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Evaluation of performance of maltodextrin and gum Arabic usage on volatiles profile of Spray-dried powders of sapota (*Manilkara zapota*) fruit

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Abstract

This work has an objective to analyse and quantify the volatile compounds content in the dried powder of sapota using two encapsulating agents, maltodextrin or gum Arabic, and a mix of these agents. The major compounds found were ethyl acetate and 1-hexanol. Regarding the concentrations of these compounds present in the atomized samples, it can be verified that for both ethyl acetate and 1-hexanol their concentrations were higher in rehydrated gum Arabic dried powder (SG) sample, being 1237.15 µg/kg and 588.08 µg/kg, respectively, compared to the other two samples, rehydrated maltodextrin dried powder (SM) and rehydrated powder containing both encapsulating agents (SMG). The results of PCA analysis showed that the first two main components PC1 and PC2 were able to explain 80.90% of the total variation of the data, of which the main component PC1 explained 59.77% and the component PC2, 21.13%. %. From the heat map the top cluster includes the samples SM e SMG, while the bottom cluster contains the samples sapota juice and SG. Therefore, the sample SG presented greater similarity with the sample of fresh sapota juice as to the composition of volatile compounds.

Keywords: sapota juice; spray drying; volatile compounds; gas chromatography-mass spectrometry.

Practical Application: This research work establishes development of the dehydrated powder of Sapota juice and to study the effect of drying process on volatile compounds using maltodextrin and gum Arabic encapsulating agents. The volatiles capture was performed using the Stir Bar Sorptive Extraction (SBSE) technique and no work has been performed using this technique earlier. It contributes significantly to the generation of new data in relation to the development and characterization of spray dried powders obtained from Sapota. This work is highly relevant not only from volatile compounds but also in its potential for commercial applications of dehydrated powder of Sapota.

1 Introduction

The sapota (*Manilkara zapota*), a fruit belonging to the Sapotaceae family, despite being a fruit originating in Mexico, is the sixth most important fruit in India, found throughout the subcontinent of the country (Ghani, 2003; National Horticulture Board, 2021). Since it has a sweet and soft pulp with a granular texture, sapota is a very popular fruit (Siddiqui et al., 2014). Another important sensory characteristic of this fruit is the aroma, which is sweet and slightly astringent (Oliveira et al., 2011). In nutritional terms this fruit is a good source of dietary fiber, rich in calories providing about 83 cal per 100 g, in addition to being a source of a wide variety of vitamins A, C, niacin, folic acid and minerals such as iron, potassium and copper (Antala et al., 2021). In Brazil, sapota can be found in abundance in the northeast region of the country, where it is most consumed fresh. As it has a high water activity, the shelf life of sapota fruits decreases after ripening, thus favoring microbial growth, and hence its commercialization and production becomes very limited. Since the amount of water present in fruits is a large problem with regard to the preservation of this fruit, dehydration proves to be a very viable method for removing water, which consequently increases the shelf life of the fruit (Jangam et al., 2008).

There are some publications applying dehydration processes to sapota fruit and fruits belonging to the same family (Bala et al., 2017; Ganjyal et al., 2003; Jangam et al., 2008; Lasekan & Yap, 2018; Chong & Wong, 2015; Kumar et al., 2018). For the production of a dehydrated product, a spray dryer is widely used which is an economical process, since it is possible to transform liquid food directly into powder (Schweiggert et al., 2008; Ahmed et al., 2010; Ray et al., 2016; Shishir & Chen, 2017). One of the most important factors in this drying process is the carrier agent, used in conjunction with raw materials having high sugar content in fruits. There are several encapsulating agents, among them, maltodextrin is widely used, as it has advantages such as being colorless, high solubility, low hygroscopicity and low cost (Phisut, 2012; Rezende et al., 2018; Shishir & Chen, 2017). Another widely used encapsulating agent is gum Arabic, and its main characteristics are its solubility, low viscosity and emulsifying properties (Silva et al., 2013).

Fruits are rich in flavor and aroma and, the volatile compounds responsible for this, are produced by metabolic pathways during maturation, harvest, post-harvest and storage of fruits. Thus, the aroma and flavor arouse great interest from

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researchers in determining the volatile composition of foods, thus discovering which volatile compounds characterize a flavor and aroma (Ferreira et al., 2009). There are a few publications which reported volatiles compounds in sapota (Monteiro et al., 2018; Uekane et al., 2017; Lasekan & Yap, 2018).

Uekane et al. (2017) analyzed the volatile fraction of three fruits native to the Brazilian Amazon region: murici (*Byrsonima crassifolia* L., Malpighiaceae), bacuri (*Platonia insignis* M., Clusiaceae) e sapota (*Manilkara sapota* L.) and showed that most of the compounds identified were esters (33%), alcohols (27%), terpenoids (18%) and ketones (9%) and approximately 12% were aldehydes, carboxylic acids and hydrocarbons. The most abundant compounds found were acetic acid, palmitic acid, 3-methyl-3-buten-1-ol and geranylacetone. Lasekan & Yap (2018) characterized the aromatic compounds in fresh and dry sapota (*Manikara zapota*, L.) and showed that the overall aroma note of the fresh fruit was mainly caused by ethyl benzoate, (E)-2-hexenal, and β caryophyllene.

In reviewing all publications, it is possible to verify that research papers involving drying techniques, such as spray drying, using the sapota fruit, as well as the study of the volatiles in their dehydrated products are still limited, and hence there is a need for further studies of the volatiles responsible for the aroma of these dried products from the sapota. Thus, the present work aimed to analyze and quantify the volatile compounds present in the dehydrated powder of fruit obtained by spray-drying of sapota, using 2 different encapsulating agents (maltodextrin and gum Arabic), the performance of which was also compared. Moreover, Stir Bar Sorptive Extraction (SBSE) technique was applied for the first time in this work for capture of volatile compounds.

2 Materials and methods

2.1 Materials and reagents

The ethyl acetate standard was obtained from Sigma-Aldrich (St. Louis Missouri, United States), C8 – C40 n-alkanes standard were obtained from Sigma-Aldrich (St. Louis Missouri, United States). The encapsulating agent maltodextrin (DE 20) was purchased from Cargil Company, Brazil and the encapsulating agent Gum Arabic was purchased from Dinâmica Company, Brazil.

2.2 Sample preparation

The sapota (*Manilkara zapota*) fruits used in this work were purchased from the Central Market in the city of Aracaju, Sergipe, Brazil. The fruits were selected in ripened stage of its maturity and later taken to the Laboratory of Flavor and Chromatographic Analysis (LAF) of the Federal University of Sergipe (UFS), São Cristóvão, Brazil. The preparation of the sample was carried out following methodology described by Araujo et al. (2021). The fruits were washed in tap water, followed by separating the pulp from the peel and seed. The pulp was homogenized in a Waring blender (Deluxe 702 A, Brazil), and later sieved to remove the fibers and the juice stored at -18 °C.

