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Characterization and extraction of volatile compounds from pineapple (*Ananas comosus* L. Merril) processing residues

Lília Calheiros de Oliveira BARRETTO^{1*}, Jane de Jesus da Silveira MOREIRA¹, João Antônio Belmino dos SANTOS¹, Narendra NARAIN¹, Raquel Anne Ribeiro dos SANTOS¹

Abstract

The aim of this study was to extract and identify volatile compounds from pineapple residues generated during concentrated juice processing. Distillates of pineapple residues were obtained using the following techniques: simple hydrodistillation and hydrodistillation by passing nitrogen gas. The volatile compounds present in the distillates were captured by the solid-phase microextraction technique. The volatile compounds were identified in a system of high resolution gas chromatography system coupled with mass spectrometry using a polyethylene glycol polar capillary column as stationary phase. The pineapple residues constituted mostly of esters (35%), followed by ketones (26%), alcohols (18%), aldehydes (9%), acids (3%) and other compounds (9%). Odor-active volatile compounds were mainly identified in the distillate obtained using hydrodistillation by passing nitrogen gas, namely decanal, ethyl octanoate, acetic acid, 1-hexanol, and ketones such as γ -hexalactone, γ -octalactone, δ -octalactone, γ -decalactone, and γ -dodecalactone. This suggests that the use of an inert gas and lower temperatures helped maintain higher amounts of flavor compounds. These data indicate that pineapple processing residue contained important volatile compounds which can be extracted and used as aroma enhancing products and have high potential for the production of value-added natural essences.

Keywords: agro-industrial residues; high resolution gas chromatography; mass spectrometry.

1 Introduction

Native to Central and South America, pineapple (*Ananas comosus* L. Merr.) can be found in tropical regions such as Philippines, Brazil, Costa Rica, Thailand, Hawaii, India, Malaysia and China. However, Brazil and Thailand are the first two positions in the fruit production globally (FOOD..., 2013; ZHENG et al., 2012; MORAIS; SILVA, 2011).

Due to its attractive sweet flavor, pineapple is widely consumed fresh, canned, in processed juices, and as an ingredient in exotic foods (TOKITOMO, 2007). The main pineapple varieties for commercial use are Smooth Cayenne, Singapore Spanish, Red Spanish and Selangor Green (MARCELLINI; DELISA; BOLINI, 2006). In Brazil, the most popular varieties are Perola and Smooth Cayenne (PINHEIRO; VILAS BOAS; LIMA, 2005).

Substantial losses of agricultural products occur across the entire productive chain from harvest to the final consumption. It is estimated that the damages to fruits and vegetables is around 30% to 40% because they are highly perishable products (MARTINS; FARIAS, 2002). In addition to posing environmental threat, residues also represent economic and social threat.

Pineapple fruit residues, mainly the rind are generated during the mechanical peeling of the fruit, but they still contain a large amount of pulp which is normally disposed of with other types of residues (SILVA; ZAMBIAZI, 2008). Mostly, the residue is used for animal feed (PRADO et al., 2003) and is not

utilized for obtaining other highly aggregated value by-products. Pineapple residue contains high concentrations of vitamins, sugars, fibers, and other constituents which can be used for human consumption (BOTELHO; CONCEIÇÃO; CARVALHO, 2002). Since residue disposal generates several tons of organic material, better economical, scientific, and technical ways to dispose of these residues should be investigated (FERRARI; COLUSSI; AYUB, 2004).

Previous scientific studies have reported the use of pineapple processing residue for animal feeding (LEMOIS et al., 2010; CUNHA et al., 2009), development of new products such as cereal bars (PAIVA et al., 2012; FONSECA et al., 2011), cakes (MARTIN et al., 2012) and jellies (SILVA; ZAMBIAZI, 2008), citric acid extraction (IMANDI et al., 2008), phenolic compounds production (CORREIA et al., 2004), bromelain from stem and rind (SILVA et al., 2010), hydrogel from peel cellulose (HU et al., 2010), nanocellulose from leaf fibres (CHERIAN et al., 2010), etc.

