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## Dynamics of the recovery of aroma volatile compounds during the concentration of cashew apple juice (*Anacardium occidentale* L.)

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### ABSTRACT

The objective of this study was to identify and quantify the recovery of aroma volatiles during the concentration of cashew apple juice and propose kinetic models. Fresh juice was concentrated in a thermal-siphon type evaporator, operating in a closed system at 700 mm Hg. The water and volatiles evaporated during concentration were recovered by condensation, generating five condensates: the first was obtained during the concentration of the juice from 10.6 °Brix (fresh juice) to 12 °Brix, the second from 10.6 to 14 °Brix, the third from 10.6 to 19 °Brix, the fourth from 10.6 to 28 °Brix and the fifth from 10.6 to 40 °Brix. The volatiles in the headspaces of the condensates were vacuum stripped (70 mm Hg) to a Porapak Q™ trap for 2 h, eluted with 300 µL of acetone, identified by GC–MS and quantified by external standardization. Trained judges rated the intensity of the cashew apple aroma perceived in the condensates using a 9 cm scale. The major classes of volatiles present in the condensates were esters (~90% of the total mass of volatiles), followed by aldehydes (~6%) and alcohols (~3%). In the first condensate the ester (580.3 µg L<sup>-1</sup>), aldehyde (39.3 µg L<sup>-1</sup>) and alcohol (23.5 µg L<sup>-1</sup>) concentrations were higher than in the remaining condensates, suggesting that a more efficient recovery of the volatiles important to the cashew apple aroma and flavor could be obtained when the beverage was concentrated from 10.6 to approximately 12 °Brix, namely, by condensing the first 23% of the water evaporated off from the juice. The power function was the kinetic model that best fitted the recovery of the esters, aldehydes and alcohols.

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### 1. Introduction

In recent years, the Brazilian production of concentrated cashew apple (*Anacardium occidentale* L.) juice has generated annual revenues of approximately US\$ 71.5 million, only surpassed by concentrated orange and grape juices, whose revenues in 2009 were respectively US\$ 1.7 billion and US\$ 152.95 million (IBGE, 2012). This shows the great economic importance and potential for the sales of this beverage, attributed mainly to its exotic aroma and flavor.

The aromatic notes characteristic of fresh cashew apple juice, are mainly credited to esters. In studies developed by Garruti, Franco, Da Silva, Janzanti, and Alves (2003), 24 of the 48 compounds identified in the fresh beverage were esters, and together they corresponded to 50% of the total mass of the volatiles isolated from the samples.

Bicalho, Pereira, Aquino Neto, Pinto, and Rezende (2000) reported that more than 80% of the total mass of volatiles isolated from fresh cashew apple pulp belonged to the class of esters. Pioneer studies carried out by Maciel, Hansen, Aldinger, and Labows (1986) with fresh cashew juice, also showed that in numerical terms, the esters represented about 50% of the volatile compounds identified in the samples.

However, thermal processing, notably the concentration step, contributes to the loss of esters in cashew apple juice, with undesirable impact to the beverage sensory quality. This was verified by Garruti, Facundo, Souza Neto, and Wagner (2008) when monitoring the concentration of esters in cashew apple juice during its processing in a Brazilian plant. The authors reported that the concentration of important odor active esters, such as ethyl propanoate, ethyl butanoate, ethyl *trans*-2-butenate, isobutyl isovalerate, ethyl 3-hexenoate and ethyl octanoate, were reduced after the heat processing of the juice, principally after the concentration step, compromising the profile and sensory quality of the concentrated juice. Sampaio, Garruti, Franco, Janzanti, and Da Silva (2011) also found that during the concentration of cashew apple juice, a considerable amount of esters were lost by evaporation, especially ethyl 2-hydroxyhexanoate, ethyl *trans*-2-butenate and

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ethyl 2-methylbutanoate, aroma active volatiles important to the cashew-like aroma prevailing in the fresh fruit.

In order to replace volatiles lost during the concentration of juices, the food industry recovers, by condensation, volatile compounds evaporating off from the juice together with the water, subsequently concentrating this fraction to give a by-product known as “water phase”, which is added back to the juice in order to recover the natural aroma and flavor of the fresh fruit (Bomben, Bruin, Thijssen, & Merson, 1973; Yanniotis, Tszilioni, Dendrinis, & Mallouchos, 2007). However the Brazilian companies still have difficulty in efficiently recovering the volatiles lost during the concentration of tropical juices such as cashew apple. This was evident in the work of Sampaio et al. (2011), in which they analyzed the cashew apple water phase produced by a juice industry in the Northeast of Brazil, and identified the presence of an excessive proportion of alcohols, in detriment of the esters.