2.3 Atomization process

The parameters for the atomization process of the sapota pulp were used in this study according to Araujo et al. (2021). For the drying process, the pulp was homogenized and filtered to obtain the juice that was used in the spray-drying process. The percentages of encapsulating agents used were based on the methodologies described by Chong & Wong (2015) (30%) and Araujo et al. (2021) (1:1), Fernandes et al. (2014) (1:1). The encapsulating materials were added to the juice in the following proportions: maltodextrin/juice (30:70 w/v); gum Arabic/juice (30:70 w/v); gum Arabic/maltodextrin/juice (15:15:70 w/w/v). The tests were carried out in a spray-dryer with spray nozzle system (LABMAQ Spray Dryer, Model MSDi 1.0). An injector nozzle with 1.2 mm diameter orifice was used, while airflow in the spray dryer was maintained at 4.00 m^3/min and air pressure of 4 kgf/cm^2. The dryer was fed through a peristaltic pump, with a rotation speed of 0.44 L/h, inlet and outlet temperature of 140 °C and 65 °C, respectively. For all the analyses, the dried powder was rehydrated to reach the °Brix corresponding to the juice with addition of maltodextrin (35.81 °Brix), gum Arabic (34.88 °Brix) and the mixture of these two encapsulating agents (35.31 °Brix) before drying process. The dried samples were stored in amber colored glass bottles, duly protected from light and the bottles were stored in a desiccator.

2.4 Extraction of volatile compounds by Stir Bar Sorptive Extraction (SBSE)

The volatiles of sapota juice and rehydrated dried powders were extracted by SBSE following the methodology recommended by Leite et al. (2018). In a 20 mL vial were added 10 g of rehydrated dried powder, 3 g of NaCl and a 10 mm PDMS twister (Gerstel, Mülheim an der Ruhr, Germany), and it was continuously stirred for 30 min at room temperature. After this extraction the twister was removed from the sample, washed with distilled water and dried with soft paper. The extraction was carried out in triplicate for each sample.

2.5 Analysis of volatile compounds by GC–MS

The extracted compounds were desorbed on a Thermal Desorption Unit (TDU) (Gerstel) equipped with a cold injection system (CIS) (Gerstel, Mülheim an der Ruhr, Germany), which preserves and concentrates the volatile compounds of the samples. The TDU temperature was programmed from 35 °C to 210 °C (10 min) at a rate of 60 °C/min, whereby the analytes were desorbed for 4 min and concentrated in the CIS-4, programmed from -35 °C to 260 °C at 10 °C/s to transfer volatiles onto the analytical column. The TDU was operated in a splitless desorption mode; the CIS-4 operated in PTV solvent vent mode and other conditions were: purge flow to split vent of 30 mL/min, vent 60 mL/min and pressure 16.40 psi. Separation and identification of the compounds was performed on a gas chromatograph (Agilent Model 7890B) coupled to a mass spectrometer (Agilent Model 5977 A MSD). The GC was equipped with a DB-5 MS capillary column (30 m × 0.25 mm. × 0.25 µm, Agilent). Helium was used as the carrier gas at a flow rate of 1.0 mL/min. The initial oven temperature was 35 °C (maintained for 4 min), then temperature

was raised at 5 °C/min up to 250 °C. Volatile compounds were tentatively or positively identified by comparing the mass spectra of the sample compounds with the NIST (National Institute of Standards & Technology) database and comparing the linear retention index (LRI) of the standards and compounds with those of literature articles and other online databases viz. Flavornet, Pherobase and PubChem.

2.6 Quantification of volatile compounds

Quantification of all identified compounds was based on a method described by Xiao et al. (2015) and Xin et al. (2022). A volume of 100 µL of the deuterated standard (D8 ethyl acetate; Sigma-Aldrich at a concentration of 10µg/mL) was added to the samples before the extraction procedure. A response factor equal to 1.00 was considered between the relative peak areas of each compound and the peak areas of the deuterated standard.

2.7 Chemometric analysis

Principal Components Analysis (PCA), Hierarchical Clustering Analysis (HCA) and Heat map visualization were performed by using XLSTAT software Trial Version 2020 (Addinsoft Inc., New York, USA). PCA reduces the extensive chromatographic data matrix based on Pearson's correlation coefficients ($p \leq 0.05$), representing efficiently in graphical form. HCA groups the data by similarities and dissimilarities, and it was performed according to Euclidean distance calculated based on the concentration of each volatile compound.

3 Results and discussion

After the drying process, the powders obtained viz. SG Rehydrated dried powder of sapota juice with gum Arabic); SM (Rehydrated dried powder of sapota juice with maltodextrin); SMG (Rehydrated dried powder of sapota juice with maltodextrin and gum Arabic) had a moisture content of 4.37, 4.18 and 3.16%, respectively and were then submitted to analysis of volatile compounds.

3.1 Analysis of volatile compounds

The volatile compounds found in the sapota juice and in the rehydrated powders belong to the chemical classes of alcohols (22.4% of the identified compounds), aldehydes (20.6%), esters (25.8%), ketones (10.3%), terpenes (17.2%) and lactones (1.7%). The alcohols was the main class which had a large number (58) of volatile compounds identified in the samples; 34 compounds were in sapota juice; 27 compounds in SG; 26 compounds in SM; 27 compounds in SMG. The identified compounds, their respective retention indices and mean concentrations present in each sample are shown in Table 1.

In this work the presence of volatile compounds in sapota juice and in rehydrated powders obtained through the atomization process was studied. What was observed is that most of the aroma compounds identified in sapota juice were also found in samples of rehydrated powders, but only 14 compounds were identified in sapota juice, and some of these were lost during the drying process, as can be seen in Table 1.

Regarding the total concentration of each chemical class of volatile compounds (Table 1), it is possible to note that for the class of alcohols, aldehydes, esters and ketones, there was a reduction in concentration if we compare the sapota juice sample with that of rehydrated powders (SM, SG, SMG). This reduction can be explained, because during the atomization process, due to the hot air flow that is used in the process, a small quantity of solids is retained in the drying chamber (Forero et al., 2015). Each encapsulating agent behaved differently in relation to the preservation of the total concentration by classes of compounds. For the class of alcohols and ethers, for example, the concentrations that was before in the sapota juice sample, were 9472.1 µg/kg of pulp for alcohols and 11656.58 µg/kg of pulp for esters while after the drying process, the sample of rehydrated powder containing gum Arabic (SG) had a concentration of 1495.12 µg/kg of pulp for alcohols and 1956.16 µg/kg of pulp for esters. However, this didn't happen with other samples containing maltodextrin (SM) and a mixture of the two encapsulating agents (SMG) thus showing the efficiency of gum Arabic as an encapsulating agent in retaining the volatile compounds.

The initial total concentrations in the sapota juice for the aldehydes and terpenes were 606.46 µg/kg of pulp and 392.14 µg/kg of pulp, respectively (Table 1). The sample of rehydrated powder containing maltodextrin (SM) showed a greater efficiency in retention of volatile compounds, when compared with other samples, obtaining the total concentrations of 345.84 µg/kg of pulp for the aldehyde class and 80.18 µg/kg of pulp for the terpenes. For the ketone class, the rehydrated powder sample containing a mixture of the two encapsulating agents (SMG) stood out from other samples with a major decrease in these compounds, the values being 125.00 µg/kg of pulp while its content was 244.21 µg/kg of pulp in the sapota juice sample. In this work, only one compound belonging to the lactone class, butyrolactone was identified, which was only found in the SM sample but it was in very low concentration (14.63 µg/kg of pulp).