The aroma volatile compounds are important properties of fruits and are vital factors to determine the attributes of fresh and processed fruit (WEI et al., 2011). A large number of volatiles have been identified in the pineapple fruit and its derivatives (MORAIS; SILVA, 2011; PINO; QUERIS, 2010; FACUNDO, 2009; ELSS et al., 2005; SPANIER et al., 1998); however, there are no studies available on the extraction and characterization of volatile compounds from pineapple processing residue.

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The present study aimed to extract volatile compounds from pineapple processing residues using the techniques of simple hydrodistillation and hydrodistillation by passing nitrogen gas. The volatile compounds present in the distillates were captured by solid-phase microextraction (SPME) and identified by high resolution gas chromatography coupled with mass spectrometry (HRGC-MS).

2 Materials and methods

2.1 Chemicals

The following reference aroma compounds (purity higher than 98%) were obtained from Sigma-Aldrich: methyl pentanoate, 2-heptanone, *z*-ocimene, (*Z*)-3-hexen-1-ol, ethyl octanoate, nonanal, decanal, benzaldehyde, methyl 3-(methylthio)propanoate, methyl benzoate, ethyl decanoate, γ -hexalactone, γ -decalactone, δ -decalactone, methyl hexanoate, ethyl hexanoate, furfural, 1-octanol, methyl decanoate, and 2-phenyl ethanol.

2.2 Samples

Pineapple processing residue was obtained from a fruit juice industry located in Estância, in the State of Sergipe, Brazil. The processing residue consisted of rind and fibers left behind during the juice extraction step, in which the fruit was depulped using a depulping machine. The samples were mixed in a blender, placed in low-density polyethylene plastic bags, and stored at -18°C . For the extraction of volatile compounds, the samples were defrosted at room temperature and diluted with distilled water (1:3 dilution).

2.3 Methods

Simple hydrodistillation

Volatile compounds were extracted from pineapple processing residue using a condenser by circulating cold water ($\pm 2^{\circ}\text{C}$). The experiments were performed in triplicate. Fifty grams of each sample were transferred to a 500 mL flat-bottom boiling flask, which was coupled to the condenser. The flask was heated on a heating mantle with different extraction conditions: temperatures of 70; 80, and 90°C for periods of 5; 10; 15, and 30 minutes. The distillates were stored in amber flasks and kept at -18°C until the capture of volatiles by SPME.

Hydrodistillation by passing nitrogen gas

For volatile compounds extraction using nitrogen gas, 200 g of pineapple processing residue previously diluted in distilled water (1:3 dilution) was transferred to a 1000 mL dual neck flat-bottom flask. The vertical neck was coupled to a condenser, and the side neck was coupled to a high purity nitrogen gas inlet tube. The flask was heated on a heating mantle at 60°C . The gas pressure applied was 0.4 kgf/cm^2 , and the extractions were performed for 5, 10, 15, and 30 minutes. The distillates were stored in amber flasks and kept at -18°C until the capture of volatiles by SPME. Experiments were performed in triplicate.

Solid phase microextraction (SPME)

A manual SPME fiber holder 57330-U and a SPME fiber assembly 50/30 μm Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) were purchased from SUPELCO (Bellefonte, PA, USA). The volatile compounds present in the distillates obtained by both hydrodistillation techniques were captured by SPME. Five milliliters of each sample were transferred to a 40 mL amber glass vial with polypropylene cap and Teflon-faced silicone septa which contained a magnetic stirring bar. The vials were heated in a water bath for 10 minutes at 40°C to achieve the equilibrium. The SPME fiber was exposed in the vial headspace for 30 minutes at the same temperature. Later, the holder was taken to the injection part of the HRGC-MS and desorption was performed for 10 minutes.

