production. Other fruit residues have similar moisture percentages, such as blackberry (*Rubus* sp.), mango *(Mangifera indica)*, and guava *(Psidium guajava)* residues (values between 70 and 90%) [11, 37].

Protein (2.60 ± 0.11%) and lipid (1.84 ± 0.03%) content in BR was higher than values reported for orange, guava, soursop, and mango residues, which ranged from 0.6 to 1.9% and from 0.25 to 0.48%, respectively [37]. Differences could be attributed to the high seed content in BR and the fact that these are mainly composed of oils [38]. Carbohydrates (9.07 ± 0.80%) and total dietary fiber (11.12 ± 0.22%) were the major components after moisture, while ashes were minor constituents (0.54 ± 0.02%), although they were higher than ash percentages reported for blackberries bagasse by Pasquel Reátegui et al. [11].

The minerals found in BR were sodium, magnesium, potassium, calcium, iron, sulfur, silicon, and chlorine (Table 1).

Table 1: Chemical composition, analysis of minerals content, and organic acids of blackberry residues.

	Blackberry residues[a]
Composition (%)	
Moisture	74.83 ± 0.76
Protein	2.60 ± 0.11
Lipids	1.84 ± 0.03
Total carbohydrates	9.07 ± 0.80
Total dietary fiber	11.12 ± 0.22
Ash	0.54 ± 0.02
Element (%)	
C	48.02 ± 4.69
N	7.54 ± 0.43
O	43.04 ± 4.16
P	0.19 ± 0.08
Na	0.01 ± 0.01
Mg	0.11 ± 0.05
K	0.81 ± 0.17
Ca	0.08 ± 0.04
Fe	0.03 ± 0.05
Cu	-
Zn	-
Mn	-
S	0.05 ± 0.01
Si	0.02 ± 0.04
Cl	0.06 ± 0.05
Organic acids (mg/100 g db)	
Oxalic acid	59.51 ± 8.18
Malic acid	5706.37 ± 123.38
Citric acid	125.54 ± 0.61
Fumaric acid	230.25 ± 2.47
Ascorbic acid	6.00 ± 1.00

[a]Mean ± standard deviation ($n = 3$).

In other blackberry studies, in addition to the mentioned minerals, copper, zinc, manganese, and selenium were also found [39–41]. These differences may be mainly attributed to the type of soil in which these fruits were cultivated; for instance, the soil of Atotonilco, Hidalgo, Mexico (where the blackberry was harvested) consists of layers of volcanic ash and pumice and a conglomerate of gravel and sand characterized by the presence of potassium and other minerals in low concentrations [42].

Many fruits accumulate organic acids in their pulp and peel at certain stages of their development [43, 44]. In the BR, ascorbic acid content was low (6.00 ± 1.00 mg/100 g db) while malic acid was in higher concentrations (5706.37 ± 123.38 mg/100 g db) (Table 1) as has been reported for several fruits such as strawberry tree (*Arbutus unedo* L., Ericaceae), blackberry (*Rubus ulmifolius* Schott), and red apples (*Malus domestica* Borkh.) [45–47]. Evidence suggests that organic acids such as malic or citric acids may have a positive health benefit as antioxidants thanks to their ability to chelate metals [48]. On the other hand, environmental factors and cultivation practices (e.g., temperature, cultivar, minerals, and water availability) could affect the content of these organic acids in fruits [43].

Table 2: Fatty acids profile of blackberry residues (mg/100 g).

Palmitic acid (C16:0)	1.68 ± 0.40
Linolenic acid (C18:3n-3)	0.43 ± 0.07
Arachidic acid (C20:0)	7.68 ± 1.88
Eicosenoic acid (C20:1)	0.20 ± 0.00

Mean ± standard deviation ($n = 2$).

3.2. Fatty Acids Profile. Table 2 shows the fatty acids profile of BR. Four fatty acids were found and corresponded to 9.99 mg/100 g of blackberry residues. The saturated palmitic (C16:0) and arachidic (C20:0) acids were found in higher quantities as compared to the polyunsaturated and monounsaturated linolenic (C18:3n-3) and eicosenoic (C20:1) acids. These same fatty acids have been found in oil seeds in different species of blackberries (*Rubus ursinus* var. Marion and *Rubus laciniatus* Willd. var. Evergreen, *Rubus ulmifolius* var. Schott, and *Rubus fruticosus*) [38, 49, 50]. However, the amount found in the BR was lower than the values reported in these studies probably due to the presence of peel and pulp in addition to seeds so that the final concentration was affected.

3.3. Total Phenolic Content and Anthocyanins. The whole blackberry fruit is a good source of antioxidant compounds and has considerable levels of phenolic compounds, including anthocyanins. In blackberry (*Rubus fruticosus* L. var. Caingangue) residues such as peel and seeds, high concentrations (366 to 736 mg GAE/100 g fresh residues) have also been reported [15]. The BR exhibited significantly higher total phenolic content ($p < 0.05$) and anthocyanins in comparison with the commercial product in which anthocyanins were absent (Table 3), probably due to complete degradation during the transformation of fresh plums of prunes [51]. The phenolic content of the BR was higher than that reported by Huang et al. [52] for whole rabbiteye blueberry fruits (*Vaccinium ashei* cv. Brightwell) including peel, seeds, and pulp, for thornless blackberry (*Rubus laciniatus* cv. Hull), and for strawberry (*Fragaria* × *ananassa* cv. Toyonoka). The values for anthocyanins in the present study were similar to those described by Dai et al. [53] for Hull blackberry puree (534 mg/100 g db).

Patras et al. [54] evaluated TPC in strawberry (*Fragaria* × *ananassa* cv. EI Santa) and blackberry (*Rubus fruticosus* cv. Loch Ness) puree, and the amount of phenols (855 and 1694 mg GAE/100 g db, resp.) was lower than the value obtained for our BR. Differences in the total concentration of phenols in vegetables could be attributed to variations in the composition of the food matrix, geographical origin, cultivar/genotype, maturity, time and type of drying, and storage time [55]. Other factors that affect the recovery of phenolic compounds are extraction methods and conditions such as extraction temperature, time, particle size, and type of solvent [11, 56, 57].

Table 3: Total phenolic content, anthocyanins, and antioxidant capacity of blackberry residues[a] and commercial product.