In fact the recovery of volatiles lost during the concentration of juices, in order to obtain a water phase with good sensory quality, is not trivial. According to Ramteke, Eipeson, and Patwardhan (1990), Ramteke, Singh, Rekha, and Eipeson (1993) and Karlsson and Trägårdh (1997), for each type of fruit the amount of water evaporating off from the juice that must be recovered by condensation to obtain the volatiles of importance to the beverage aroma and flavor is a fundamental information, defining various conditions regarding the recovery of the volatiles during the juice concentration process. For this reason, studies which evaluate the dynamics of recovery of the volatiles of importance to the aroma and flavor of fruit juices during their concentration, have already been carried out for mangoes, pineapples and guavas (Ramteke et al., 1990), apples (Bomben et al., 1973; Sulc, 1984), banana (Khalil, 1990), and for grapes, apricots, peaches, strawberries and plums (Sulc, 1984).

Studies carried out by Ramteke et al. (1990) concerning the dynamics of evaporating volatiles during the concentration of various juices, showed that in order to recover 90% of the volatiles present in mango juice, one needs to condense only the first 30% of water evaporated off during the concentration of the juice. Similarly, during the concentration of apple juice, the greater part of the volatiles associated with the aroma/flavor of the fruit is recovered by condensing the first 10% of the water originally present in the juice (Bomben et al., 1973). The recovery of more of the evaporated water leads to an undesirable dilution of the water phase.

For cashew apple juice, despite the attempt reported by Ramteke, Eipeson, Sing, Chikkaramu, and Patwardhan (1984), information concerning the dynamics of recovery of the aroma volatiles lost during the beverage concentration is not available. This makes it difficult for the Brazilian juice producers to optimize the conditions of their aroma recovery operations. Thus the objective of this research was to characterize the dynamics of the recovery of aroma volatiles evaporated from cashew apple juice during its concentration. This information will aid the industries and researchers to develop and improve alternative processes to recover the mentioned volatiles, producing water phase and concentrate beverage with enhanced sensory quality.

## 2. Material and methods

### 2.1. Raw material

10 kg of pseudofruits from the clone CCP 76 of cashew apple tree named “early dwarf”, cultivated in Artur Nogueira (São Paulo, Brazil) was used. After the removal of the peduncle of mature pseudofruits, the material was washed, ground in a blender and filtered through cotton cloth. The cashew apples were sanitized by immersion in a 100 mg L<sup>-1</sup> free chlorine solution for 20 min and then rinsed with running water.

### 2.2. Juice concentration

Three and a half kilos of cashew apple juice was concentrated in a thermal-siphon type pilot evaporator (Precision Scientific Co., USA),

operating in a closed system with 700 mm Hg pressure and a steam heating temperature of 110 °C, until the beverage reached 40 °Brix. During concentration, the juice remained at a temperature of 38 °C ± 2 °C. The water vapor and the volatiles freed from the juice were recovered by condensation using a stainless steel column cooled by water at 2 °C.

Each time about 20% of the total amount of water that should be removed from the juice for it to reach 40 °Brix had been evaporated, the system was paralyzed and the °Brix of the juice measured in triplicate using a Carl Zeiss bench refractometer model 844976 Jen (Carl Zeiss Industrial Metrology, Maple Grove, Minnesota, USA). Using the difference in °Brix between each concentration step, the mass of water evaporated from the juice was calculated. It was called “mass of water theoretically evaporated from the juice” and compared with the mass of water recovered by condensation in order to estimate the efficiency of the recovery process.

The water and the volatiles recovered by condensation were collected, giving rise to 5 different condensates. These contained all the water and evaporated volatiles obtained when the juice was concentrated from approximately 11 to 12 °Brix (condensate<sub>11/12</sub>), from 12 to 14 °Brix (condensate<sub>12/14</sub>), from 14 to 19 °Brix (condensate<sub>14/19</sub>), from 19 to 28 °Brix (condensate<sub>19/28</sub>), and from 28 to 40 °Brix (condensate<sub>28/40</sub>). The condensates were collected in a hermetically closed kitassato and weighed using a precision electronic balance. They were then transferred to the amber colored glass flasks and frozen until used to elaborate the samples. These procedures were carried out in two independent batches (repetitions), thus processing a total of 7 kilos of cashew apple juice.

In sequence, using the condensates described above, four samples were prepared that were representative of all the water and volatiles recovered when the juice was concentrated from 11 to 14 °Brix (condensate<sub>11/14</sub>), from 11 to 19 °Brix (condensate<sub>11/19</sub>), from 11 to 28 °Brix (condensate<sub>11/28</sub>) and from 11 to 40 °Brix (condensate<sub>11/40</sub>). Eq. (1) shows the calculations used to elaborate a 300 g sample representative of condensate<sub>11/14</sub>, as from a mixture of condensate<sub>11/12</sub> and condensate<sub>12/14</sub> in quantities proportional to those recovered during concentration of the juice.

$$300 \text{ g of condensate}_{11/14} = \left[ \frac{(m_{11/12})}{(m_{11/12} + m_{12/14})} \right] \times 300 + \left[ \frac{(m_{12/14})}{(m_{11/12} + m_{12/14})} \right] \times 300 \quad (1)$$

where  $m_{11/12}$  is the mass of all the material condensed when the juice was concentrated from 11 to 12 °Brix and  $m_{12/14}$  is the mass of all the material condensed when the juice was concentrated from 12 to 14 °Brix. Similar procedures were used to prepare samples representative of each of the four condensates analyzed in the present research, that is: condensate<sub>11/14</sub>, already described; condensate<sub>11/19</sub> – containing the water and volatiles recovered when the juice was concentrated from 11 to 19 °Brix, condensate<sub>11/28</sub> – containing the water and volatiles recovered when the juice was concentrated from 11 to 28 °Brix and finally condensate<sub>11/40</sub>, the latter containing all the water and volatiles recovered throughout the entire concentration of the juice.