High resolution gas chromatography-mass spectrometry (HRGC-MS)

The HRGC-MS was performed using a Varian high resolution gas chromatograph (model CP3800) with a polyethylene glycol polar capillary column (CP WAX 52CB; 30 m \times 0.25 mm i.d. \times 0.25 μm film thickness; Varian, Middelburg, The Netherlands) coupled with a mass spectrometer (Saturn 2000R). The following analytical conditions were applied: sample injection (1 μL); carrier gas helium (99.999% pure) and flow rate 1 mL/min; splitless mode; injector temperature 220°C . The oven temperature was programmed starting at 30°C (3 minutes), later increased at a rate of 2°C/min to 120°C , which was maintained for 1 minute, followed by the same increase of 2°C/min to 250°C . Mass spectra were recorded in the electron-impact (EI) mode at 70 eV, and the mass range scanned was m/z 33-1000.

Identification of volatile compounds

Chromatographic linear retention index (LRI) values were calculated using a homologous series of alkane standards (C8-C30) (PolyScience Corp., Niles, WI, EUA; purity 99.997%), which were analyzed under the same HRGC-MS conditions described above. The identification of the volatile components was performed comparing injections of commercial highly pure standards compounds (purity higher than 98%) with those of unknown compounds under the same chromatographic conditions. When high-purity chemicals were not available, the comparison was made with retention data and LRI reported in the literature (ADAMS, 2007; KONDOYAN; BERDAGUÉ, 1996; JENNINGS; SHIBAMOTO, 1980) for tentative identification of the compounds. Comparison between the mass spectra of the National Institute of Standards and Technology (NIST, USA) and that of the unknown compounds spectra was also made for tentative identification (SCHÖSSLER et al., 2009).

Statistical analysis

The data were analyzed using the Statistic 7.0 software version (STATSOFT, 2010). The analyses were performed to identify the relationships between hydrodistillation techniques and the chromatographic profile of the volatile compounds of

the pineapple residue distillates. The results of the individual experiments were used to calculate the mean values and standard deviations. Analysis of variance (ANOVA) was performed to analyze the results. Significance of differences was defined at the 5% threshold ($P < 0.05$).

3 Results and discussion

Figure 1 presents the chromatogram of the volatiles captured by SPME obtained from pineapple processing residue treated by simple hydrodistillation at 80 °C for 5 minutes. These extraction conditions were chosen based on the previous chromatographic data obtained for the other extracts (80 °C for 10, 15, and 30 minutes; and 90 °C for all these times), which indicated aroma modification and compounds characterizing sample degradation. For extraction conditions using the lowest temperature (70 °C), although there was no degradation, the peaks were much smaller when compared to those at 80 °C/5 minutes extraction condition.

The typical chromatogram (Figure 1) shows 35 volatile compounds identified from the headspace of the pineapple processing residue distillate were identified, 29 of them were positively identified based on spectra retention indices data and standards injection, while 6 compounds were tentatively

identified based on spectra and retention indices data. The peaks marked in Figure 1 are shown in Table 1. Among the identified components were esters (37%), alcohols (29%), aldehydes (9%), ketones (9%), acids (6%), and other compounds (11%).

The main volatile compounds identified in the distillate of pineapple processing residue obtained by simple hydrodistillation were (Z)-3-hexen-1-ol (35.58%), methyl octanoate (26.11%), 2-phenyl-1-ethanol (13.51%), 2-methyl-3-buten-2-ol (8.69%), and 1-hexanol (3.84%).

Figure 2 shows the chromatogram of volatile extracts obtained from pineapple processing residue treated by hydrodistillation by passing nitrogen gas at 60 °C for 30 minutes and gas pressure of 0.4 kgf/cm². The time periods of 5, 10, and 15 minutes presented smaller peaks when compared to those of extraction condition of 30 minutes. Thus the time period of 30 minutes was selected as the best time for this technique. The advantage of hydrodistillation by passing nitrogen gas was the use of lower temperature than that used in the simple hydrodistillation technique. This also avoided sample modification by caramelization. In this method, 34 volatile compounds were identified; 28 of which were positively identified, and 6 were tentatively identified. The peaks observed in Figure 2 are shown in Table 2.