Although samples containing encapsulating agents have proved to be efficient in relation to the retention of the total concentration of various chemical classes, it is possible to observe (Table 1) that in relation to aroma compounds belonging to the esters class, that many of the compounds present in the sapota juice sample were not identified in the samples of rehydrated dried powders, although some compounds such as ethyl acetate, ethyl butanoate, butyl acetate, diethyl butanedioate and 3-methyl-1-butyl acetate also were present in the rehydrated powders samples.

Among the identified compounds, some were identified in all samples; these being 1-hexanol, benzyl alcohol, phenylethyl alcohol, isoborneol, 3-phenylpropanol, hexanal, (e)-2-hexenal, benzaldehyde, decanal, ethyl acetate, acetophenone (Table 1). However, there was a significant difference ($p \leq 0.05$) between the samples of sapota juice and rehydrated dried powders (SM, SG and SMG). For the compounds 1-hexanol, benzyl alcohol, phenylethyl alcohol, benzaldehyde and ethyl acetate, its highest concentrations were found in the sample of rehydrated powder containing gum Arabic (SG) and their concentrations were 588.08, 160.36, 387.01, 110.08 and 1237.15 µg/kg of pulp, respectively. It is worth mentioning that the compounds 1-hexanol (sweet

Table 1. Volatile compounds in fresh sapota juice and in various rehydrated dried powders.

	Compounds	LRI_L	LRI_E	Sapota Juice	Rehydrated dried powder			Odor descriptors
				Concentration (µg/kg)	SM	SG	SMG	
					Concentration (µg/kg)			
	Alcohols							
1	2,3-butanediol	783	782	nd	95.54 ± 0.75	22.48 ± 0.96	nd	fruity, creamy, buttery
2	1,3-butanediol	784	785	nd	nd	nd	2.48 ± 0.38	-
3	1-hexanol[b,c]	819	827	7235.37 ± 0.76[a]	66.02 ± 0.80[c]	588.08 ± 1.20[b]	61.83 ± 1.36[c]	pungent, fruity, alcoholic, sweet
4	3-hexen-1-ol[b]	799	828	nd	nd	42.94 ± 0.23[b]	44.33 ± 0.47[a]	green leafy
5	1-butanol, 2-ethyl-	794	834	37.81 ± 0.35	nd	nd	nd	alcoholic
6	1-heptanol	972	972	29.45 ± 0.15	nd	nd	nd	musty, leafy, herbal, green, sweet,
7	benzyl alcohol[a,c,d,e,f]	1041	1041	256.17 ± 0.62[a]	96.29 ± 0.42[c]	160.36 ± 1.80[b]	78.85 ± 1.29[d]	floral, rose, phenolic, balsamic
8	1-octanol[b,c]	1076	1054	412.91 ± 0.45	nd	nd	nd	waxy, green, citrus, floral
9	1-nonanol	1075	1074	nd	nd	32.27 ± 0.68	nd	fresh, clean, floral, orange
10	phenylethyl alcohol	1119	1119	936.14 ± 1.63[a]	122.55 ± 0.86[d]	387.01 ± 0.71[b]	173.49 ± 0.16[c]	sweet, floral, fresh,
11	3-phenylpropanol[f]	1235	1232	564.25 ± 0.56[a]	252.55 ± 0.72[b]	213.97 ± 0.46 [c]	88.27 ± 0.62[d]	spicy, cinnamon, fruity, floral
12	4-methoxyphenethyl alcohol	1377	1374	nd	nd	43.80 ± 0.74	nd	-
13	2,5-bis(1,1-dimethylethyl) phenol	1519	1514	nd	nd	4.21 ± 0.22	nd	-
	Total			**9472.1**	**632.95**	**1495.12**	**449.25**	
	Aldehydes							
14	hexanal[f]	781	799	93.68 ± 0.16[a]	66.38 ± 0.23[b]	36.49 ± 0.43[c]	38.12 ± 0.10[c]	green, leafy, fruity, woody
15	(*E*)-2-hexenal[a,b]	797	821	49.53 ± 1.23[b]	56.65 ± 0.07[a]	17.40 ± 0.29[c]	23.70 ± 0.93[c]	green leafy
16	heptanal[e]	892	896	nd	6.13 ± 0.75[c]	26.11 ± 0.47[a]	13.11± 0.61[b]	fresh, aldehydic, fatty, green
17	*(E,E)*-2,4-hexadienal[b,f]	908	908	nd	25.16 ± 0.23[a]	21.43 ± 0.51[b]	8.93 ± 0.46[c]	green, fruity, citrus, waxy
18	(Z)-2-heptenal	956	958	nd	4.92 ± 0.12	nd	nd	green, fatty, oily
19	benzaldehyde[d,e,f]	960	960	429.18 ± 0.85[a]	94.86 ± 1.36[c]	110.08 ± 0.66[b]	95.76 ± 1.14[c]	sweet, almond, cherry
20	(*E,E*)-2,4-heptadienal	999	997	19.04 ± 0.75	nd	nd	nd	fatty, green, oily, aldehydic
21	Octanal	1005	1005	nd	3.90 ± 0.66[c]	20.46 ± 0.36[a]	6.30 ± 0.72[b]	waxy, citrus, orange peel,
22	benzeneacetaldehyde	1048	1047	nd	25.98 ± 1.44	nd	33.63 ± 0.93	green, sweet, floral, honey
23	Nonanal	1108	1107	nd	35.41 ± 0.42[a]	23.04 ± 0.56[c]	31.21 ± 0.62[b]	waxy, rose, orange peel
24	decanal[e]	1205	1205	15.03 ± 1.29[a]	14.25 ± 0.82[a]	12.16 ± 1.02[a]	15.05 ± 2.03[a]	sweet, waxy, orange peel
25	Undecanal	1276	1300	nd	12.20 ± 0.17	nd	nd	waxy, soapy, floral, citrus,
	Total			**606.46**	**345.84**	**267.17**	**262.81**	
	Terpenoids							
26	Myrcenol	1076	1056	nd	27.40 ± 0.54	nd	nd	fresh, citrus, floral, clean

LRI_L: Linear Retention Index from literature (National Institute of Standards & Technology, 2009); LRI_E: Linear Retention Index on HP5-MS obtained experimentally. [a] Laohakunjit et al. (2007); [b]Monteiro et al. (2018); [c]Uekane et al. (2017);[d] MacLeod & Troconis (1983);[e]Lasekan & Yap (2018);[f] Pino et al. (2003). Concentrations expressed as Mean ± SD (n=3); SM: Rehydrated dried powder of Sapota juice with maltodextrin; SG: Rehydrated dried powder of Sapota juice with gum arabic; SMG: Rehydrated dried powder of Sapota juice with maltodextrin and gum Arabic. The mean values followed by the superscript letters in rows do not differ statistically from each other. The Tukey test was applied at a 5% probability level. nd = not detected.