All these condensates were immediately stored in amber flasks, hermetically sealed, frozen at – 18 °C and maintained at this temperature until used for the chemical and sensory analyses.

### 2.3. Sensory evaluation of the condensates

The intensity of the aroma characteristic of cashew apple present in the five condensates – condensate<sub>11/12</sub>, condensate<sub>11/14</sub>, condensate<sub>11/19</sub>, condensate<sub>11/28</sub> and condensate<sub>11/40</sub> – was evaluated with three repetitions by four previously selected panelists and trained as recommended by Meilgaard, Civille, and Carr (2007).

The condensates were evaluated in black-bottomed wine glasses covered with watch glasses and coded with three-digit numbers. The panelists were requested to smell the condensates and indicate the intensity of the aroma characteristic of cashew apple present in each one, using a 9 cm non-structured scale anchored at the extreme left by the term “weak”, in the center of the scale by the term “moderate” and at the extreme right by the term “strong”. The order of the presentation of the samples was balanced between the panelists.

The present study was approved by the Committee of Ethics in Research of the University of Campinas under protocol number 1058/2008.

#### 2.4. Isolation of the volatiles recovered in the condensates

The volatiles in the headspaces of the five condensates were vacuum stripped (70 mm Hg) to a Porapak Q<sup>TM</sup> trap during 2 h and then eluted with 300  $\mu\text{L}$  of acetone (Franco & Rodrigues-Amaya, 1983; Garruti et al., 2003). The Porapak Q<sup>TM</sup> polymer (80–100 mesh) was purchased from Supelco (Bellefonte, PA, USA), and prior to use in the experiment was heated at 170 °C for 8 h in a flow of pure nitrogen. Every 2 h the polymer was removed from the incubator and rinsed with 3 mL of hexane followed by 3 mL of acetone to guarantee its total cleanliness. The hexane and acetone were chromatographic grade (Mallinckrodt Baker, Phillipsburg, NJ, USA). Three hundred grams of each sample of condensate – condensate<sub>11/12</sub>, condensate<sub>11/14</sub>, condensate<sub>11/19</sub>, condensate<sub>11/28</sub> and condensate<sub>11/40</sub> – was used to extract the volatiles, to which 30% w/w of NaCl was added to avoid possible enzymatic reactions, as suggested by Franco and Rodrigues-Amaya (1983). The isolates containing the volatile compounds present in each condensate were stored in screw-topped, Teflon-lined glass flasks and maintained at –18 °C.

#### 2.5. Gas chromatography–mass spectrometry (GC–MS)

The volatiles present in the five isolates were identified following overall procedures described by Sampaio et al. (2011). It was used an Agilent 7890/5975 gas chromatograph mass spectrometer (Palo Alto, CA, USA) operating with an ionization voltage of 70 eV, scan mode and  $m/z$  range from 35 to 550. Separation of the volatiles was attained through a DB-Wax (JW Scientific, Folsom, CA, USA) fused silica capillary column (30 m  $\times$  0.25 mm, 0.25  $\mu\text{m}$ ), maintained at 50 °C for 8 min, raised to 110 °C at 4 °C  $\text{min}^{-1}$ , and then to 200 °C at 16 °C  $\text{min}^{-1}$  and maintained at 200 °C for 20 min, giving a total of approximately 48 min of analysis time (Garruti et al., 2003). The splitless mode injector was maintained at 200 °C and helium was the carrier gas at a flow rate of 1.5 mL  $\text{min}^{-1}$ . The injection volume was 2  $\mu\text{L}$ . The volatiles were also separated using a DB-5 (JW Scientific, Folsom, CA, USA) fused silica capillary column (30 m  $\times$  0.25 mm, 0.25  $\mu\text{m}$ ) under the same conditions as described above.

##### 2.5.1. Identification of the volatiles

To assist with the volatiles identification, initially a C7–C28 series of straight-chain alkanes (Polyscience 211 C kit, Chicago, USA) dissolved in an acetone solution was injected in the GC–MS under the same conditions previously described. The alkanes were separated using the two columns mentioned previously. Thus for each of the chromatographic columns (DB-Wax and DB-5) the linear retention indexes (LRI) of the volatile compounds were calculated for the GC–MS data as recommended by Van Den Dool and Kratz (1963) and compared with the indices reported in the literature (Cacho, Moncayo, Palma, Ferreira, & Culleré, 2012; Galvão, Narain, Santos, & Nunes, 2011; Garruti et al., 2003; Pherobase, 2011; Sampaio et al., 2011; Valim, Rouseff, & Lin, 2003).