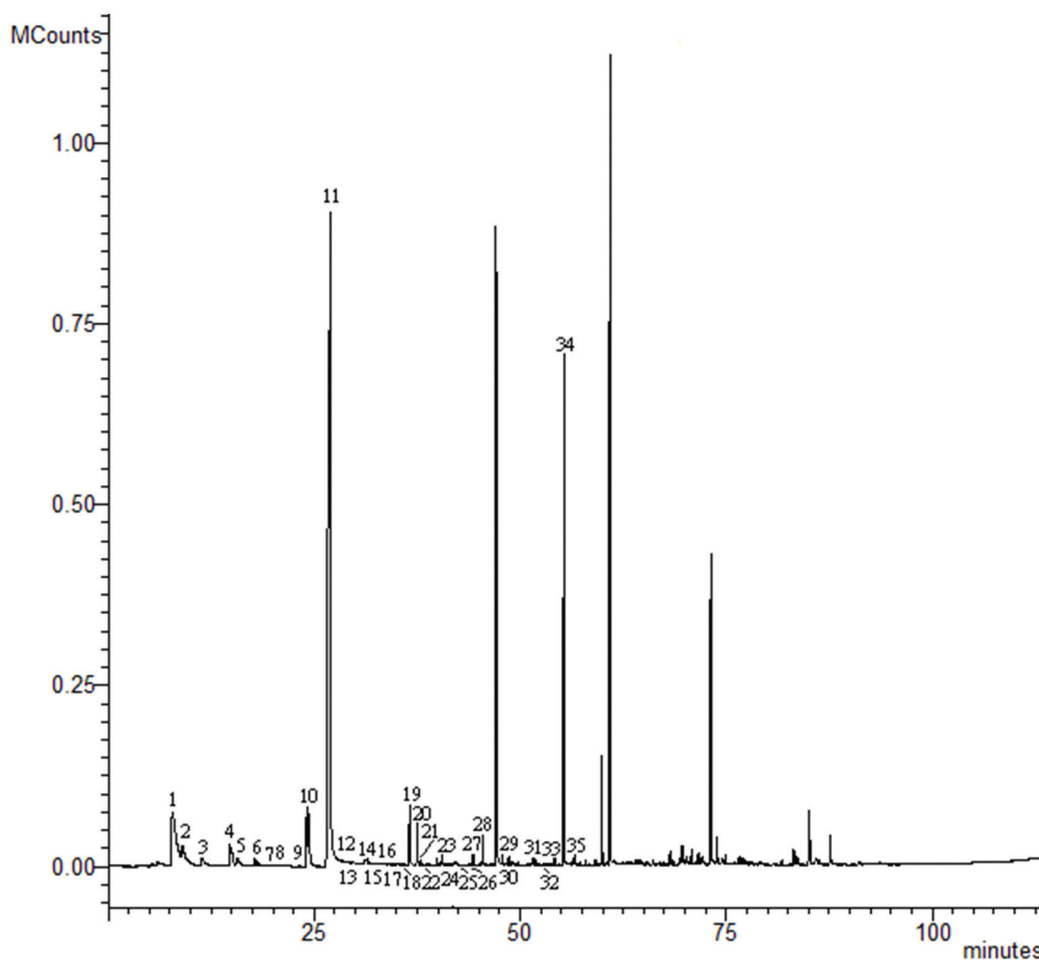


Figure 1. Chromatogram of volatile compounds captured by SPME of pineapple residue distillate previously treated by simple hydrodistillation.

Table 1. Volatile compounds identified in the distillate of pineapple processing residue obtained by simple hydrodistillation.