Table 1. Continued...

	Compounds	LRI_L	LRI_E	Sapota Juice	Rehydrated dried powder			Odor descriptors
				Concentration (µg/kg)	SM	SG	SMG	
					Concentration (µg/kg)			
27	Linalool oxide	1078	1074	nd	nd	16.83 ± 0.45	nd	floral, herbal, earthy, green
28	Camphor	1152	1151	15.45 ± 0.94[a]	5.92 ± 0.54[c]	nd	7.35 ± 0.73[b]	camphor, minty, herbal, woody
29	Isoborneol	1174	1172	285.92 ± 1.25[a]	34.49 ± 0.92[b]	30.02 ± 0.88[a]	26.92 ± 0.31[b]	camphoreous, herbal
30	Levomenthol	1180	1172	nd	12.37 ± 0.46	nd	nd	peppermint, mentholic, minty
31	Terpinen-4-ol[c,f]	1185	1184	nd	nd	nd	12.73 ± 0.69	pepper, woody, earth, musty, sweet
32	Eugenol[c,f]	1367	1367	72.90 ± 0.36[a]	nd	nd	7.23 ± 0.57[b]	sweet, spicy, woody
33	Farnesane	1382	1379	nd	nd	nd	16.11 ± 0.91	-
34	Isoeugenol	1367	1396	nd	nd	5.71 ± 0.47	nd	sweet, spicy, floral
35	Methyleugenol	1412	1411	17.87 ± 0.25[b]	nd	nd	24.75 ± 0.58[a]	sweet, fresh, spicy, cinnamon
	Total			**392.14**	**80.18**	**52.56**	**95.09**	
	Esters							
36	Ethyl acetate[a,e,f]	584	584	6059.78 ± 2.34[a]	59.97 ± 6.52[c]	1237.15 ± 1.54[b]	86.94 ± 0.98[c]	fruity, sweet, grape
37	Butyl acetate	786	789	298.13 ± 0.87[a]	nd	7.62 ± 0.24[b]	nd	fruity, banana
38	Ethyl butanoate[b]	782	799	329.16 ± 0.34[a]	nd	12.87 ± 0.57[b]	nd	fruity, juicy fruit, pineapple
39	Ethyl 2-methyl-butanoate	794	830	22.92 ± 1.20	nd	nd	nd	sweet, green, apple, fruity
40	3-methyl-1-butyl acetate	839	853	3983.44 ± 0.75[a]	41.96 ± 0.55[b]	687.89 ± 0.41[b]	nd	banana, sweet, fruity
41	3-methyl-3-butenyl acetate	848	861	22.24 ± 0.85	nd	nd	nd	fruity
42	Heptyl pentanoate	865	899	33.91 ± 0.74	nd	nd	nd	fruity, apple, green apple
43	Methyl hexanoate	924	924	35.25 ± 0.45	nd	nd	nd	fruity, pineapple
44	Ethyl hexanoate	1002	1002	88.90 ± 0.78	nd	nd	nd	sweet, fruity, pineapple, green
45	Hexyl acetate	1017	1017	319.76 ± 1.35	nd	nd	nd	fruity, apple,sweet
46	Methyl benzoate[d,e,f]	1100	1084	105.13 ± 0.41	nd	nd	nd	phenolic, almond, floral
47	Benzyl acetate	1171	1169	60.83 ± 0.21	nd	nd	nd	sweet, floral, fruity
48	Ethyl benzoate[c,d,e,f]	1178	1177	206.79 ± 0.14	nd	nd	nd	fruity, sweet
49	Diethyl butanedioate	1186	1188	53.82 ± 0.87[a]	nd	10.63 ± 0.25[b]	nd	fruity, apple
50	Ethyl benzenepropanoate[b]	1358	1353	36.52 ± 0.58	nd	nd	nd	spicy
	Total			**11656.58**	**101.93**	**1956.16**	**86.94**	
	Ketones							
51	2-heptanone[f]	870	870	nd	nd	11.81 ± 0.26	nd	fruity, spicy, sweet, herbal, coconut
52	6-methyl-5-hepten-2-one[b,c]	989	988	42.04 ± 0.28[a]	4.70 ± 0.98[c]	nd	5.46 ± 0.76[b]	citrus green, lemongrass, apple

LRI_L: Linear Retention Index from literature (National Institute of Standards & Technology, 2009); LRI_E: Linear Retention Index on HP5-MS obtained experimentally. [a] Laohakunjit et al. (2007); [b]Monteiro et al. (2018); [c]Uekane et al. (2017);[d] MacLeod & Troconis (1983);[e]Lasekan & Yap (2018);[f] Pino et al. (2003). Concentrations expressed as Mean ± SD (n=3); SM: Rehydrated dried powder of Sapota juice with maltodextrin; SG: Rehydrated dried powder of Sapota juice with gum arabic; SMG: Rehydrated dried powder of Sapota juice with maltodextrin and gum Arabic. The mean values followed by the superscript letters in rows do not differ statistically from each other. The Tukey test was applied at a 5% probability level. nd = not detected.

Table 1. Continued...

	Compounds	LRI_L	LRI_E	Sapota Juice	Rehydrated dried powder			Odor descriptors
				Concentration (µg/kg)	SM	SG	SMG	
					Concentration (µg/kg)			
53	(*E*,*E*)-3,5-octadien-2-one	1075	1068	nd	nd	nd	81.86 ± 0.86	fruity, green, grassy
54	acetophenone[c]	1071	1071	202.17 ± 0.28[a]	27.29 ± 0.25[b]	14.16 ± 0.47[c]	11.08 ± 0.73[d]	sweet, pungent, almond, acacia
55	3,5-octadien-2-one	1098	1096	nd	nd	nd	26.60 ± 0.96	fruity, fatty, mushroom
56	benzophenone	1642	1644	nd	2.67 ± 0.34	nd	nd	balsam, rose, geranium
	Lactones							
57	butyrolactone	919	917	nd	14.63 ± 0.76	nd	nd	creamy, oily, fatty, caramel
	Total				**14.63**			
	Others							
58	2,6,10-trimethyl-tetradecane	1552	1555	nd	nd	nd	4.03 ± 0.79	-
	Total						**4.03**	

LRI_L: Linear Retention Index from literature (National Institute of Standards & Technology, 2009); LRI_E: Linear Retention Index on HP5-MS obtained experimentally. [a] Laohakunjit et al. (2007); [b]Monteiro et al. (2018); [c]Uekane et al. (2017);[d] MacLeod & Troconis (1983);[e]Lasekan & Yap (2018);[f] Pino et al. (2003). Concentrations expressed as Mean ± SD (n=3); SM: Rehydrated dried powder of Sapota juice with maltodextrin; SG: Rehydrated dried powder of Sapota juice with gum arabic; SMG: Rehydrated dried powder of Sapota juice with maltodextrin and gum Arabic. The mean values followed by the superscript letters in rows do not differ statistically from each other. The Tukey test was applied at a 5% probability level. nd = not detected.

and fruity aroma) and ethyl acetate (sweet and fruity aroma), were in highest concentrations in the sapota juice samples, the values being 7235.37, and 6059.78 µg/kg of pulp, respectively. Moreover, after the drying process, these aroma compounds were well retained in juice containing gum Arabic. Another compound that showed a high concentration was 3-methyl-1-butyl acetate (banana odor, sweet and fruity aroma), which was in a concentration of 3983.44 µg/kg of pulp in sapota juice sample remained in a significant concentration (687.89 µg/kg of pulp) in the SG sample, but it was not found in the SMG sample.