The individual compounds were identified using the same criteria followed by Sampaio et al. (2011) which included: i) comparison of their mass spectra with those provided by the Nist library (NIST/EPA/NIH Mass Spectral Library, version 2.0, 2008) and with pure

standard analyzed in the same GC–MS system using the same methodological conditions, and ii) comparing their linear retention index with those published in the literature for columns with the same polarity.

As described by Sampaio et al. (2011), the compounds were considered positively identified if their mass spectra and retention indices were similar to those of the pure standards. They were considered identified when their mass spectra matched those available in the computerized library and their retention indices were comparable to those reported in the literature (Cacho et al., 2012; Galvão et al., 2011; Garruti et al., 2003; Pherobase, 2011; Sampaio et al., 2011; Valim et al., 2003).

##### 2.5.2. Quantification of the volatiles present in the condensates

The volatiles contained in the five isolates were quantified by external standardization using eight standards from the following chemical classes: esters, ketones, aldehydes, alcohols, carboxylic acids and terpenes. Ethyl isovalerate and ethyl cinnamate were used for the ester class; 2,3-butanedione for ketones, hexanal for aldehydes, 3-hexen-1-ol for alcohols, 2-methylbutanoic acid for carboxylic acids,  $\beta$ -myrcene for terpenes and hydrocarbons, and carveol for alcoholic terpenes. Each standard was prepared in nine different concentrations varying from 0.0025 to 50 mg  $\text{L}^{-1}$ . The standard solutions were prepared by dissolving pure standards in acetone (Mallinckrodt Baker, Phillipsburg, NJ, USA). A predictive statistic model was calculated for each standard between the concentration (mg  $\text{L}^{-1}$ ) of the same versus the unit of area obtained using the GC–MS. The statistic models were used to quantify the volatile compounds identified in the five condensates. For this, each isolate was analyzed in three replications. The compounds were quantified using the results obtained from the DB-Wax column.

#### 2.6. Identification of the odor quality and importance of the volatiles in the condensates

The odoriferous importance of the volatiles present in the isolates of the condensate<sub>11/12</sub> and condensate<sub>11/40</sub> was determined by GC–Olfactometry using the Osme technique (Da Silva, Lundahl, & McDaniel, 1994; Garruti et al., 2003; Miranda-Lopez, Libbey, Watson, & McDaniel, 1992; Sampaio et al., 2011).

The isolates were analyzed using an Agilent 7890 (Palo Alto, USA) high-resolution gas chromatograph (GC). The column (DB-Wax) and oven conditions were the same as those used in the GC–MS analysis. Hydrogen was the carrier gas at a flow rate of 1.5 mL  $\text{min}^{-1}$ . The volume injected was 2  $\mu\text{L}$ . For the GC-olfactometric evaluation, the GC was modified such that the chromatographic column was disconnected from the flame ionization detector (FID) and connected to another base of the chromatograph with no detector, maintained at 250 °C. A glass tube was fitted to this base, which sent the chromatographic effluents leaving the column directly to the panelist's nose, as described in Sampaio et al. (2011).

The effluents from the column were carried to the panelist's nose using previously purified and moistened synthetic air. Four trained panelists evaluated the chromatographic effluents with four repetitions, registering both the intensity and time of duration of each olfactory stimulus on a scale shown on the computer monitor. They also described to the researcher, the quality of the odor they detected. The evaluation of the odor intensities was carried out using a 10 point hybrid scale, anchored at the left and right extremities by the intensity terms of “none” and “highly”, respectively.

After each analysis, the software known as SCDTI (Cardello, Da Silva, & Damásio, 2003) integrated the data collected, producing an aromagram where higher peaks and/or those with a greater area, indicated the compounds with greater odoriferous importance. A consensus Osmegram was constructed for the condensates, averaging all the peaks detected at least twice by at least two panelists.



The isolates of condensate<sub>11/12</sub> and condensate<sub>11/40</sub> were also analyzed using the same GC operating at the same conditions mentioned above but with the chromatographic column connected to the FID. Thus, by comparing the Osmogram with the chromatogram obtained by the GC analysis, together with the identification of the volatiles by GC–MS, it was possible to identify amongst all the volatiles present in the chromatograms, the odor active ones.

### 3. Results and discussion

#### 3.1. Samples recovered by condensation

Table 1 shows for each step of the process of concentration of the cashew apple juice, the mass of water theoretically evaporated from the juice – calculated from the difference in °Brix between each concentration step – and the mass of water recovered by condensation. It can be seen that the mass of water recovered by condensation falls very close to the interval of  $\pm 1$  standard deviation from the mean of the mass of water theoretically evaporated from the juice. For a study carried out in a pilot plant this indicates the recovery process was efficient and that the large majority of water and volatiles evaporated from the juice during its concentration was effectively recovered by condensation, building validity to the experiment.

Considering all the water recovered by condensation until the juice reached 40 °Brix, approximately 23.20% was recuperated during the 1st step of concentration, when the juice went from 10.6 to 12.05 °Brix, 44.01% during the 2nd step, 64.91% during the 3rd step and 80.18% during the 4th step (Table 1). These values were very close to those initially proposed, which were 20%, 40%, 60% and 80%, respectively.