Peak	Compound	Area (%)			LRI ^b	LRI from literature	
		Mean ^f	±	SD ^a		A ^c	B ^d
1	2-methyl-3-buten-2-ol	8.69	±	0.06	1074	1078	1080
2	methyl pentanoate	1.62	±	0.09	1085	1093	-
3	2-pentanol	0.94	±	0.01	1114	1113	1091
4	2-heptanone	0.76	±	0.04	1172	1160	-
5	methyl hexanoate	1.27	±	0.05	1174	1177	1177
6	limonene	0.90	±	0.04	1187	1188	1206
7	z-ocimene	0.05	±	0.03	1206	1205	1228
8	1-pentanol	0.36	±	0.03	1221	1222	-
9	ethyl hexanoate	0.31	±	0.01	1227	1224	1230
10	1-hexanol	3.84	±	0.02	1323	1331	1316
11	(Z)-3-hexen-1-ol	35.58	±	0.07	1363	1362	-
12	methyl octanoate	26.11	±	0.10	1366	1378	1378
13	nonanal	0.02	±	0.08	1377	1373	1382
14	(E)-2-hexen-1-ol	0.05	±	0.03	1390	1389	-
15	acetic acid	0.08	±	0.15	1408	1410	-
16	ethyl octanoate	0.48	±	0.02	1437	1422	1423
17	furfural	0.04	±	0.02	1441	1426	1449
18	benzaldehyde	0.04	±	0.02	1496	1492	1502
19	2,3-butanediol ^e	0.06	±	0.01	1512	1494	1512
20	propanoic acid ^e	1.80	±	0.09	1522	1525	-
21	linalool	1.07	±	0.07	1538	1519	1506
22	methyl 3-(methylthio)propanoate	0.04	±	0.07	1542	1540	-
23	1-octanol	0.14	±	0.06	1546	1535	1519
24	dimethyl succinate ^e	0.05	±	0.04	1563	-	1558
25	methyl decanoate	0.16	±	0.01	1580	-	1581
26	methyl benzoate	0.04	±	0.01	1600	1601	1600
27	ethyl decanoate	0.11	±	0.01	1616	1615	1624
28	α-terpineol	0.03	±	0.02	1650	1650	1661
29	ethyl benzoate	0.33	±	0.05	1656	1663	1647
30	γ-hexalactone ^e	0.96	±	0.09	1677	1677	-
31	2-phenyl-ethyl-acetate	0.19	±	0.05	1786	1780	-
32	geraniol ^e	0.05	±	0.04	1797	1801	1797
33	ethyl-phenyl-acetate	0.11	±	0.04	1826	1823	-
34	2-phenyl ethanol	13.51	±	0.11	1859	1865	1869
35	γ-octalactone ^e	0.18	±	0.09	1879	1877	1883

^fMean – mean value; ^aSD – standard deviation (SD/average × 100) of three injections; ^bLRI – linear retention index; ^cA – Kondjoyan and Berdagué (1996); ^dB – Jennings and Shibamoto (1980); ^eVolatile compounds tentatively identified.

Pineapple processing residue was composed mainly of esters (35%), followed by ketones (26%), alcohols (18%), aldehydes (9%), acids (3%), and other compounds (9%). The major volatile compounds obtained by hydrodistillation by passing nitrogen gas were 1-hexanol (60.19%), ethyl phenyl-acetate (14.54%), 2-methyl-3-buten-2-ol (9.33%), γ-butyrolactone (2.08%), 2-phenyl ethyl-acetate (1.72%), and ethyl octanoate (1.19%).

The performance of both methods applied in this study, simple hydrodistillation and hydrodistillation by passing nitrogen gas, were compared. The results showed some compounds with similar area percentage ($P < 0.05$) such as 2-methyl-3-buten-2-ol (8.69% and 9.33%, respectively), methyl pentanoate (1.62% and 1.06%, respectively), linalool (1.07%

and 1.08%, respectively), and γ-hexalactone (0.96% and 1.09%, respectively) (Table 3).

Wei et al. (2011) identified 44 volatile compounds when characterizing pineapple pulp and core by headspace-solid phase microextraction (HS-SPME) and gas chromatography-mass spectrometry (GC/MS). Some of them are the same as those identified in the present study, in which pineapple residues were characterized: methyl hexanoate, ethyl hexanoate, methyl 3-(methylthio) propanoate, methyl octanoate, ethyl decanoate, α-terpineol, nonanal, and decanal. However, the ones with the highest odor activity values were ethyl hexanoate, nonanal, and decanal, which were also identified in this study.