Regarding the compounds 3-phenylpropanol, hexanal, (E)-2-hexenal, acetophenone and isoborneol the highest concentrations were obtained in the sample of rehydrated powder containing maltodextrin (SM), the values being 252.55, 66.38, 56.65, 27.29 and 34.49 µg/kg of pulp, respectively. As for decanal, its highest concentration (15.05 µg/kg of pulp) among the dried samples was in the sample of rehydrated powder containing a mixture of the two encapsulating agents (SMG).

There are already some publications (Uekane et al., 2017; Monteiro et al., 2018; Lasekan & Yap, 2018; MacLeod & Troconis, 1983; Pino et al., 2003; Laohakunjit et al., 2007) which studied the aroma compounds present in the sapota pulp, dehydrated fruit and fruits of the same family. Among the volatile compounds identified in these publications, those with the highest concentrations were 1-hexanol (7235.37 µg/kg of pulp), 3-methyl-1-butyl acetate (3983.44 µg/kg of pulp) and ethyl acetate (6059.78 µg/kg of pulp) (Table 1), were observed in the sapota juice sample.

The other sapota aroma volatiles are mostly benzyl-derivatives, in addition to alkyl benzoates and methyl salicylate, according to the literature (Uekane et al., 2017). Pino et al. (2003) also analyzed volatile compounds on sapota through an SDE extract and they obtained 34 compounds, ten of these were also found in this work. They reported the concentration of ethyl acetate being 1300 µg/kg of pulp, this value is much lower than found in this work in sapota juice sample (6059.78 µg/kg of pulp), while the value in the SG sample was very close (1237.15 µg/kg of pulp).

One factor that may explain the variation in the concentration of volatile compounds found in the present work is the extraction technique used in comparison with those reported in the literature The SBSE technique has been widely used and efficient for the extraction of volatile compounds in different matrices, and has advantages such as not using organic solvents, requiring a small amount of sample, having a low detection limit (LoD) and high extraction capacity of volatile and semi-volatile compounds compared to other extraction techniques (Starowicz, 2021; Franc et al., 2009). High et al. (2019) investigated the sensitivity, selectivity and reproducibility of the SAFE, SPME, SBSE and HSSE techniques for the analysis of volatile compounds from spray-dried New Zealand sheep milk and concluded that of the applied extraction techniques, SBSE demonstrated the best potential for extracting volatile compounds from this matrix. The SBSE technique detected 45 volatile compounds, while the SPME technique detected only 20 compounds. Regarding sensitivity, the SPME and HSSE methods were the least sensitive, detecting only a concentration of 173 µg/kg and 271 µg/kg respectively, while the SBSE extraction method detected a concentration of volatile compounds of 657 µg/kg. Bicchi et al. (2002) evaluated the efficiency of HSSE, SBSE and SPME extraction techniques on volatile compounds from roasted Arabica coffee and coffee brew and observed that the HSSE and SBSE techniques had a greater concentration capacity of volatile compounds than the SPME technique

On comparing the data obtained in this work, it can be concluded that these was a decrease in concentrations of most of the compounds present in the sapota juice sample in their dried samples (Table 1). Furthermore, there was not much difference in the amount of compounds identified in the dried samples; however for most of the compounds, the sample of rehydrated powder containing gum Arabic (SG) presented more relevant concentrations, than the others containing only maltodextrin (SM) or a mixture of encapsulating agents (SMG). The significant presence of these compounds quantified in dried sapota powder can be attributed to the efficiency of gum Arabic as an encapsulating agent, as well as to the spray-drying process (Forero et al., 2015).

3.2 Chemometric analysis

Chemometric analysis, Principal Components Analysis (PCA), Hierarchical Clustering Analysis (HCA), and heat map visualization were used to verify the effect of the atomization process with different encapsulating agents such as gum Arabic and maltodextrin, on the profile of volatile compounds in sapota juice.

The results of PCA of the data generated for sapota juice samples and their rehydrated powders are shown in Figure 1, which shows the variability in relation to the concentrations of volatile compounds. The score (Figure 1a) and loading (Figure 1b) plots obtained showed that the first two main components PC1 and PC2 were able to explain 80.90% of the total variation of the data, of which the main component PC1 explained 59.77% and the other component PC2, 21.13%.

The score plot (Figure 1a) shows the distribution of all studied samples in the first two main components while the loadings graph (Figure 1b) shows the distribution of the studied variables, that is, the volatile compounds, in the two main components. The sapota juice sample was allocated to PC1 positive axis. In contrast, the samples of the rehydrated powders were grouped in PC1 negative axis. The sample of rehydrated powder containing gum Arabic (SG) was allocated in a positive axis PC2, and the samples of rehydrated powders containing maltodextrin (SM), and gum Arabic and maltodextrin (SMG) were allocated to PC2 negative axis. All esters (compound numbers 36 to 50), 1-hexanol (3), 2-ethyl-1-butanol (5), 1-heptanol (6), benzyl alcohol (7), 1-octanol (8), phenylethyl alcohol (10), 3-phenylpropanol (11), benzaldehyde (19), (E,E)-2,4-heptadienal (20), isoborneol (29), and eugenol (32) were on the positive axes of PC1 and PC2. Furthermore, hexanal (14), (E)-2-hexenal (15), decanal (24), camphor (28), methyleugenol (35), 6-methyl-5-hepten-2-one (52), and acetophenone (54) were on the positive axis of PC1 and on the negative axis of PC2. These volatile compounds were correlated with sapota juice.