	BR[a]	Commercial product
Parameters		
TPC (mg GAE/100 g db)	4016.43 ± 13.44*	1362.98 ± 52.84
Anthocyanins (mg/100 g db)	364.53 ± 7.36*	0.00 ± 0.00
Antioxidant capacity		
ABTS (mg AAE/100 g db)	5422.38 ± 71.50*	267.06 ± 13.86
DPPH (μmol TE/100 g db)	13656.27 ± 532.66*	16655.88 ± 272.02
FRAP (μmol Fe(II)/100 g db)	12511.44 ± 147.39*	2842.23 ± 109.41

Mean ± standard deviation ($n = 3$). * indicates significant difference ($p < 0.05$) between samples.

Table 4: Dietary fiber properties of blackberry residues and commercial product.

	BR[a]	Commercial product[b]
Parameters		
Total dietary fiber (%)	44.26 ± 0.09*	8.06 ± 0.12
Soluble dietary fiber (%)	5.90 ± 0.52*	1.96 ± 0.08
Insoluble dietary fiber (%)	38.35 ± 0.39*	6.09 ± 0.29
Properties		
Water retention capacity (g/g)	2.94 ± 0.10*	0.00 ± 0.00
Swelling capacity (mL/g)	5.00 ± 0.00*	0.00 ± 0.00
Fat absorption capacity (mL/g)	1.98 ± 0.03*	1.49 ± 0.02

[a]BR: blackberry residues. [b]Prune. * indicates significant difference ($p < 0.05$) between samples.

3.4. Antioxidant Capacity. BR presented high antioxidant capacity by ABTS and FRAP, but low by DPPH ($p < 0.05$) compared with the CP (Table 3). The antioxidant capacity by ABTS and the iron reduction ability of the BR were 20 and 4 times, respectively, those of the CP which in contrast had a DPPH value 1.2 times that of the BR. The reported antioxidant capacity of fruit residues such as pulp, peel, and seeds from strawberry (*Fragaria vesca* L.), carrot (*Daucus carota* L. spp. *sativus* var. *atrorubens* Alef.), and blueberry (202 mg VCEAC/100 g, 10190 μmol TE/100 g db, and 10400 μmol Fe(II)/100 g db, for ABTS, DPPH, and FRAP, resp.) [30, 58, 59] was lower than the values found for BR in the present study. The responsible compounds for the antioxidant activity are mainly phenols [60], but some pigments such as anthocyanins also contribute to the amount of phenolic compounds. The basic structural orientation of these compounds determines their antioxidant activity since the hydroxyl group has a higher ability to donate hydrogen atoms to free radicals [61]. Moreover, the position of the hydroxyl groups appears to be more important than their number to act as antioxidants [62]. Phenolic compounds also contribute by transferring an electron to reduce the ferric ion (III) to its ferrous (II) form [60]. But the presence of lipids as tocopherols that are found in seeds [38] may impact the overall antioxidant activity of the evaluated BR that does contain seeds.

3.5. Dietary Fiber and Functional Properties. It is well known that agroindustrial waste is rich in dietary fiber. Dietary fiber contains significant amounts of antioxidant compounds and other substances with positive effects on health [63]. Total, soluble, and insoluble dietary fiber and functional properties of BR and CP are described in Table 4. In general, the percentage of total dietary fiber and their properties was higher in BR compared with CP. The analyzed functional properties such as WRC, SC, and FAC were significantly higher ($p < 0.05$) in BR (2.94 ± 0.10 g/g, 5.00 ± 0.00 mL/g, and 1.98 ± 0.03 mL/g, resp.). The total fiber content of the BR was similar to the one described for grapefruit fiber (44 g/100 g) but SC was higher than that reported for oat and apples fibers (2.3 and 3.4 mL/g, resp.) [64, 65]. In terms of WRC (9.7 g/g), our values were lower than those reported by Basanta et al. [66] who evaluated cherry fibers. The content of soluble dietary fiber affects SC, and insoluble dietary fiber determines the WRC and FAC [67]; hence, the properties of dietary fiber depend on the proportion of SDF and IDF. Since the BR contains IDF, it may help to ease constipation, increase excretion, and bind bile salts, cholesterol, and carcinogenic compounds allowing their excretion through the feces [68, 69]. Intake of IDF may also prevent conditions such as colon cancer, hypercholesterolemia, and intoxication. The BR also exhibited a high capacity to increase its volume in excess of water, which may contribute to enhancing satiety thanks to its content of SDF [64, 70].

3.6. Color Parameters. Color is an important quality attribute because it can condition consumer acceptability. Table 5 shows the color parameters for BR and CP. The luminosity (L^*) for both samples was significantly different ($p < 0.05$); BR presented low values, and according to the a^* and b^* axes, the BR and CP were found in the reddish quadrant. The *Hue* angle can be between 0° and 90°, representing the red (0°)

Table 5: Color parameters of blackberry residues and commercial product.

Color parameters	BR[a]	Commercial product[b]
L^*	16.73 ± 0.00*	37.97 ± 0.68
a^*	16.77 ± 0.03*	2.49 ± 0.08
b^*	10.88 ± 0.00	11.38 ± 0.35
h°	32.98 ± 0.06*	77.64 ± 0.63
Chroma	20.00 ± 0.02*	11.65 ± 0.33

[a]BR: blackberry residues. [b]Prune. * indicates significant difference ($p < 0.05$) between samples.

and orange-yellow (90°) colors; based on this value, the BR is within the red-orange tone, and the CP exhibited a yellow tone.

Color saturation (chroma) was between 0 and 60 and results showed that the CP had lower saturation than the BR [71, 72]. Malien-Aubert et al. [73] evaluated the color of some commercial extracts from grapes *(Vitis vinifera)*, elderberry *(Sambucus nigra)*, purple carrot *(Daucus carota)*, red radish *(Raphanus sativus)*, black currant *(Ribes nigrum)*, red cabbage *(Brassica oleracea)*, and chokeberry *(Aronia melanocarpa)*; the chroma values from these two last sources were close to the values obtained for the BR (around 20). Red, blue, and purple colors in fruits depend on the anthocyanins and also the number and orientation of the hydroxyl and methoxyl groups of the molecule; for example, an increase in hydroxylation produces a shift to blue tones whereas an increase in methoxylation generates red coloration [74]. This suggests that the BR may contain a high amount of methoxyl groups. According to the European Union, all dyes derived from anthocyanins are recognized as natural dyes under the E163 classification, ranging from E163a (cyanidin: red food coloring) to E165f (petunidin: dark red food coloring) [72]. Therefore, based on our results, the BR could be a good source of natural dyes thanks to the presence of anthocyanins.