#### 3.2. Aroma recovery in the condensed samples

Fig. 1 shows the relationship between the intensity of the characteristic cashew apple aroma perceived by the sensory panel in each condensate, and the % water recovered in the respective condensate.

The results shown in Fig. 1 reveal that the intensities of the cashew aroma in the condensates containing 23.2% and 44.0% of recovered water were close to the term “moderate” (4.5) on the sensory scale, decreasing as more water was recovered. Since the aroma present in the condensates came from the volatile compounds recovered in the samples, these results suggest that the greater part of the odoriferous volatiles that confer the cashew-like aroma on the juice was lost when the beverage was concentrated between approximately 10.6 and 14 °Brix (Table 1), corresponding to the first 44% of water evaporated from the juice. In the same way, studies carried out with apple (Bomben et al., 1973; Sulc, 1984), banana (Khalil, 1990), and mango and guava (Ramteke et al., 1990) juices, showed that the majority of the volatiles associated with the characteristic aroma/flavor

of these juices could be recovered by condensation in the first steps of the concentration process.

When the data in Fig. 1 were fitted to a statistical model, it was found that they could be significantly fitted ( $p=0.0004$ ) to the power function curve, which is, in fact, the model that best explains the relationship between chemical stimuli and sensory perception (Da Silva et al., 1994; Moskowitz, 1970; Stevens, 1957, 1961).

#### 3.3. Volatile compounds recovered in the condensed samples

Table 2 shows the concentrations ( $\mu\text{g L}^{-1}$ ) of the volatile compounds identified in each of the five condensates listed in Table 1. As can be seen, 70 volatiles were identified in condensate<sub>11/12</sub>, 51 in condensate<sub>11/14</sub>, and a total of 48 volatiles in condensate<sub>11/19</sub>, condensate<sub>11/28</sub> and condensate<sub>11/40</sub>.

Of the volatiles identified in condensate<sub>11/12</sub>, 39 were esters (55.7% of the total identified), 10 aldehydes, 9 alcohols, 6 ketones, 2 terpenes, 2 ethers, 1 acid and 1 hydrocarbon.

It can be seen that the profiles of the chemical classes identified in the five condensates were highly similar, although the majority of the chemical classes were found in greater concentrations in condensate<sub>11/12</sub> than in the other condensates. For example, the esters, aldehydes and alcohols were found in the concentrations of, respectively,  $580.31 \mu\text{g L}^{-1}$ ,  $39.34 \mu\text{g L}^{-1}$  and  $23.48 \mu\text{g L}^{-1}$  in condensate<sub>11/12</sub>, and in much lower concentrations in condensate<sub>11/14</sub> (respectively  $179.84 \mu\text{g L}^{-1}$ ,  $10.35 \mu\text{g L}^{-1}$  and  $4.96 \mu\text{g L}^{-1}$ ), and in the other condensates (Table 2). These results indicate that, in fact, the chemical classes recognized as having the greatest odoriferous impact on the cashew apple juice aroma, be the esters, alcohols or aldehydes (Garruti et al., 2003), can be recovered together in the first 23.2% of water leaving the juice during its concentration, this representing concentration from approximately 10.6 °Brix to 12.05 °Brix in the present study.

In condensate<sub>11/12</sub> the esters represented 90% of the total mass of volatiles extracted ( $580.31 \mu\text{g L}^{-1}$ ), followed by the aldehydes (6%), alcohols (3.5%) and terpenes (0.5%). Only the esters ethyl propanoate, ethyl butanoate, ethyl 2-methylbutanoate, ethyl isovalerate, isoamyl acetate, ethyl crotonoate and ethyl hexanoate together, represented 80% of the total mass of volatiles present in condensate<sub>11/12</sub>.

In the other condensates the esters also represented almost 90% of the total mass of volatiles present in each one. However, the concentration of these volatiles in the samples continued decreasing as the juice concentration progressed, such that the esters were 12 times more concentrated in condensate<sub>11/12</sub> than in condensate<sub>11/40</sub>.

The following esters were found amongst those identified in greater concentrations in the five cashew apple condensates: ethyl hexanoate, ethyl isovalerate, ethyl crotonoate, ethyl butanoate, ethyl propanoate and ethyl 2-methylbutanoate. These esters were also found in high proportions in the fresh cashew apple juice analyzed by Garruti et al. (2003). Ethyl hexanoate, ethyl isovalerate and ethyl 2-methylbutanoate were also the esters found in the greatest proportions in a cashew apple

**Table 1**

Samples recovered by condensation in each step of the cashew apple juice concentration process, °Brix of the juice at the end of each step, mass of water evaporated (g), mass of water recovered (g) and % water recovered in each step as compared to the total amount recovered up to the point when the juice reached 40 °Brix.