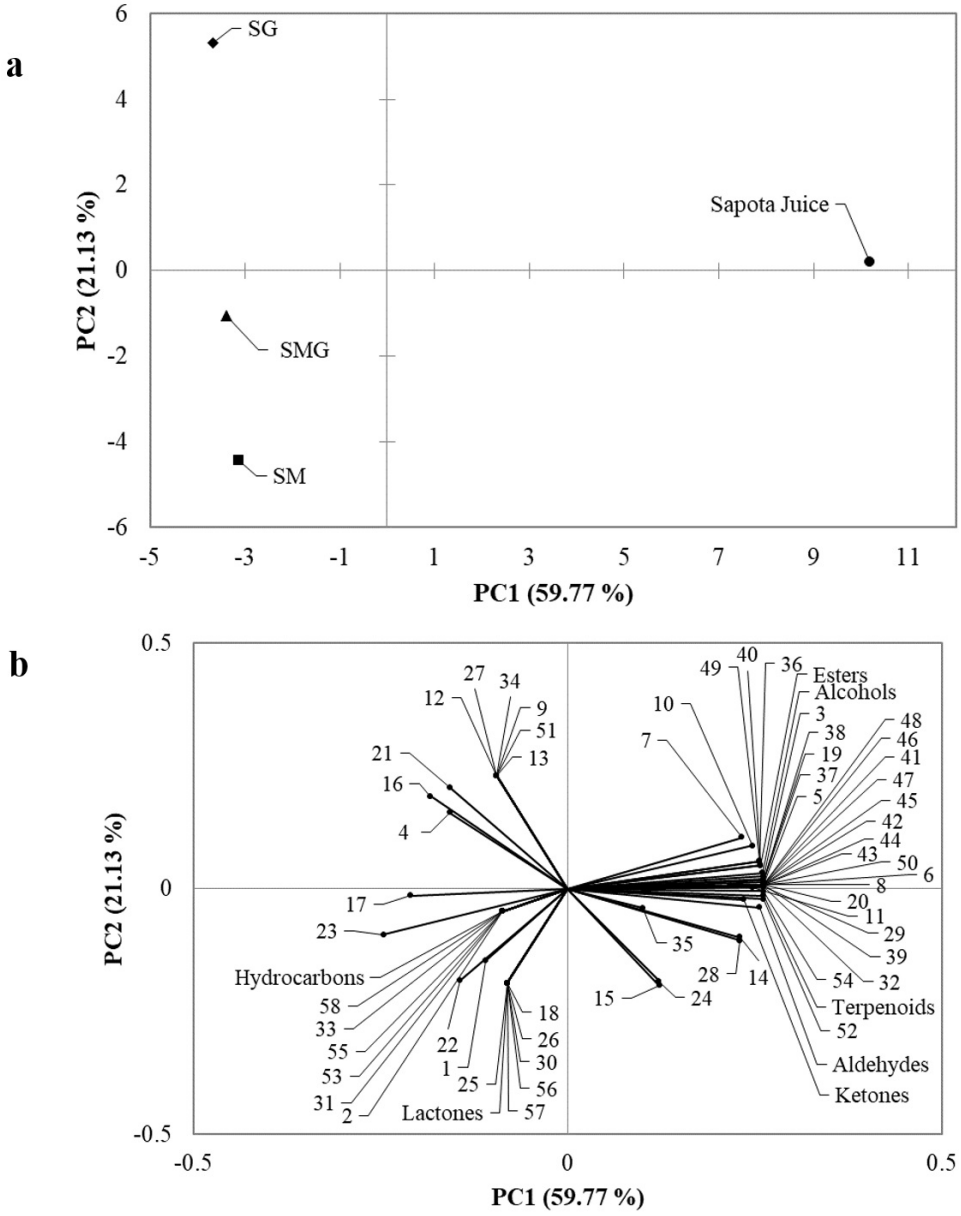

Figure 1. Principal Components Analysis of concentrations of volatile compounds from sapota juice and their rehydrated dried powders. (a) Score plot; (b) Loading plot, volatile compounds are represented by same numbers as presented in Table 1.

3-hexen-1-ol (4), 1-nonanol (9), 4-methoxyphenethyl alcohol (12), 2,5-bis(1,1-dimethylethyl) phenol (13), heptanal (16), octanal (21), linalool oxide (27), isoeugenol (34), and 2-heptanone (51) were on the negative axis of PC1 and on the positive axis of PC2. These volatile compounds were correlated with the sample SG. 2,3-butanediol (1), 1,3-butanediol (2), (E,E)-2,4-hexadienal (17), (Z)-2-heptenal (18), benzeneacetaldehyde (22), nonanal (23), undecanal (25), myrcenol (26), levomenthol (30), terpinen-4-ol (31), farnesane (33), (E,E)-3,5-octadien-2-one (53), 3,5-octadien-2-one (55), benzophenone (56), butyrolactone (57), and 2,6,10-trimethyl-tetradecane (58) were on the negative axes of PC1 and PC2. These volatiles were associated with the samples SM and SMG. These results corroborated with the data presented in Table 1, demonstrating that the atomization changes the volatile profile of sapota juice by the loss or generation of volatile compounds during the process.

However, the changes in volatile compounds of rehydrated sapota dried powders were different for the studied encapsulating materials. From the heat map (Figure 2), all samples were grouped into two clusters according to the amount of each compound. The top cluster includes the samples SM e SMG, while the bottom cluster contains the samples sapota juice and SG.

Thus, the sample of rehydrated powder containing gum Arabic (SG) presented greater similarity with the sample of sapota juice in relation to the composition of volatile compounds. In both samples, esters and alcohols presented higher concentrations in relation to the other volatile classes in the same sample,