Morais and Silva (2011) also emphasized that ethyl hexanoate is one of the most important compounds related

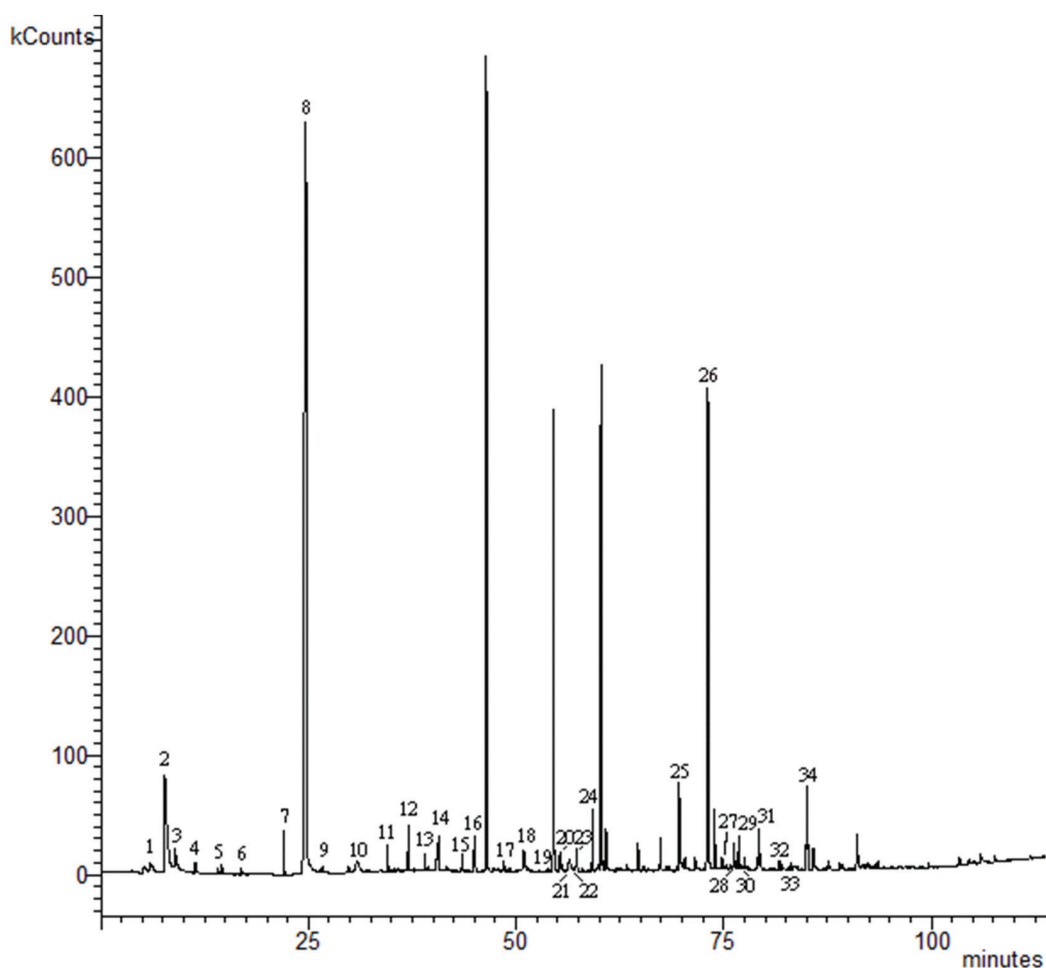


Figure 2. Chromatogram of volatile compounds captured by SPME of pineapple residue distillate previously treated by hydrodistillation by passing nitrogen gas.

to the pineapple flavor. According to Facundo (2009), the compound ethyl hexanoate is related to the aroma note described as 'pineapple'. This compound was also identified in the pineapple processing residue distillate obtained by simple hydrodistillation technique. Ethyl octanoate, 2-heptanone, and γ -hexalactone were also identified by Facundo (2009) and classified as flavor compounds that have aroma notes such as fruity, floral, citric, herb, and honey. These compounds were identified in the pineapple processing residue distillate obtained by both hydrodistillation techniques in this study.