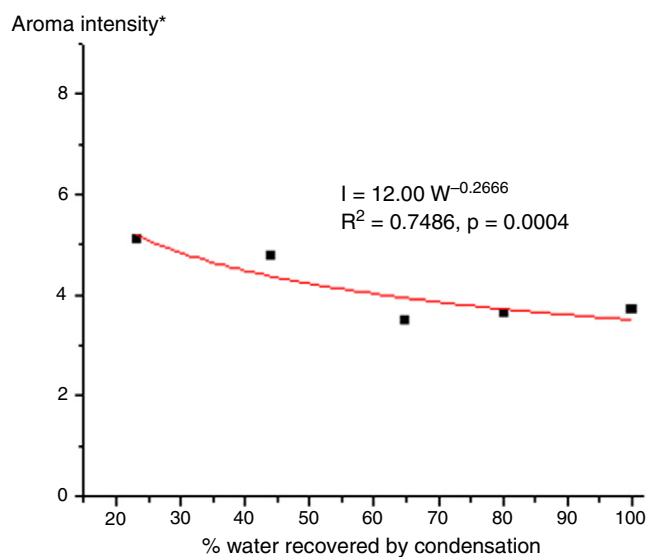
Concentration steps	Samples recovered by condensation in each step	°Brix of juice ( $\pm$ SD) <sup>a</sup>	Mass (g) of water theoretically evaporated ( $\pm$ SD) <sup>b</sup>	Mass (g) of water recovered by condensation ( $\pm$ SD) <sup>c</sup>	% water recovered <sup>d</sup>
Start		10.55 $\pm$ 0.30			
1st step	Condensate <sub>11/12</sub>	12.05 $\pm$ 0.25	495.77 $\pm$ 52.36	581.28 $\pm$ 0.54	23.20%
2nd step	Condensate <sub>11/14</sub>	14.25 $\pm$ 0.39	958.45 $\pm$ 69.56	1104.57 $\pm$ 8.15	44.01%
3rd step	Condensate <sub>11/19</sub>	19.25 $\pm$ 0.11	1619.93 $\pm$ 10.74	1628.89 $\pm$ 16.95	64.91%
4th step	Condensate <sub>11/28</sub>	28.00 $\pm$ 0.14	2207.47 $\pm$ 6.47	2012.11 $\pm$ 22.91	80.18%
5th step	Condensate <sub>11/40</sub>	40.00 $\pm$ 0.07	2595.25 $\pm$ 1.59	2509.61 $\pm$ 13.87	100.00%

<sup>a</sup> Mean and standard deviations (SD) referring to the two repetitions of the juice concentration process.

<sup>b</sup> Mean mass of water evaporated from the juice, and respective standard deviation (SD), calculated from the difference in °Brix between each concentration step.

<sup>c</sup> Mean mass and standard deviations (SD) of water recovered considering the two repetitions of the juice concentration process.

<sup>d</sup> Percent of water recovered in each step relative to the total water recovered until the juice reached 40 °Brix.



**Fig. 1.** Percentage of water recovered in the condensates during the concentration of the cashew apple juice from 11 to 40 °Brix versus the intensity of the characteristic cashew apple aroma present in each condensate. \*Values on the intensity scale: 0 = none; 4.5 = moderate; 9 = strong.  $I$  = aroma intensity,  $W$  = % of water in the condensate.

water phase recovered by a Brazilian industry concentrating cashew apple juice (Sampaio et al., 2011). However in this water phase, the alcohols, not the esters, were the greatest chemical class present in the sample, demonstrating a failure in the aroma recovery process set by the juice producer.

The aldehydes were the second largest class of volatiles found in the condensates, being present in concentrations of  $39.34 \mu\text{g L}^{-1}$  in condensate<sub>11/12</sub>,  $10.79 \mu\text{g L}^{-1}$  in condensate<sub>11/14</sub> and  $4.17 \mu\text{g L}^{-1}$  in condensate<sub>11/40</sub> (Table 2). This indicates that the majority of the aldehydes evaporated off from the cashew apple juice during its concentration could be recovered in the first 23.2% of water leaving the juice.

Amongst the alcohols, which were the third most abundant class of volatiles in the condensates, 3-methyl-1-butanol was present in the greatest concentration, followed by 2-methyl-1-propanol. 3-methyl-1-butanol was also identified as the alcohol found in the greatest proportions in fresh cashew juice (Garruti et al., 2003) and in the water phase analyzed by Sampaio et al. (2011).

In the present study the terpenes appeared as minority compounds, being identified in concentrations of  $0.61 \mu\text{g L}^{-1}$  in condensate<sub>11/12</sub> and  $0.12 \mu\text{g L}^{-1}$  in condensate<sub>11/14</sub>. These results are in agreement with various studies that also identified the terpenes as minority volatiles both in the fresh and processed cashew apple juices (Garruti et al., 2003; Maciel et al., 1986; Valim et al., 2003). In the study carried out by Bicalho et al. (2000), thirteen terpenes were identified as minority volatiles, found in concentrations close to  $6 \mu\text{g kg}^{-1}$  in the volatiles present in the pseudofruit.

The ketones were also identified as minority volatiles, with a concentration of  $0.1 \mu\text{g L}^{-1}$  in condensate<sub>11/12</sub>, a result similar to that found by other researchers in fresh cashew apple juice (Bicalho et al., 2000; Garruti et al., 2003).