4. Conclusions

The results demonstrated that blackberry residues are a good source of organic acids and contain elevated amounts of phenolic compounds and anthocyanins and antioxidant capacity compared to the prune-based commercial product. These residues also exhibited better dietary fiber functional properties and a rich reddish color which make them a potential source of components with a beneficial role in human health, particularly for patients with chronic noncommunicable diseases.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

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Research Article

Production of Nutritious Flour from Residue Custard Apple (*Annona squamosa* L.) for the Development of New Products

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Currently, the fruit processing industry generates a high volume of waste in fruits that have not reached a quality standard for consumption or by-products generated throughout the production process. To reduce this waste, mitigating measures, such as reuse in food formulations, have been proposed. In this work the custard apple bagasse flour *(Annona squamosa* L.*)* (CAB) was produced and incorporated into cookie formulations in different proportions (5 to 50%) evaluating its acceptability. The CAB flour was characterized by physicochemical analysis, proximate composition, mineral analysis, determination of the phenolic content, and antioxidant capacity. The results of the physicochemical and proximate characterizations show that the processed flour presents values and specifications suitable for food formulations. The mineral composition of the CAB flour responds to more than 20% of the daily intake of nutrients, highlighting the Cu, Fe, Mn, Zn, Ca, and Mg. The composition of phenolic compounds for CAB flour and cookies formulations presented values ranging from 200 to 658 mg GAE/100 g, similar to flour and formulations prepared of residues tropical fruit, while $DPPH^{\bullet}$ inhibition showed a variation of 9.68–10.75%. Cookies made from the CAB flour showed high acceptability making the flour promising in the nutritional incorporation in food formulations.

1. Introduction

The production, commercialization, and consumption of tropical fruits have increased significantly in the international market due to their sensory, nutritional, and therapeutic properties; however, the food industry generates high amount of residues from these fruits [1, 2]. According to a recent survey by the Food and Agriculture Organization of the United Nations (FAO), about 1.3 billion tons of foods are wasted worldwide each year, which accounts for one-third of total food industry production [3, 4]. Allied to this fact, the fruit processing industry deals with a large percentage of by-products, such as peel, seeds, and bagasse, which increase the proportion of residues at the end of processing.

The custard apple *(Annona squamosa* L.*)* belongs to the Annonaceae family, with about 129 genus and more than 2000 species, being a fructiferous found in tropical and subtropical regions, adapted to the climatic conditions of China, Africa, South America, Australia, India, Mexico, the United States, and Thailand. The expansion of custard apple consumption is related to the medicinal and nutritional properties, as well as its pleasant flavor. The fruit of the tree is widely known and consumed due to its medicinal and nutritional properties, as well as its pleasant flavor. The health related components of the fruit include vitamins A, B, C, E, and K1, antioxidants, polyunsaturated fatty acids, and the presence of essential minerals. Furthermore, this plant has proved that it has a variety of compounds like acetogenins which are responsible for antifeedant, antimalarial, cytotoxic, and immunosuppressive activities and diterpenes isolated from the title plant have anti-HIV principle and antiplatelet aggregation activity [5–7].

Brazil is the second largest producer of custard apple in the world, after only Mexico (Seagri 2015), especially semiarid region of the Northeast, which owns 94% of the area under cultivation in Brazil, with the characteristic feature of family farming [8]. One of the bottlenecks in custard apple production is the high postharvest metabolic activity, accelerating the maturation process over a limited period of time, and limiting fruit shelf life. Due to its high moisture content, it becomes extremely perishable, especially in regions with high temperature, associated with absence or inadequate postharvest management and microbial infection, constituting a negative factor in its conservation [9]. In addition, the literature reports that about 30% to 40% of fruits harvested do not reach the best standards, being marketed at lower prices [8].

Recently, mitigating measures aimed at reducing fruit waste and reuse of by-products throughout production are reported in the literature [3, 10, 11]. Mostly, fruits that do not reach commercial standards, as well as after treatment residues, present similar or higher contents of bioactive compounds in relation to the final product [12]. In this way, the development of new products or food formulations through the incorporation of these residues becomes an alternative to minimize losses during the processing of fruits.

In this context, for the development of new products or food formulations, a nutritious meal was developed from custard apple bagasse, evaluating the physicochemical properties, proximate composition, mineral analysis, total phenolic content, and antioxidant activity. After obtaining the CAB flour, cookies type biscuits were prepared in different concentrations, verifying the organoleptic properties and acceptability among the consumers.

2. Materials and Methods

2.1. Obtaining and Sanitizing of Custard Apple Fruit. The custard apple used in this study was purchased from a local supermarket (Alagoas, Brazil). Initially, the sanitization occurred by passing the fruits through a washing step with running water in order to discard the parts that could not be used, after which they were immersed into a solution of chlorinated water at 200 ppm. They were then dried at 25°C for 30 min and manually peeled for separation of the pulp, seed, and peel.

2.2. Preparation of Custard Apple Bagasse Flour. After the cleaning process, separation of the components, and removal of the juice, the bagasse was spread on nonstick trays and taken to a greenhouse to dry at 65°C for 24 h. Then it was crushed with the help of a domestic blender and then sieved to obtain the flour. Finally, it was packed in a sealed glass container and stored until the analyses were carried out.

2.3. Physicochemical Characterization. For determination of pH, total titratable acidity in citric acid, soluble solids, and vitamin C was weighed 5 g of flour and diluted in 50 mL of distilled water; the contents were stirred until the particles were uniformly suspended, allowing the mixture to stand for 30 min, followed by filtration on filter paper [13]. The pH was measured in a pH meter (Ionlab model pH 200 ATC) calibrated with pH 4 and 7 buffer solution, while the soluble solids (°Brix) analysis was measured in a refractometer (RUDOLPH J47). Total titratable acidity in citric acid was achieved by titration with 0.1 mol/L potassium hydroxide solution (KOH) until it reached a pH of 8.1. The results were expressed in grams of citric acid/100 g sample [13]. The vitamin C content was obtained by titrating the sample diluted with 0.03 mol/L iodine solution with starch indicator. The results were expressed as percentages per 100 g of sample [14]. The result was calculated by using

$$\text{Ascorbic acid } (\%) = \frac{V}{F} \times P, \tag{1}$$

where V is volume of potassium iodate used in titration of the sample, F is potassium iodate equivalence factor according to normality (0.8806), and P is samples weight (g); results were expressed in percentage of ascorbic acid.