Tokitomo et al. (2005) prepared an aroma distillate from fresh pineapple using solvent-assisted flavor evaporation and detected 29 odor-active compounds. Some of them were also identified in the distillate of pineapple processing residue obtained by hydrodistillation by passing nitrogen gas such as γ -octalactone (fruity, coconut-like), δ -octalactone (coconut-like), γ -decalactone (fruity, sweet, peach-like), and γ -dodecalactone (fruity, sweet).

Elss et al. (2005) reported the presence of the following volatile compounds when characterizing aroma of fresh pineapple juice and its water phase extracts: 2-methyl-3-buten-

2-ol, methyl pentanoate, butyl acetate, hexanal, 2-pentanol, 1-butanol, ethyl hexanoate, limonene, α -ocimene, linalool, furfural, acetic acid, α -terpineol, geraniol, and γ -octalactone. These compounds were also identified in the distillates obtained in this study.

Spanier et al. (1998) analyzed volatile components present in fresh-cut pineapple and the effect of its storage. Some of these compounds showed important odor activity and were similar to the ones found in this study such as acetic acid, 1-hexanol, and nonanal.

Although ethyl hexanoate is an important pineapple aroma volatile compound (MORAIS; SILVA, 2011), it was not present in high amounts in the pineapple processing residue distillates obtained by both hydrodistillation methods (0.31% for simple hydrodistillation and not detected using hydrodistillation by passing nitrogen gas). However, other major volatile compounds were identified in the distillate obtained using hydrodistillation by passing nitrogen gas system, namely, decanal, ethyl octanoate, acetic acid, 1-hexanol, and many ketones such as γ -hexalactone, γ -octalactone, δ -octalactone, γ -decalactone and γ -dodecalactone (Table 2). This suggests that the use of an inert

Table 2. Volatile compounds identified in the distillate of pineapple processing residue obtained by hydrodistillation by passing nitrogen gas.

Peak	Compound	Area (%)			LRI ^b	LRI from literature	
		Mean ^f	±	SD ^a		A ^c	B ^d
1	butyl acetate	0.36	±	0.09	1056	1059	1059
2	2-methyl-3-buten-2-ol	9.33	±	0.12	1073	1078	1080
3	methyl pentanoate	1.06	±	0.08	1085	1093	-
4	2-pentanol	0.62	±	0.15	1113	1113	1091
5	ethyl 2-butenate	0.21	±	0.13	1161	1161	-
6	2-heptanone	0.39	±	0.05	1168	1160	-
7	<i>z</i> -ocimene	0.23	±	0.04	1208	1205	1228
8	1-hexanol	60.19	±	0.11	1330	1331	1316
9	(<i>Z</i>)-3-hexen-1-ol	0.18	±	0.05	1361	1362	-
10	acetic acid	0.13	±	0.03	1411	1410	-
11	ethyl octanoate	1.19	±	0.05	1427	1422	1423
12	furfural	0.02	±	0.01	1445	1426	1449
13	decanal	0.84	±	0.07	1488	1478	1485
14	benzaldehyde	0.07	±	0.01	1495	1492	1502
15	2,3-butandiol ^c	0.09	±	0.01	1510	1494	1512
16	linalool	1.08	±	0.08	1530	1519	1506
17	methyl 3-(methylthio)propanoate	0.06	±	0.01	1541	1450	-
18	dimethyl succinate ^c	0.44	±	0.01	1564	-	1558
19	methyl decanoate	0.04	±	0.01	1580	-	1581
20	γ -butyrolactone ^c	2.08	±	0.02	1593	1592	-
21	methyl benzoate	0.12	±	0.01	1609	1601	1600
22	ethyl decanoate	0.08	±	0.01	1613	1615	1624
23	α -terpineol	0.04	±	0.01	1649	1650	1661
24	γ -hexalactone	1.09	±	0.18	1670	1677	-
25	2-phenyl-ethyl-acetate	1.72	±	0.09	1776	1780	-
26	ethyl-phenyl-acetate	14.54	±	0.10	1844	1823	-
27	2-phenyl ethanol	0.69	±	0.06	1858	1865	1869
28	γ -octalactone ^c	0.77	±	0.01	1879	1877	1883
29	δ -octalactone	0.03	±	0.01	1929	1887	1929
30	ethyl cinnamate	0.03	±	0.01	2097	-	2095
31	γ -decalactone	1.08	±	0.04	2104	2094	2101
32	δ -decalactone	0.07	±	0.01	2148	2145	2144
33	γ -dodecalactone ^c	1.09	±	0.05	2317	2317	2317
34	δ -dodecalactone ^c	0.04	±	0.01	2351	-	2358