Finally, with respect to acids, only condensate<sub>11/12</sub> presented detectable amounts, and even so, only 2-methylbutanoic acid ( $<0.01 \mu\text{g L}^{-1}$ ). These results are similar to those reported by Garruti et al. (2003), Valim et al. (2003) and Bicalho et al. (2000), who also detected the presence of this class of volatiles as minority in cashew apple juice.

Overall, the data shown in Table 2 indicated that the most important compounds for the aroma and flavor of the fresh cashew apple juice, notably the esters, alcohols and aldehydes, could be recovered during the first step of juice concentration, which in this study was when the juice was concentrated from 10.6 to 12 °Brix.

### 3.4. Dynamics of the recovery of volatiles during cashew apple juice concentration

Fig. 2 shows the recovery dynamics of each chemical class of volatiles that left the juice before it reached a concentration of 40 °Brix. As can be seen, the kinetic model that best fitted the experimental data was the power function, which was also the case for the fit of the sensory data (Fig. 1). For each chemical class, the predictive models generated by the use of this function produced determination coefficients ( $R^2$ ) between 0.98 and 0.99, showing a high level of significance ( $p < 0.001$ ).

Considering the exponential coefficients of the fitted models (Fig. 2), it can be seen that the three classes of volatiles presented very close recovery rates throughout the concentration of the juice, with the recovery rate of the alcohols being slightly better than that of the esters and aldehydes. Similarly, Ramteke et al. (1990) showed that the recovery rates of the esters, alcohols and aldehydes were very similar during the concentration of mango juice, which was not the case with pineapple juice, for which they found lower recovery rates for the esters as compared to the alcohols and aldehydes.

Fig. 2 makes it clear that by recovering the first 23% of water evaporated from the juice, that is, that lost when the juice was concentrated from 10.6 to 12.05 °Brix, a condensate was obtained with the greatest concentrations of the volatiles from all the chemical classes. This is in agreement with the results generated by the trained sensory panel (Fig. 1), which, on evaluating the aroma of the condensates, showed that the condensate originating from the first 23% of water evaporated from the juice, possessed the greatest intensity of cashew apple aroma.

Various studies achieved similar results, reporting that in order to recover the greater part of the volatiles associated with the characteristic aroma/flavor of juices, such as that of apple (Bomben et al., 1973; Sulc, 1984), banana (Khalil, 1990), plum (Sulc, 1984), and mango and guava (Ramteke et al., 1990), one must recover by condensation the first 10 to 35% of the water evaporated off from the juice during its concentration. In the study of Ramteke et al. (1990) it was evident that about 90% of the esters, aldehydes and alcohols present in mango juice were recovered in the first 25 to 35% of water evaporated off from the juice during its concentration. However, in the case of pineapple juice, the authors showed that in order to obtain a condensate containing about 90% of the esters initially present in the fresh juice, more than 75% of the water evaporated off had to be recovered, indicating that different fruits can present distinct dynamics with respect to the loss and recovery of volatiles, which must be known if required to recover the aromas lost during industrial juice processing.

### 3.5. Odoriferous importance of the volatiles recovered by condensation

Table 3 shows the odoriferous importance of the volatiles present in the condensate containing the first 23.2% of recovered water (condensate<sub>11/12</sub>) and in the condensate containing 100% of the water recovered from the juice (condensate<sub>11/40</sub>). In this table, volatiles presenting intensities equal or above a score of 4 on the 10-point scale were considered as being those with the greatest odoriferous impact on the condensates.

Thirty volatiles with odoriferous impact were perceived in the chromatographic effluents from condensate<sub>11/12</sub>, of which 29 were identified by GC–MS. Of these, 21 were esters, 2 aldehydes, 2 alcohols, 2 ketones, 1 terpene and 1 hydrocarbon. Thus the esters were the chemical class presenting the greatest odoriferous impact on condensate<sub>11/12</sub>, representing 76% of the total area of the aromagram. The majority of these had already been identified as volatiles contributing fruity and sweet notes of importance to the aroma and flavor of cashew apple juice (Garruti et al., 2003; Maciel et al., 1986) or for the water phase produced by the Brazilian industry during the concentration of cashew apple juice (Sampaio et al., 2011).

Of the 5 volatiles with the greatest odoriferous impact in the condensate containing the first 23.2% of water recovered (condensate<sub>11/12</sub>), 4