The water absorption capacity (WAC) was determined by the weight difference of the water absorbed by the sample. Initially 0.5 g of sample was mixed with 5 mL of distilled water contained in a test tube, centrifuging at 3000 rpm for 15 min. After centrifugation the sample remained standing for 30 min and the water residue was weighed [15]. Calculation of the water adsorption capacity is shown in

$$\text{WAC } (\%) = \frac{\text{Weight of water } (\text{g})}{\text{Weight of sample } (\text{g})} \times 100. \tag{2}$$

2.4. Proximate Composition Characterization. For the ash determination, approximately 1 g of flour was weighed in a preincinerated crucible. The calcination process was carried out for 5 h at 550°C. The crucible with the sample was left in a desiccator until it cooled and was later weighed [16].

The moisture content was quantified from 1 g of the sample in a preheated crucible and tared. The sample was heated at 105°C in a greenhouse for 3 h, cooled in a desiccator to room temperature, and weighed [13].

The lipid content was determined by weighing about 1 g of sample on a previously tared and oven dried filter paper at 105°C for 30 min. Then, the sample cartridge was introduced into the Soxhlet extractor by flowing with petroleum ether over the 6 h period. Subsequently, the cartridge was removed from the extractor and dried in an oven at 105°C for 30 min and then placed in a desiccator until it reached room temperature to be weighed [16].

For the determination of total nitrogen, a microdigester/distiller (Tecnal-mod.TE 0363/TE007) was used by the Kjeldahl method [17].

To determine the crude fiber, approximately 2 g of the sample (W) was weighed on the analytical balance and defatted by the Soxhlet method using petroleum ether followed by drying at 60°C for 1 h to remove the solvent. After defatting the sample, acid digestion (1.25% H_2SO_4) was refluxed for 30 min from boiling and washing of the sample with hot water until the neutralization of the same. Then alkaline (1.25% NaOH) digestion was carried out with reflux for 30 min from boiling and washing the sample with hot water until

neutralization thereof. After washing the sample using 5 mL of acetone and 5 mL of ethyl alcohol, transfer of the sample to Gooch's crucible for vacuum filtration [13]. Finally, the sample was placed in the oven at 105°C until constant weight and then weighed (W_1) and placed in the muffle at 550°C for 2 h and again weighed (W_2) to calculate the percentage of crude fiber, according to

$$\text{Crude fiber content (\%)} = \frac{(W_1 - W_2)}{W} \times 100. \quad (3)$$

The total carbohydrate content of the sample was calculated as (4). Its total caloric value was calculated by applying the conversion values for carbohydrates (4 kcal), lipids (9 kcal), and protein (4 kcal) [3, 18].

$$\text{Total crude carbohydrates (\%)} = 100 - (\text{Moisture} + \text{crude protein} + \text{fat} + \text{ash}). \quad (4)$$

2.5. Mineral Composition Characterization. The phosphorus and potassium quantification was performed by the vanadomolybdic method in the spectrophotometer (Hach DR 5000) and flame photometry (Micronal B462), respectively [19]. The calcium, magnesium, copper, iron, manganese, and zinc minerals were determined by acid digestion using the method [20]. After digestion, the minerals were quantified by the Atomic Absorption Spectrophotometric (AAS) technique (Analytik Jena, AA 300).

2.6. Preparation of Hydroethanolic Extracts. The hydroethanolic extracts of the cookies biscuit samples were obtained by the hydroalcoholic extraction method according to Oliveira et al. [21] with some modifications. A sample containing 5 g of CAB flour and cookies with 0%, 5%, 15%, 30%, and 50% of flour were homogenized in 20 mL of 80% ethanol, under constant stirring at 30°C, for 20 min. After this step, the samples were centrifuged at 3500 rpm for 20 min. To the obtained precipitate, 20 mL of 80% ethanol was added to perform a further extraction for 20 min and then centrifugation was performed for 20 min. The same procedure was repeated one more time. All the supernatants were pooled and concentrated using the rotavaporator (BuchiRotavapor R-114) at 40°C and then stored in a glass vessel and kept under refrigeration at 4°C for further analysis.

2.7. Determination of Total Phenolic Content. The concentration of the phenolic compounds of the CAB flour hydroethanolic extracts and cookies with 0%, 5%, 15%, 30%, and 50% of the CAB flour was determined using the Folin-Ciocalteu reagent (FCR) method, as described by Cicco et al. [22] with some modifications. 120 μL aliquots of the extracts were placed in the test tubes with 180 μL of Milli Q water. Then, 300 μL of FCR and, after 2 min, 2.4 mL of 5% (w/v) sodium carbonate were added, totaling a volume of 3 mL. The final concentration within the cuvette was 100 μg/mL of the hydroethanol extracts (80%). The tubes were vortex and kept in the dark in a water bath at a temperature of 40°C for 20 min. Then, a 3 mL aliquot of the sample was placed in a quartz cuvette (capacity = 3 mL, optical path = 1 cm) and the absorbance at 760 nm was measured using a spectrophotometer (Mutispec-1501 model Shimadzu, Japan). The total phenol content was obtained from the calibration curve prepared in a concentration range (1.12–10.53 mg/L) and expressed as mass of gallic acid equivalents per gram of extract (mg GAE/g).

2.8. Activity of 2,2-Diphenyl-b-Picrylhydrazyl Radical (RSA-DPPH$^\bullet$). The determination of the antioxidant capacity of CAB flour hydroethanolic extracts and cookies with 0%, 5%, 15%, 30%, and 50% of CAB flour was made according to Sánchez-Moreno et al. [23] with some modifications. The antioxidant capacity was determined by monitoring the reaction between the DPPH$^\bullet$ free radical and the extracts by measuring the absorbance at 516 nm in a spectrophotometer (UV-vis model Multispec-1501 Shimadzu, Japan). Hydroethanolic solutions of cookies with 0%, 5%, 15%, 30%, and 50% CAB flour were prepared so that their final concentration inside the cuvette was 100 μg/mL. Thus, 0.30 ml of powder extract dissolved in methanol (0.25, 0.5, and 1.0 mg ml^{-1}) was mixed with 2.7 ml of DPPH$^\bullet$ radical solution (40 μg/mL in methanol) in a 3 ml quartz cuvette. The mixture was homogenised using the micropipette pointer (pushing and pulling liquids) and kept in the dark prior to analysis. The DPPH absorption values at 516 nm were obtained every 5 min during a period of 50 min. First, a blank was made with 0.3 mL of sample solution and 2.7 mL of methanol.