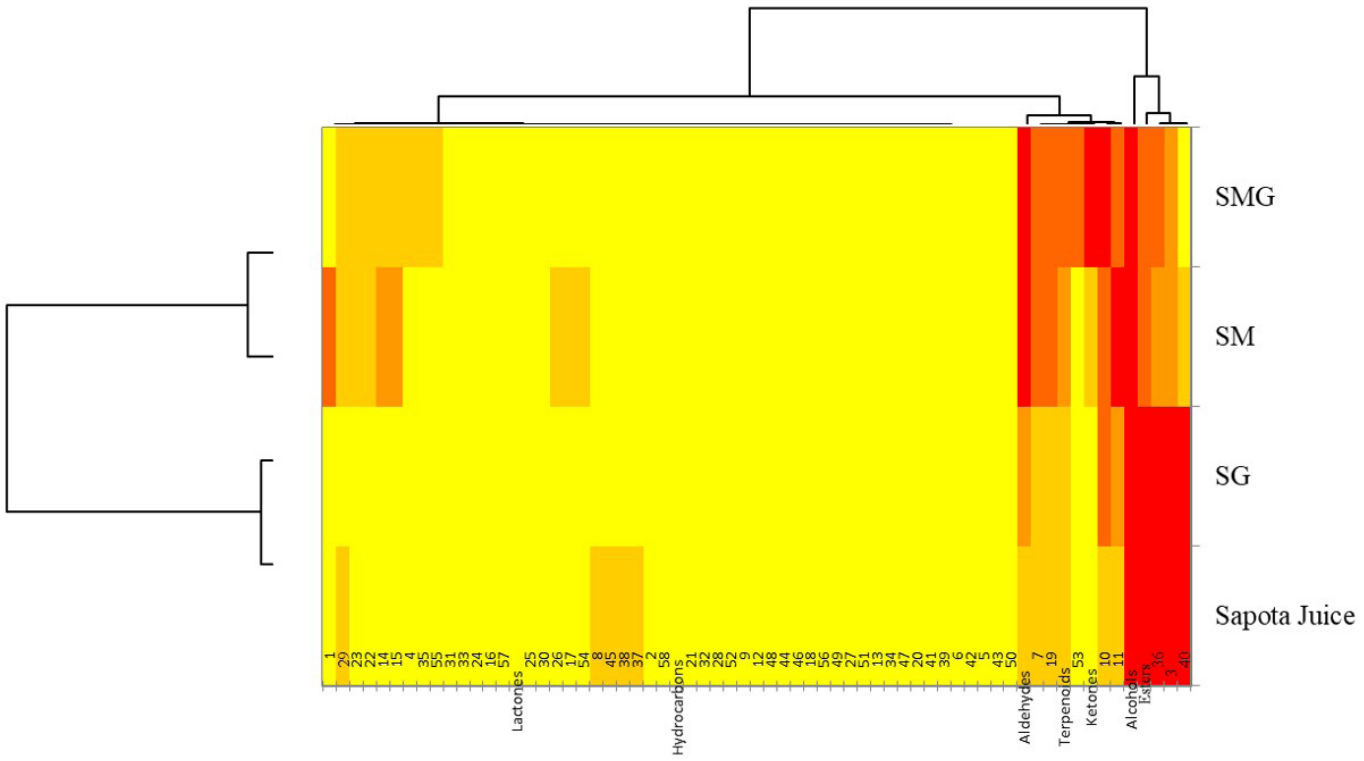

Figure 2. Hierarchical clustering analysis and heat map visualization generated data matrix of concentrations of volatile compounds of sapota juice and theirs rehydrated dried powders. The color scale represents the concentration of each volatile compound, ranging from red to yellow, which represents high and low concentration, respectively. Volatile compounds are represented by same numbers as presented in Table 1.

highlighting ethyl acetate (36), 3-methyl-1-butyl acetate (40), and 1-hexanol (3).

4 Conclusion

This work aimed to study the influence of the encapsulating agents of maltodextrin and gum Arabic on volatile compounds of sapota (*Manilkara zapota*) powder obtained by spray-drying. The major compounds with higher concentrations were ethyl acetate and 1-hexanol. Regarding the concentrations of these compounds present in the dried samples, it was verified that ethyl acetate and 1-hexanol concentrations were higher in sample SG compared to the other two samples, SM and SMG. From the heat map, the sample of rehydrated dried powder containing gum Arabic (SG) presented greater similarity with the sample of sapota juice in relation to the composition of volatile compounds. In both samples, esters and alcohols was the classes that presented higher concentrations in relation to the other volatile classes in the same sample, highlighting ethyl acetate, 3-methyl-1-butyl acetate, and 1-hexanol. In view of the results obtained, it can be concluded that the sample of rehydrated dried powder of sapota juice formulated with the use of gum Arabic (SG) showed higher concentrations in relation to most of the volatile compounds, as well as retained more volatile compounds present in sapota juice. Thus, it can be concluded that for sapota juice, the spay-drying process was more efficient when gum Arabic was used as the encapsulating agent which led to obtaining high quality product.

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References

Ahmed, M., Akter, M. S., Lee, J. C., & Eun, J. B. (2010). Encapsulation by spray drying of bioactive components, physicochemical and morphological properties from purple sweet potato. *Lebensmittel-Wissenschaft + Technologie*, 43(9), 1307-1312. http://dx.doi.org/10.1016/j.lwt.2010.05.014.

Antala, D. K., Satasiya, R. M., & Chauhan, P. M. (2021). Design, development and performance evaluation of transportation container for sapota fruit. *Journal of Food Science and Technology*, 58(10), 4024-4033. http://dx.doi.org/10.1007/s13197-020-04865-w. PMid:34471326.

Araujo, H. C. S., Jesus, M. S., Leite Neta, M. T. S., Gualberto, N. C., Matos, C. M. S., Rajan, M., Rajkumar, G., Nogueira, J. P., & Narain, N. (2021). Effect of maltodextrin and gum arabic on antioxidant activity and phytochemical profiles of spray-dried powders of sapota (*Manilkara zapota*) fruit juice. *Drying Technology*, 39(3), 392-404. http://dx.doi.org/10.1080/07373937.2020.1839487.

Bala, S., Kumar, J., & Beniway, B. S. (2017). Changes in ADF and NDF content in different stages during, storage, drying in pulp and peel of sapota (*Manilkara zapota* L.). *Annals of Applied Biology*, 33, 215-219. Retrieved from https://www.cabdirect.org/cabdirect/abstract/20183035314

Bicchi, C., Iori, C., Rubiolo, P., & Sandra, P. (2002). Headspace Sorptive Extraction (HSSE), Stir Bar Sorptive Extraction (SBSE), and Solid Phase Microextraction (SPME) applied to the analysis of roasted Arabica coffee and coffee brew. *Journal of Agricultural and Food Chemistry*, 50(3), 449-459. http://dx.doi.org/10.1021/jf010877x. PMid:11804511.

Chong, S. Y., & Wong, C. W. (2015). Production of spray-dried sapodilla (*Manilkara Zapota*) powder from enzyme-aided liquefied puree. *Journal of Food Processing and Preservation*, 39(6), 2604-2611. http://dx.doi.org/10.1111/jfpp.12510.

Fernandes, R. V. de B., Borges, S. V., & Botrel, D. A. (2014). Gum arabic/starch/maltodextrin/inulin as wall materials on the microencapsulation of rosemary essential oil. *Carbohydrate Polymers*, 101, 524-532. http://dx.doi.org/10.1016/j.carbpol.2013.09.083. PMid:24299808.

Ferreira, L., Perestrelo, R., & Câmara, J. S. (2009). Comparative analysis of the volatile fraction from *Annona cherimola* Mill. cultivars by solid-phase microextraction and gas chromatography–quadrupole mass spectrometry detection. *Talanta*, 77(3), 1087-1096. http://dx.doi.org/10.1016/j.talanta.2008.08.011. PMid:19064096.

Forero, D. P., Orrego, C. E., Peterson, D. G., & Osorio, C. (2015). Chemical and sensory comparison of fresh and dried lulo (*Solanum quitoense* Lam.) fruit aroma. *Food Chemistry*, 169, 85-91. http://dx.doi.org/10.1016/j.foodchem.2014.07.111. PMid:25236202.

Franc, C., David, F., & de Revel, G. (2009). Multi-residue off-flavour profiling in wine using stir bar sorptive extraction–thermal desorption–Gas Chromatography–Mass Spectrometry. *Journal of Chromatography. A*, 1216(15), 3318-3327. http://dx.doi.org/10.1016/j.chroma.2009.01.103. PMid:19233369.

Ganjyal, G. M., Hanna, M. A., & Devadattam, D. S. K. (2003). Processing of sapota (Sapodilla): drying. *Food Engineering and Physical Properties*, 68(2), 517-520. http://dx.doi.org/10.1111/j.1365-2621.2003.tb05704.x.

Ghani, A. (2003) *Medicinal plants of Bangladesh: chemical constituents & uses* (2nd ed., pp. 1-16). Dhaka: Asiatic Society of Bangladesh.

High, R., Bremer, P., Kebede, B., & Eyres, G. T. (2019). Comparison of four extraction techniques for the evaluation of volatile compounds in spray-dried new zealand sheep milk. *Molecules*, 24(10), 1917. http://dx.doi.org/10.3390/molecules24101917. PMid:31109044.

