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Aroma retention during drying of caja-umbu fruit pulp

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Abstract

This study was aimed to obtain and characterize the dried powder of cajá-umbu (Spondias spp) fruit pulp obtained by spray-drying and lyophilization. Spray-drying of the pulp was done at different temperatures. Analysis of bioactive compounds and volatile compounds was performed. The total phenolic compounds content was high in the dried powder obtained at the temperature of 140 °C. The volatiles analysis of dried powders revealed that the powder dried at 140°C contained a larger number of compounds. The cajá-umbu powder showed that it is a better alternative for storage and conservation since it retained the majority of volatile compounds.

Keywords: *Cajá-umbu, volatile compounds, gas chromatography, mass spectrometry*.

1. Introduction

Aroma is an important characteristic in exotic tropical fruits, which has increasingly attracted consumers due to their peculiar sensorial attributes. Brazil is one of the countries that have a large variety of these fruits, which are usually produced widely and are mostly consumed in fresh form or in the form of juices, ice cream, jams and jellies. Among the large variety of tropical fruits grown mainly in the Brazilian northeast region, fruits belonging to the genus *Spondias*, involving umbu (*Spondias tuberosa* Arruda Camara), yellow mombin (*Spondias mombin* L.), cajá-umbu (*Spondias spp)*, purple mombin (*Spondias purpurea* L.) and amberella (*Spondias dulcis* L. syn. *Spondias cytherea* Sonn.) are prominent. The volatile composition of these fruits pertain to several chemical classes, such as tannins, terpenoids (sesquiterpenes and monoterpenes), flavonoids, etc.[1,2]

The cajá-umbu (*Spondias* spp.) owing to its particular sensorial characteristics, such as aroma and flavor, is a fruit which is very much appreciated in the northeastern region of Brazil. It is native to the semi-arid regions and is cultivated between the states of Rio Grande do Norte, Ceará, Piauí, Pernambuco and Bahia. Its sensorial characteristics possessing strong acidic exotic aroma and attractive color, make this fruit to be highly demanding in the regions of the country where it is found.[3,4,5]

Being a climacteric fruit, the shelf life of cajá-umbu fruit is low and thus it is commercialized in the near by places where it is produced. It is therefore a great challenge to transport and commercialize this fruit to places far from its cultivation. In order to preserve this fruit and to develop new products with increased commercial value, dehydration techniques such as freeze drying and spray drying can be used.

To the best of the author´s knowledge no studies have been performed until the present moment on drying of cajá-umbu fruit and also to identify volatile and bioactive compounds in cajá-umbu dehydrated by these two techniques of drying. Thus the objective of this study was to evaluate the effect of two drying processes - freeze-drying and spray-drying of cajá-umbu pulp (*Spondias* spp) on the retention of volatile and bioactive compounds.

2. Materials and Methods

2.1. Fruit purchase and pulp extraction

The ripe mature fruits of cajá-umbu were acquired from the Center of Supply (CEASA) of the city of Aracaju/SE. These were then taken to the Laboratory of Processing of Vegetable Origin, located in the Department of Food Technology at the Federal University of Sergipe (UFS). The selected fruits were submitted to selection, sanitization pulping and packaging in polyethylene packages.

2.2. Spray Drying

For the Spray dryer process, experiments were performed by varying the drying air temperature (100, 120, 140 and 160°C) while maltodextrin concentration was maintained at 15%. The atomization was performed in a spray dryer with a nozzle atomization system of the brand LABMAQ (model MSDi 1.0, Brazil) with injector nozzle having 1.2 mm diameter orifice, air flow of 4.00 m³/min and pressure of air of 4 kgf/cm². The spray dryer was fed through a peristaltic pump, with rotation speed adjusted as a function of the maximum speed; this flow being 0.52 L/h. The dried powder was stored in glass containers with a screw cap, then sealed and stored in a desiccator at room temperature (25 ± 2 ^{0}C).

2.3. Lyophilization

The drying of pulp was also performed in a Christ Freeze Dryer, (model Alpha 1-4 LSC, Germany). The pressure used was 0.42 mbar, at a sublimation temperature within the chamber was - 58°C.