The percentage of DPPH$^\bullet$ radical sequestering capacity (% RSA, DPPH$^\bullet$) of each extract was calculated as

$$\%\ \text{RSA} = \frac{1 - AC}{AD} \times 100, \quad (5)$$

where AC is the absorbance of the solution when the extract was added at a particular concentration in 30 min and AD is the absorbance of the DPPH$^\bullet$ solution.

2.9. Preparation of Cookies. Table 1 presents the formulations of the cookies made from the CAB flour varying the proportions. Initially, the wheat and CAB flour were added and homogenized, followed by the addition of the other ingredients. The cookies after molding were baked at 180°C for 25 min. They were then cooled and packed in plastic containers.

2.10. Sensory Analysis. The sensory analysis of the biscuits was performed with 33 people without previous training in the age group of 16 to 59 years, with 18 women and 15 men. Samples were identified in nonrepetitive digits, randomly served on a plate with a glass of water to rinse the mouth before the subsequent test. The samples were evaluated according to colour, flavor, aroma, appearance, texture, and overall acceptance by means of the hedonic scale of 9 points (1 = greatly disagree to 9 = I liked very much), through an evaluation card analyzing the organoleptic properties [24].

2.11. Statistical Analysis. The results obtained on the design of the sensorial acceptability were analyzed statistically using

Table 1: Formulations used to prepare cookies.

Ingredients	Formulation				
	0%	5%	15%	30%	50%
Wheat flour (g)	120.0	114.0	102.0	84.0	60.0
CBA flour (g)	0.0	6.0	18.0	36.0	60.0
Brown sugar (g)	75.0	75.0	75.0	75.0	75.0
Crystal sugar (g)	45.0	45.0	45.0	45.0	45.0
Margarine (g)	50.0	50.0	50.0	50.0	50.0
Yeast (g)	0.5	0.5	0.5	0.5	0.5

Table 2: Characteristics of CBA flour.

Parameter	CBA flour
Acidity (g citric acid/100 g)	0.12 ± 0.00
Vitamin C (% ascorbic acid)	1.31 ± 0.04
pH	5.63 ± 0.05
Soluble solids (°BRIX)	7.5 ± 0.04
Water absorption capacity (g/100 g)	4.36 ± 1.36
Moisture (%)	10.41 ± 1.06
Ash (%)	2.17 ± 0.29
Fat (%)	5.35 ± 0.42
Protein (%)	5.68 ± 0.00
Crude fibre (%)	3.45 ± 0.59
Carbohydrate (%)	76.39 ± 1.77
Calories (Kcal/100 g)	376.43

Values expressed as mean ± standard deviation.

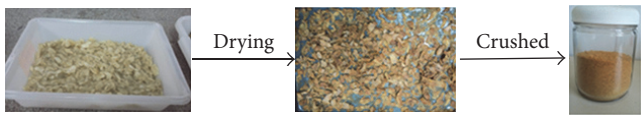

Figure 1: Steps to obtain the CBA flour.

Analysis of Variance (ANOVA) and Tukey test at the 5% level of significance, to compare the means using the Action Stat Pro.

3. Results and Discussion

Figure 1 shows the dehydration and crushing stages to obtain the CAB flour. After the drying process, there was a final yield of 20%, which was caused by the elimination of water and other components.

3.1. Physicochemical and Proximate Composition Characterization of CAB Flour. The values obtained for the physicochemical characterization of the CAB flour are expressed in Table 2.

The pH value found for the CAB flour was 5.63, while the total acidity was 0.12 g citric acid/100 g. The determination of these parameters, mainly the acidity, can provide important data to evaluate the conservation state of the product, since the processes of decomposition by hydrolysis or oxidation can affect the sensory and nutritional characteristics of the product [3]. The value found was lower than that of common wheat flour, minimizing the propagation of bacteria and consequently the deterioration of the product.

The CAB flour presented a solids content of 7.5 °Brix, associated with the presence of sucrose and glucose, providing a bittersweet flavor to the final product [27].

The water absorption capacity was 4.36 g, resulting in values higher than those found for flour produced from soybean, corn, rice, and beans [3, 10, 28]. This property determines the ability of the food to absorb water by the starch granules. In this way, the greater the property, the easier the access of the water between the starch chains, thus improving its application in products such as breads and cakes, due to the maintenance of moisture, increasing the yield of the final products. Moreover, this parameter can be vital in retaining the taste and palatability of food [10].

The moisture content was 10.41%, a value lower than the 15 % allowed for vegetable flours by Brazilian legislation [29]. Low moisture content prolongs the storage time of food and contributes to the textural quality and the inhibition of chemical and biochemical reactions, due to the fact that it minimizes the proliferation of undesirable microorganisms such as fungi and bacteria [3, 30].

The ash content of a food product refers to the inorganic residue remaining from the burning of organic matter, which, according to the Brazilian Government Food Agency legislation, should be at most 4.0% [31]. From the results the low ash content was observed, around 2.17%, associated with the presence of minerals present in the pulp, which were preserved after the dehydration process, showing that the preparation of flour from fruits and vegetables is rich in minerals required for human consumption [32, 33].

Since the fat determination represents the amount of fat present in the food, its value high fat content of any product can lead to rapid degradation process. Consequently, this degradation alters the organoleptic properties and nutritional value, transforming the food into a toxic product due to the formation of free radicals [34]. The fat content found for the CAB flour was 5.35%, being within the standards established by ANVISA [31], and it should present a total fat level of up to 6 g/100 g in the elaboration of any food products processing.

The protein content found for the CAB flour was 5.68%, lower than that established by ANVISA [31], making this flour inadequate for incorporation into food formulations for the purpose of protein enrichment.

The CAB flour had crude fiber content, around 3.5%. According to Sumczynski et al. [35] the main constituents of the crude fiber are composed of cellulose and lignin are

Table 3: Mineral composition of CBA flour.