Jangam, S. V., Joshi, V. S., Mujumdar, A. S., & Thorat, B. N. (2008). Studies on dehydration of sapota (*Achras zapota*). *Drying Technology*, 26(3), 369-377. http://dx.doi.org/10.1080/07373930801898190.

Kumar, P., Chatli, M. K., Mehta, N., Malav, O. P., Verma, A. K., Kumar, D., & Rathour, M. (2018). Antioxidant and antimicrobial efficacy of sapota powder in pork patties stored under different packaging conditions. *Korean Journal for Food Science of Animal Resources*, 38(3), 593-605. http://dx.doi.org/10.5851/kosfa.2018.38.3.593. PMid:30018502.

Laohakunjit, N., Kerdchoechuen, O., Matta, F. B., Silva, J. L., & Holmes, W. E. (2007). Postharvest survey of volatile compounds in five tropical fruits using headspace-solid phase microextraction (HS-SPME.). *HortScience*, 42(2), 309-314. http://dx.doi.org/10.21273/HORTSCI.42.2.309.

Lasekan, O., & Yap, S. P. (2018). Characterization of the aroma compounds in fresh and dried sapodilla (*Manikara zapota*, L.) by the application of aroma extract dilution analysis. *CYTA: Journal of Food*, 16(1), 801-806. http://dx.doi.org/10.1080/19476337.2018.1485748.

Leite, M. T. S. Na., Jesus, M. S., Silva, J. L. A., Araujo, H. C. S., Sandes, R. D. D., Shanmugam, S., & Narain, N. (2019). Effect of spray drying on bioactive and volatile compounds in soursop (*Annona muricata*) fruit pulp. *Food Research International*, 124, 70-77. http://dx.doi.org/10.1016/j.foodres.2018.09.039. PMid:31466652.

MacLeod, A. J., & Troconis, N. G. (1983). Volatile flavor components of sapodilla fruit (*Achras sapota* L.). *Journal of Agricultural and Food Chemistry*, 30(3), 515-517. http://dx.doi.org/10.1021/jf00111a026.

Monteiro, S. S., Ribeiro, S. R., Soquetta, M. B., Pires, F. J., Wagner, R., & Da Rosa, C. S. (2018). Evaluation of the chemical, sensory and volatile composition of sapota-do Solimões pulp at different ripening stages. *Food Research International*, 109, 159-167. http://dx.doi.org/10.1016/j.foodres.2018.04.033. PMid:29803438.

National Horticulture Board – NHB. (2021). *Horticulture crops for 2019-20, India (1st advance estimates).* Retrieved from http://nhb.gov.in/StatisticsViewer.aspx?enc=MWoUJibk35dW2 g36TUJ.WAoZqESmAYFi7h2ielsmjlINTcFI1rG/kLbq8ZQbWUvuM

National Institute of Standards & Technology – NIST. (2022). Retrieved from: https://webbook.nist.gov/.

Oliveira, V. S., Afonso, M. R. A., & Costa, J. M. C. (2011). Caracterização físicoquímica e comportamento higroscópico de sapoti liofilizado. *Revista Ciência Agronômica*, 42, 342-348. https://doi.org/10.1590/S1806-66902011000200012.

Phisut, N. (2012). Spray drying technique of fruit juice powder: Some factors influencing the properties of product. *International Food Research Journal*, 19, 1297-1306.

Pino, J. A., Marbot, R., & Aguero, J. (2003). Volatile components of sapodilla fruit (Manilkara achras L.). *Journal of Essential Oil Research*, 15, 374-375. https://doi.org/10.1080/10412905.2003.9698614.

Ray, S., Raychaudhuri, U., & Chakraborty, R. (2016). An overview of encapsulation of active compounds used in food products by drying technology. *Food Bioscience*, 13, 76-83. http://dx.doi.org/10.1016/j.fbio.2015.12.009.

Rezende, Y. R. R. S., Nogueira, J. P., & Narain, N. (2018). Microencapsulation of extracts of bioactive compounds obtained from acerola (*Malpighia emarginata* DC) pulp and residue by spray and freeze drying: Chemical, morphological and chemometric characterization. *Food Chemistry*, 254, 281-291. http://dx.doi.org/10.1016/j.foodchem.2018.02.026. PMid:29548455.

Schweiggert, U., Hofmann, S., Reichel, M., Schieber, A., & Carle, R. (2008). Enzyme-assisted liquefaction of ginger rhizomes (*Zingiber officinale* Rosc.) for the production of spray-dried and paste-like ginger condiments. *Journal of Food Engineering*, 84(1), 28-38. http://dx.doi.org/10.1016/j.jfoodeng.2007.04.013.

Shishir, M. R. I., & Chen, W. (2017). Trends of spray drying: a critical review on drying of fruit and vegetable juices. *Trends in Food Science & Technology*, 65, 49-67. http://dx.doi.org/10.1016/j.tifs.2017.05.006.

Siddiqui, M. W., Longkumer, M., Ahmad, M. S., Barman, K., Thakur, P. K., & Kabir, J. (2014). Postharvest biology and technology of sapota: a concise review. *Acta Physiologiae Plantarum*, 36(12), 3115-3122. http://dx.doi.org/10.1007/s11738-014-1696-4.

Silva, P. I., Stringheta, P. C., Teófilo, R. F., & De Oliveira, I. R. N. (2013). Parameter optimization for spray-drying microencapsulation of jaboticaba (*Myrciaria jaboticaba*) peel extracts using simultaneous analysis of responses. *Journal of Food Engineering*, 117(4), 538-544. http://dx.doi.org/10.1016/j.jfoodeng.2012.08.039.

Starowicz, M. (2021). Analysis of volatiles in food products. *Separations*, 8(9), 157. http://dx.doi.org/10.3390/separations8090157.

Uekane, T. M., Nicolotti, L., Griglione, A., Bizzo, H. R., Rubiolo, P., Bicchi, C., Rocha-Leão, M. H., & Rezende, C. M. (2017). Studies

on the volatile fraction composition of three native Amazonian Brazilian fruits: Murici (*Byrsonima crassifolia* L., Malpighiaceae), bacuri (*Platonia insignis* M., Clusiaceae), and sapodilla (*Manilkara sapota* L., Sapotaceae). *Food Chemistry*, 219, 13-22. http://dx.doi.org/10.1016/j.foodchem.2016.09.098. PMid:27765208.

Xiao, Z., Dai, X., Zhu, J., & Yu, H. (2015). Classification of Chinese Rice wine according to geographic origin and wine age based on chemometric methods and SBSE-TD-GC-MS analysis of volatile compounds. *Food Science and Technology Research*, 21(3), 371-380. http://dx.doi.org/10.3136/fstr.21.371.

Xin, P., Zhang, S., Xu, X., Lao, F. & Wu, J. (2022). Volatile and non-volatile profiles in jujube pulp co-fermented with lactic acid bacteria. *LWT - Food Science and Technology* , 154, 112772. https://doi.org/10.1016/j.lwt.2021.112772.