2.4. Separation and Identification of Volatile Compounds

The volatile compounds were captured through the purge and trap system (Tekmar Mark, Model ATOMX), using the Vocarb3000 trap and analyzed in the gas chromatography system coupled to a mass spectrometer (GC- QqQ MS, Agilent 7000) according to the method with adaptations reported by Narain et al.[5] The volatile compounds were analyzed in GC-MS system using a Carbowax column (30 m x 0.25 mm x 0.25 µm). The oven temperature was programmed: initiation at 35°C (held for 2 min) then raised at a rate of 10°C to 100°C (held for 1 min) and finally increased at 3°C/min to 250°C. Volatile compounds were tentatively or positively identified by comparing their mass spectra with the compounds from NIST (National Institute of Standards & Technology) database and comparing the linear retention index (LRI) of the standards and compounds with those of literature publications and other databases (Flavornet, PubChem, Pherobase).

3. Results and Discussion

3.1. Volatile compounds of dehydrated powder

Dehydration reduces the moisture content and therefore can result in the loss of volatile compounds in the final product at the end of the process.[6] Studies on the influence of temperature are very important to guarantee the quality of the final dehydrated product.[7] When developing a powder obtained by spray drying or lyophilization it is necessary to always standardize the best temperature where a powder can be obtained with the aroma very close to that of the fresh fruit. Thus, it is important to study the volatile composition of the product obtained by dehydration at various temperatures in order to define the best condition for obtaining an aromatic product at the end of the process.

In the Table 1 presents the data on number of peaks obtained on the analysis of dried powders obtained at different temperatures used for spray drying and by lyophilization. The volatiles analysis of dried powders revealed that the spray dried product at 140°C contained higher number (29) of compounds, compared to lyophilization which had only 26 compounds.

Table 1: Number of volatile compounds found in various dried powders obtained by spray and freeze drying of cajá-umbu pulp.

Dehydration type	Temperature	Number of peaks
Spray-drying	100°C	18
Spray-drying	120°C	20
Spray-drying	140°C	29
Spray-drying	160°C	19
Lyophilization	-58°C	26

The most representative class of compounds for the dried powders obtained by atomization were the terpenes, and the compound with the highest concentration was limonene at all the operating temperatures of the Spray-dryer (Table 2). Other terpenes identified were α-pinene, cis-α-bisabolene, 3-carene, β-pinene, *m*-xylene. Narain et al.[5] reported the presence of these compounds in ripe cajá-umbu (*Spondias* sp) fruit pulp when the extraction was performed by the Purge & Trap extraction technique and compounds were separated by using Carbo-WAX column and identified in a GC-MS system.

Tong Chin et al.[8] studied the influence of the temperature on the retention capacity of the volatile compounds markers of the atomized durian pulp and monitored the propanethiol and ethyl propanoate compounds at two temperatures of 170°C and 130°C, and reported that at a temperature of 170°C a reduction in the number of volatile compounds occurred. In the present work as seen in Table 1, the temperature at which a higher number of peaks was obtained and also a higher intensity was observed was at 140°C. At this temperature it is also possible to detect the appearance of compounds which at other temperatures were not found, for example, 3-carene, *m*-xylene while there was an increase in the concentration of the esters such as ethyl acetate, ethyl butanoate and ethyl lactate.

The use of high temperatures can also lead to the formation of new compounds in dehydrated products. Gozales-Palomare et al.[9] working with dehydrated powder of *Hibiscus sabdariffa* at temperatures of 150, 160, 170, 180, 190, 200 and 210°C reported the formation of furfuraldehyde in the dehydrated product at temperatures of 180-210°C. Similar results were found for dehydrated cajá-umbu powders at temperatures of 140 and 160°C, being higher in 160°C. According to Nunes et al.[10], the presence of furfural in ODG is probably expected as heating increases ascorbic acid degradation. Therefore, furfural analysis could be used to monitor product quality during guava drying processes as it may be considered as a marker of heating.