Mineral	Mineral content (mg·100 g^{-1})	DRI[a] (mg) Adult	% DRI from 100 g of flour
Phosphorus	80	700	11.42
Potassium	500	4700	10.6
Copper	4.0	0.9	444.4
Iron	7.8	8.0	97.5
Manganese	0.71	2.3	30.8
Zinc	8.5	11	77.27
Calcium	189.9	1000	18.99
Magnesium	262.5	420	65.62

[a]Dietary reference intake (DRI) for adults, according to Food and Nutrition Information Center [25].

parts of fruits. Despite this low fiber content, the CAB flour is classified according to ANVISA [31], as a fiber source food, since it had a fiber content higher than 3.0%.

The calorific value of 376.63 Kcal/100 g found for CAB flour meets 18% daily values established by ANVISA [31]. This high caloric concentration is the result of the amount of carbohydrates present in the custard apple, in particular sucrose and glucose, according to the results of the analysis of soluble solids (°BRIX), evidencing the feasibility of the incorporating of this flour in formulations for energy enrichment.

3.2. Mineral Composition Characterization. Knowledge of the mineral composition of food products is fundamental to achieve food and nutritional security, obtaining information and subsidies for public health authorities to establish nutritional goals, quality control, and evaluation of nutrient intake by individuals [36]. The results obtained for mineral composition (Table 3) were quite significant, with emphasis on Cu, Fe, Mn, Zn, Ca, and Mg, serving more than 20% of the daily nutrient intake index [36, 37].

One of the most important minerals in the human diet, which prevents the incidence of anemia, is iron. The deficiency of this mineral occurs when the demand for Fe is high, especially in the growth phase, menstrual cycle, and pregnancy. According to USDA [37] the daily recommendation of iron intake for children and adults of both sexes ranges from 7 mg to 27 mg. The CAB flour had a content of 7.8 mg/100 g, a result superior to foods such as milk (0.02 mg/100 g), oatmeal (2.09 mg/100 g), rice flour (2.7 mg/100 g), wheat flour (0.8 mg/100 g), and corn flour (1.1 mg/100 g) [38, 39]. The National Health Surveillance Agency [31] recommends that wheat and maize flour should be enriched with iron for a final concentration of 4.2 mg/100 g. The incorporation of this flour into baked goods such as breads, cakes, salads, and biscuits may be a viable alternative for iron supplementation, given that this nutrient is the most common nutritional deficiency in developing countries, especially in Brazil [31, 38, 40].

Calcium was one of the main minerals present in CAB flour with 189.8 mg/100 g, contributing with 18.99% of the nutritional recommendation [37]. The amount of calcium found in this work is higher compared to wheat flour (18 mg/100 g), corn flour (34 mg/100 g), rye flour (24 mg/100 g), *Annona crassiflora* flour (117 mg/100 g), and similar to and/or the same as soybean flour (206 mg/100 g) and acerola bagasse flour (264.32 mg/100 g) [41–43]. This mineral is abundant in the human body, contributing about 2% of body weight, is responsible for mineralization of bones and teeth, performing intracellular regulation in most body tissues, muscular contraction, and nervous function [44]. The incorporation of CAB flour in food products may be promising as a source of calcium for lactose intolerant individuals, since milk and its derivatives are considered the best sources of calcium.

Copper and zinc are essential micronutrients for humans and vital components of various enzymes responsible for cellular metabolism [45]. According to the results, the 100 g portion of CAB flour had 4.0 mg Cu, which was higher than that found for wheat flour (1.51 mg/100 g), rice flour (2.2 mg/100 g), maize flour (0.9 mg/100 g), and papaya peel flour (1.09 mg/100 g) [46, 47], while the Zn content in the CAB flour was 8.5 mg/100 g and higher than the *Annona crassiflora* flour (3.41 mg/100 g), wheat flour (7.59 mg/100 g), papaya peel flour (0.23 mg/100 g), apple pomace flour (0.17 mg/100 g), and orange pomace flour (0.23 mg/100 g), demonstrating to be promising for food formulations [41, 42, 46, 48]. The minerals Cu and Zn present in CAB flour account for more than 75% of the daily nutrient intake index (DRI) of nutrients, especially for copper [36, 37].

The potassium content for CAB flour was 500 mg/100 g, contributing with 10.5% of the nutritional recommendation [37]. The potassium content was higher than rice flour (97.4 mg/100 g), wheat flour (150 mg/100 g), and corn flour (148.7 mg/100 g) and lower than quinoa flour (553.8 mg/100 g) [46]. This mineral is very important for the human body, acts as the main enzymatic cofactor maintaining the acid-base balance of the human body, playing an essential role in the functioning of nerves and muscles, and reducing the risk of stroke and coronary heart disease [49].

Magnesium is the essential intracellular cation for the physiological metabolism of the human body, because it intervenes to regulate the activity of more than 300 enzymatic reactions. The CAB flour had a content 262.5 mg/100 g of magnesium, representing 65.62% of the daily nutritional recommendation, a proportion higher than green banana flour (30.84 mg/100 g), wheat flour (8.25 mg/100 g), rice flour (7.3 mg/100 g), bean flour (47.20 mg/100 g), papaya Hawaii flour (210 mg/100 g), banana green flour (30,80 mg/100 g), and *Annona crassiflora* flour (14.23 mg/100 g) [42, 46, 47, 50, 51]. Scientific research has shown that even minimal variations in the concentration of this mineral can deplete certain

Table 4: Total content of phenols and DPPH• radical of the hydroethanolic extracts of the CBA flour and cookies with 0%, 5%, 15%, 30%, and 50% of the CBA flour and fruit residues flours as reference.