^fMean – mean value; ^aSD – standard deviation (SD/average x 100) of three injections; ^bLRI – linear retention index; ^cA – Kondjoyan and Berdagué (1996); ^dB – Jennings and Shibamoto (1980); ^eVolatile compounds tentatively identified.

gas and lower temperatures helped maintain higher amounts of odor-active compounds (FACUNDO, 2009; TOKITOMO et al., 2005) than the use of simple hydrodistillation technique. Therefore, the use of nitrogen gas led to more volatile compounds of interest for the flavor industry.

Oliveira et al. (2012) used hydrodistillation techniques for extraction of volatiles from yellow passion fruit residues and obtained aroma compounds similar to the ones present in the fresh fruit. Hydrodistillation by passing nitrogen gas was also preferred when compared with the simple system.

The volatile compounds identified in pineapple processing residue indicate that they have a great potential for the production of aromatic natural essences which could later

be added to products such as pineapple juice concentrate enhancing its sensorial quality and increasing competitiveness of this type of product in the global market.

4 Conclusion

This study reports for the first time the volatiles profile of pineapple processing residue, which contains a reasonable amount of flavor compounds similar to those present in the fresh fruit juice. The use of an inert gas and lower temperatures helped maintain higher amounts of odor-active compounds such as decanal, ethyl octanoate, acetic acid, 1-hexanol, γ -hexalactone, γ -octalactone, δ -octalactone, γ -decalactone, and γ -dodecalactone. The results indicate that pineapple processing residue have potential for aromatic essence production.

Table 3. Comparison between hydrodistillation methods – Common volatile compounds identified in the distillate of pineapple processing residue.

Peak		Compound	Area (%)	
SIMP	N ₂		SIMP	N ₂
1	2	2-methyl-3-buten-2-ol	8.69 ^a	9.33 ^a
2	3	methyl pentanoate	1.62 ^a	1.06 ^a
3	4	2-pentanol	0.94 ^a	0.62 ^a
4	6	2-heptanone	0.76 ^a	0.39 ^b
7	7	z-ocimene	0.05 ^a	0.23 ^b
10	8	1-hexanol	3.84 ^a	60.19 ^b
11	9	(Z)-3-hexen-1-ol	33.58 ^a	0.18 ^b
15	10	acetic acid	0.08 ^a	0.13 ^a
16	11	ethyl octanoate	0.48 ^a	1.19 ^b
17	12	furfural	0.04 ^a	0.02 ^a
18	14	benzaldehyde	0.04 ^a	0.07 ^a
19	15	2,3-butandiol	0.06 ^a	0.09 ^a
21	16	linalool	1.07 ^a	1.08 ^a
22	17	methyl-3-(methylthio)-propanoate	0.04 ^a	0.06 ^a
14	18	dimethyl succinate	0.05 ^a	0.44 ^b
25	19	methyl decanoate	0.16 ^a	0.04 ^b
26	21	methyl benzoate	0.04 ^a	0.12 ^b
27	22	ethyl decanoate	0.11 ^a	0.08 ^a
28	23	α-terpineol	0.03 ^a	0.04 ^a
30	24	γ-hexalactone	0.96 ^a	1.09 ^a
31	25	2-phenyl-ethyl-acetate	0.19 ^a	1.72 ^b
33	26	ethyl-phenyl-acetate	0.11 ^a	14.54 ^b
34	27	2-phenyl ethanol	13.51 ^a	0.69 ^b
35	28	γ-octalactone	0.18 ^a	0.77 ^b

SIMP – Simple hydrodistillation; N₂ – Hydrodistillation by passing nitrogen gas. Values with different superscript letters (a, b) are significantly different (P < 0.05).

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