Table 2: Volatile compounds present in dried powder of cajá-umbu pulp

Compound	LRI_{Lit}	Dried powder-Spray drying				FDP
		100°C	160°C	140°C	120°C	
Ethanal	724	0.322	3.007		2.188	
Pentanal	935		0.544	0.674		
Verbenone				1.837		
Furfural	1455		2.64	0.415		
Acetone		1.966	1.727	2.396	2.524	
3-Methyl-4-propenyl-oxetan-2-one			0.21		0.129	
Ethyl acetate	907	1.841	1.104	2.181	2.007	
Ethyl lactate						16.964
Ethyl butanoate	1037	1.92		0.695	0.431	
Prenylacetate		0.696		0.586	0.38	
Propyl butanoate						8.432
Ethyl isovalerate	1100					1.039
Ethyl octanoate	1436					0.265
Pyrrole	915					6.248
m-xylene	1150			0.461		
3,4-Dimethyl styrene		0.145		0.606	0.129	
Ethanol	932	4.212	3.95	9.914	3.919	
δ-Octalactone						2.054
δ-Nonalactone						1.239
3-Aminobutanoic acid						4.627
α-Pinene	1030	0.644	0.577	3.934	0.604	
1R-α-Pinene	1055					6.22
(*Z*) Hydrated sabinene	1123					0.354
3-Carene	1148			0.116		0.237
1,3,8-*p*-Menthatriene	1158		0.082	1.606		
β-Pinene	1138	0.129	0.114	0.843	0.519	
α-Terpinene	1178					5.001
Limonene	1177	18.92	16.759	15.797	19.37	
(+)-Limonene	1201					18.714
Cyclofenchene						4.628
cis-β-Ocimene	1234			0.463		
Ocimene	1225					4.852
o-Cymene	1261	0.322	0.49	1.277	0.355	
(*E*)-β-Ocimene	1242					1.557
Copaene	1488					0.68
Alloaromadendrene	1599	17.35	18.704	10.537	16.67	
Isocaryophyllene	1570					2.588
cis-α-bisabolene	1543	1.317	1.789	0.824	1.319	
(3*Z*)-2,7-Dimethyl-3-octen-5-yne		0.309	0.207	0.955	0.27	

* LRI - Linear Retention Index; FDP - Freezed dried powder

An interesting factor to be emphasized is the higher presence of esters in the cajá-umbu pulp powder obtained by freeze-drying than in powder obtained by spray-drying. Compounds such as ethyl lactate (16.96%), which possesses a mild odor and propyl butanoate (8.432%) characterized by a fruity odor, were found in significant concentrations. Tietel et al.[11] studying the changes in concentrations of volatile compounds of tangerine reported that a slight increase in temperature caused a decrease in the concentration of esters such as ethyl acetate, ethyl butanoate, ethyl hexanoate. Similar behavior was observed in this study when compared to the lyophilization technique that uses low temperatures, when compared with the spray-drying technique that uses high temperatures.

Another aspect that probably influenced a lower retention of esters in the spray-drying was the retention capacity of the wall material in relation to these compounds. Rosenberg et al.[12] studying the factors that affect ester retention in the spray-drying technique observed that these compounds are not readily retained in the carrier material with a concentration of less than 30%. For the dehydrated cajá-umbu pulp, a similar behavior was observed to that reported by Rosenberg et. al.[12] since the carrier material (maltodextrin) concentration was 15%, thus not resulting in a better retention of the esters.

4. Conclusions

The pulp dehydration in the spray dryer maintained the nutritional and organoleptic characteristics of the fresh pulp resulting in a promising product for marketing purposes. In the identification of the volatile composition of the dried powders, 29 compounds were identified in the spray-drying process at 140°C and 26 compounds in the dehydrated powder by lyophilization. However, the major compound identified was limonene in both dehydration techniques. At all temperatures (100, 120, 140 and 160°C) of dehydration tested by spray drying, the highest number of volatile compounds were identified in the dried powder at a temperature of 140°C. Thus, it is concluded that the optimum dehydration temperature of the cajá-umbu pulp by the spray-drying process is 140°C.

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