Samples	Yield (%)	TPC mg EAG/g dry extract	TPC mg EAG/100 g flour	DPPH• RSA%
CAB flour	84.62	7.78 ± 0.33^{ab}	$658.\ 34 \pm 27.92^{a}$	9.68 ± 0.91^{a}
0% CAB flour	45.49	5.44 ± 2.73^{ab}	247.46 ± 124.18^{bc}	10.75 ± 0.91^{a}
5% CAB flour	44.28	4.50 ± 1.36^{ab}	199.26 ± 60.22^{c}	9.91 ± 1.33^{a}
15% CAB flour	54.00	3.73 ± 0.01^{b}	201.42 ± 0.54^{c}	10.43 ± 1.85^{a}
30% CAB flour	46.11	4.93 ± 0.79^{ab}	227.32 ± 36.42^{c}	10.46 ± 0.14^{a}
50% CAB flour	54.86	9.35 ± 3.56^{a}	512.94 ± 195.3^{ab}	9.88 ± 3.63^{a}
Reference samples				
Wheat flour[a]	nd	6.96 ± 0.1	nd	nd
Quinoa flour[a]	nd	2.81 ± 0.1	nd	nd
Acerola bagasse flour[b]	nd	10.82 ± 0.09	nd	nd
Residue pequi flour[c]	nd	17.42 ± 0.53	nd	nd
Residue passion fruit flour[d]	2.5	41,2 ± 4,2	103 ± 10.4	24,7
Residue pineapple flour[d]	30.2	9.1 ± 1,3	275 ± 38.0	19,8
Residue acerola flour[d]	7.1	94.6 ± 7.4	681 ± 53.5	81.6

[a]Chlopicka et al., [26]. [b]Marques et al., 2013. [c]Leão et al., 2017. [d]Oliveira et al., 2009. nd = not determined. Averages followed by the identical letters in the column do not differ statistically by the Tukey test ($p < 0.05$).

cardiac arrhythmias, affecting cell metabolism, growth, and proliferation [52].

Manganese plays an important role in the development of bones and cartilage, as well as in the healing of wounds and constituents of various enzymes [53]. The CAB flour presented 0.71 mg/100 g constituting 30% of the daily diet of this mineral, inferior index to that found in the *Annona crassiflora* flour (1.85 mg/100 g), but superior to banana flour (0.14 mg/100 g) and wheat flour (0.43 mg/100 g), demonstrating its potential in food formulations [50, 54].

Phosphorus is a mineral present in all cells of the human body playing important role in protein synthesis, growth, maintenance, and repair of cells and tissues. The CAB flour had 11.42% of the daily intake index with 80 mg/100 g, a result inferior to that found in wheat flour (90.8 mg/100 g), rice (95.4 mg/100 g), and maize flour (81.3 mg/100 g) and higher than the *Annona crassiflora* flour (62.09 mg/100 g) [42, 46]. The values of minerals found make the incorporation of CAB flour as supplementation and nutrient supplementation in food products promising, such as wheat cereal flours, used in bread products, which are generally deficient in minerals essential for human nutrition [10].

3.3. Total Phenolic Content and Antioxidant Capacity DPPH• Radical. Table 4 shows the yields obtained from the hydroethanol extracts, the total phenolic content (TPC), DPPH• antioxidant activity of the CAB flour of the 0%, 5%, 15%, 30%, and 50% CAB flour, and samples of references of other fruits as a comparison parameter. The CAB flour presented an extraction efficiency compared to the cookies, associated with the higher fiber content present. Oliveira et al. [21] evaluated methanolic extraction for pineapple, acerola, and passion fruit flours, with a higher extract yield for pineapple flour, attributing this characteristic to the higher fiber content in relation to other fruits.

According to Table 4, the TPC for extracts of CAB flour and cookies 0%, 5%, 15%, 30%, and 50% of CAB flour varied from 3.73 to 9.35 mg GAE/g. The samples of CAB flour and the cookie with 50% of flour presented higher concentrations of phenolic compounds in relation to the other cookies produced because of the superior amount of fiber. The value of phenolic content found for CAB flour showed superiority and/or similarity when compared to wheat, quinoa, and pineapple flour but obtained values lower than flours obtained from the residues of pequi, passion fruit, and mainly acerola in dry residue extract. However, when considering the extraction yield and the phenolic concentration converted to 100 g of CAB flour, a superior result was obtained in relation to passion fruit flour and similar to acerola flour. According to Oliveira et al. [21], this characteristic is essential in the dietary prescription of flours, since the CBA flour obtained greater extraction of phenols, being the most recommended to the detriment of the fruit flours shown in Table 4. In this way, CAB flour presents a significant amount of phenols being able to act as natural antioxidants capable of reducing degenerative diseases such as arteriosclerosis, cardiovascular disease, and cancer [10].

The results observed for the sequestering activity of the DPPH• radical of the hydroethanol extracts showed a variation of 9.68–10.75%, showing no significant difference between the samples. However, when comparing the results of CAB flour and cookie formulations, a lower result was obtained in comparison to the flour of other fruits, according to Table 4. These results can be related to the part of the fruit residue that is being reused for elaboration of the flour. Leão et al. [55] report that phenolic compounds are associated with

Table 5: Evaluation of the tasting tests of cookie with different formulations.

Parameter	Formulation, age range (27 to 59 years)				
	0%	5%	15%	30%	50%
Appearance	6.4 ± 2,06[a]	7.1 ± 1,40[a]	7.2 ± 1,52[a]	6.9 ± 1,86[a]	7.4 ± 1,30[a]
Colour	6.8 ± 1,78[a]	7.2 ± 1,94[a]	7.2 ± 2,04[a]	7.4 ± 1,35[a]	7.8 ± 1,37[a]
Texture	6.1 ± 2,38[a]	6.4 ± 2,16[a]	6.2 ± 2,05[a]	5.7 ± 1,57[a]	6.0 ± 2,67[a]
Aroma	6.5 ± 1,68[a]	6.5 ± 1,84[a]	6.9 ± 1,48[a]	6.5 ± 1,95[a]	7.0 ± 2,07[a]
Flavor	6.7 ± 1,86[a]	6.9 ± 1,70[a]	6.7 ± 1,90[a]	6.6 ± 1,80[a]	6.7 ± 2,21[a]
Overall acceptance	6.2 ± 2,06[a]	6.5 ± 1,68[a]	6.6 ± 1,80[a]	6.6 ± 1,58[a]	6.3 ± 2,35[a]
Parameter	Formulation, age range (16 to 24 years)				
	0%	5%	15%	30%	50%
Appearance	5.3 ± 1,57[ab]	6.3 ± 1,74[a]	5.5 ± 1,72[ab]	5.6 ± 2,11[ab]	5.8 ± 2,12[ab]
Colour	5.5 ± 2,17[ab]	6.3 ± 1,81[a]	6.4 ± 1,82[a]	6.1 ± 2,11[ab]	6.4 ± 2,43[a]
Texture	4.4 ± 2,25[b]	6.2 ± 2,02[a]	5.5 ± 2,54[ab]	4.0 ± 2,54[b]	4.1 ± 2,32[b]
Aroma	6.0 ± 2,09[ab]	6.3 ± 1,88[a]	6.2 ± 2,16[ab]	6.0 ± 2,23[ab]	5.7 ± 2,56[ab]
Flavor	5.7 ± 2,29[ab]	6.5 ± 2,33[a]	6.5 ± 2,03[a]	5.2 ± 2,55[ab]	4.1 ± 2,42[b]
Overall acceptance	5.8 ± 1,74[ab]	6.6 ± 2,08[a]	6.2 ± 2,15[ab]	5.0 ± 1,95[b]	4.6 ± 2,11[b]

Results expressed as mean ± standard deviation; 9-point hedonic scale (1 = greatly disagreed to 9 = greatly liked). Averages followed by the identical letters in the column do not differ statistically by the Tukey test ($p < 0.05$).

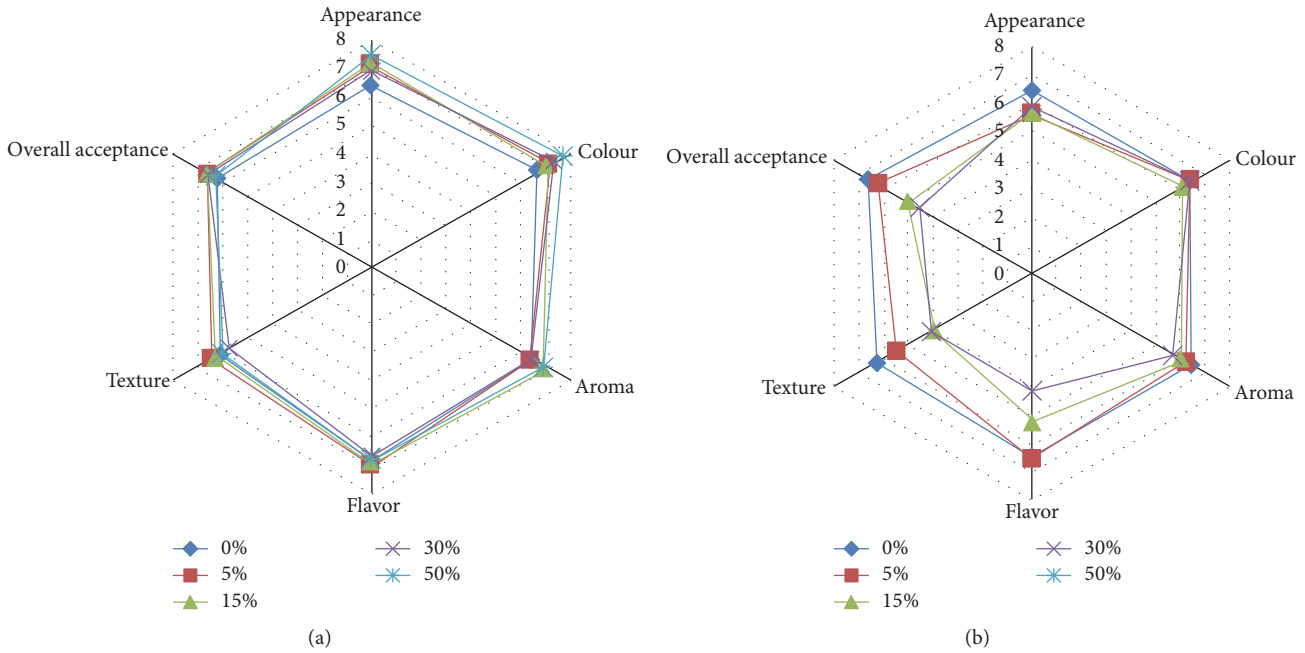

Figure 2: Graphical representation for organoleptic properties: age range of 27 to 59 years and (b) age range of 16 to 24 years.

important functions in plants, including pigmentation and defense, as well as in fruit peels and seeds and to a lesser extent in pulp, justifying the low DPPH• value found for the CAB flour and cookies made from the bagasse pulp.

3.4. Sensory Analysis of Cookies. Table 5 presents the evaluations of the organoleptic properties in relation to the age range of the tasters.

The results of the acceptability tests showed that the age group was a preponderant factor during the evaluation of cookies as observed in Figure 2. According to Pineli et al. [43] the process of consumers choice and perception of food is multifactorial, associated with several nonsensory aspects such as brand, familiarity with product, and price.

Consumers in the age group (a) presented higher scores on all organoleptic properties and on the overall evaluation regardless of the proposed formulation. Despite the 50% substitution of wheat flour, there was no decay in the acceptability of the consumers, and, for most of the parameters evaluated, preference was higher compared to the sample containing only wheat flour, allowing the nutritional increase in the proposed formulations. On the other hand, for consumers in the age group (b), there was a decrease in the evaluations, but, for the 5% and 15% formulations, the testers stated

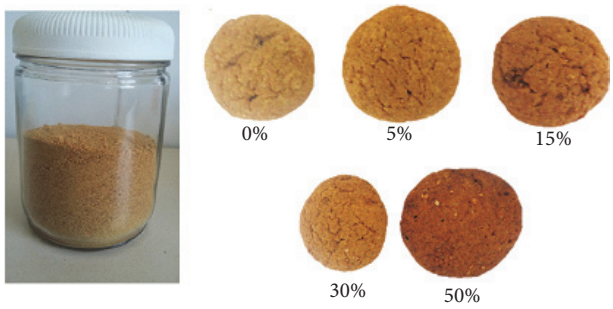

Figure 3: CAB flour and cookies with different formulations.

that they liked it slightly (score 6.0), presenting a higher acceptability index than the sample containing only wheat flour. These results demonstrate that cookies made from the CAB flour presented similar acceptability to the cookies of commercial wheat flour, grape marc, banana, rice, and tapioca [50, 56, 57] before the evaluators, highlighting the age group A and making it promising in the supplementation and incorporation in food formulations improving the nutritional properties. Cookies made with different proportions of CAB flour are shown in Figure 3.

4. Conclusion

The physicochemical and proximate characteristics of the CAB flour presented values and specifications for elaboration of food products, besides containing high concentration of phenolic components. The incorporation of the CAB flour in the formulation of cookies showed good acceptance before the consumers, presenting, in some formulations, preference greater when compared to the cookie elaborated with 100% of wheat flour. The results indicate that flour made from custard apple bagasse pulp can be incorporated in food formulations to improve nutritional properties, being an alternative to add value to the residue and to minimize losses along the production chain of custard apple.

Conflicts of Interest

This paper did not lead to any conflicts of interest regarding the publication of this manuscript.

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