**Table 2**  
Volatile compounds identified and quantified in the five cashew apple condensates.

LRI DB-Wax	LRI DB-5	Compounds identified grouped according to chemical class	Condensate <sub>11/12</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/14</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/19</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/28</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/40</sub> ( $\mu\text{g L}^{-1}$ )
		<i>Esters</i>	580.31	179.84	98.14	95.78	46.01
931		Ethyl propanoate <sup>b</sup>	30.59	11.52	7.20	6.63	3.45
952	721	Ethyl 2-methylpropanoate <sup>b</sup>	2.03	0.79	0.42	0.47	0.09
1002		Methyl butanoate <sup>b</sup>	2.69	0.87	0.30	0.33	0.08
1007		Ethyl acrylate <sup>b</sup>	0.26	nd	nd	nd	nd
1021		Methyl 2-methylbutanoate <sup>b</sup>	0.86	0.28	0.03	0.03	<0.01
1023		2-Methylpropyl acetate <sup>b</sup>	0.23	nd	nd	nd	nd
1028	802	Methyl isovalerate <sup>a</sup>	8.91	2.36	0.98	1.16	0.41
1045	820	Ethyl butanoate <sup>a</sup>	52.49	17.48	9.82	9.55	4.62
1059	860	Ethyl 2-methylbutanoate <sup>a</sup>	31.57	9.33	4.47	4.53	2.03
1077	867	Ethyl isovalerate <sup>a</sup>	137.47	49.25	27.23	26.83	14.28
1078	828	Butyl acetate <sup>b</sup>	nd	0.18	nd	nd	nd
1111		Ethyl 2-methyl-2-propenoate <sup>b</sup>	0.85	0.15	<0.01	0.06	<0.01
1127	884	Isoamyl acetate <sup>a</sup>	14.03	3.37	1.69	1.69	0.78
1139		Ethyl pentanoate <sup>b</sup>	5.85	1.37	0.69	0.68	0.29
1158		3-Methylpropyl butanoate <sup>b</sup>	1.69	0.39	0.26	0.17	tr
1166	857	Ethyl crotonoate <sup>b</sup>	89.34	22.37	12.66	12.87	5.64
1181	1002	Ethyl 3-methylpentanoate <sup>b</sup>	9.94	2.50	1.21	1.17	0.53
1188	934	Methyl hexanoate <sup>b</sup>	3.28	0.72	0.35	0.34	<0.01
1192		Ethyl 4-methylpentanoate <sup>b</sup>	3.03	0.74	0.33	0.34	0.15
1221		Butyl butanoate <sup>b</sup>	0.30	nd	nd	nd	nd
1226		Ethyl 3-methyl-2-butenolate <sup>b</sup>	0.58	nd	nd	nd	nd
1242	967	Ethyl hexanoate <sup>b</sup>	158.45	47.64	26.19	25.19	11.78
1260	959	Ethyl <i>trans</i> -2-pentenoate <sup>b</sup>	1.63	0.29	0.13	0.13	tr
1268		Isopentyl 2-methylpropanoate <sup>b</sup>	2.21	0.60	nd	nd	nd
1293		2-Methylbutyl-3-methylbutanoate <sup>b</sup>	1.41	0.37	0.20	0.17	<0.01
1298	1004	Ethyl <i>cis</i> -3-hexenoate <sup>b</sup>	2.27	0.44	0.21	0.21	<0.01
1302	1007	Ethyl <i>trans</i> -3-hexenoate <sup>b</sup>	0.42	nd	nd	nd	nd
1313		3-Hexenyl acetate <sup>b</sup>	0.12	nd	nd	nd	nd
1321		Ethyl 4-methyl-2-pentenoate <sup>b</sup>	0.09	nd	nd	nd	nd
1333	1094	Ethyl heptanoate <sup>b</sup>	1.22	0.43	0.03	0.23	0.31
1342	1051	Ethyl 2-hexenoate <sup>b</sup>	5.38	1.26	0.70	0.70	0.31
1433	1196	Ethyl octanoate <sup>b</sup>	9.36	4.79	2.93	2.30	1.26
1472	1187	Ethyl 4-octenoate <sup>b</sup>	0.56	0.21	nd	nd	nd
1554		Ethyl <i>trans</i> -2-octenoate <sup>b</sup>	0.61	<0.01	<0.01	<0.01	<0.01
1663	1169	Ethyl benzoate <sup>b</sup>	0.52	0.14	0.11	<0.01	<0.01
1749		Phenylethyl acetate <sup>b</sup>	0.07	nd	nd	nd	nd
		<i>Alcohols</i>	23.48	4.96	2.54	2.96	1.20
1103		2-Methyl-1-propanol <sup>a</sup>	5.10	1.29	0.62	0.78	0.32
1153		Butanol <sup>b</sup>	0.43	nd	nd	nd	nd
1216		3-Methyl-1-butanol <sup>b</sup>	15.65	3.45	1.92	2.18	0.88
1359	876	Hexanol <sup>b</sup>	0.57	<0.01	nd	nd	nd
1455		1-Octen-3-ol <sup>b</sup>	0.31	nd	nd	nd	nd
1460	976	Heptanol <sup>b</sup>	0.21	nd	nd	nd	nd
1569		Octanol <sup>b</sup>	0.98	0.22	nd	nd	nd
1670		Nonanol <sup>b</sup>	0.23	nd	nd	nd	nd
		<i>Aldehydes</i>	39.34	10.35	6.89	6.05	4.17
1087		Hexanal <sup>a</sup>	16.71	4.16	2.01	2.46	1.56
1144		2-Methyl-4-pentenal <sup>b</sup>	0.75	nd	nd	nd	nd
1184		Heptanal <sup>b</sup>	1.64	<0.01	0.54	<0.01	<0.01
1285		Octanal <sup>a</sup>	4.04	1.04	0.79	0.65	0.58
1386	1102	Nonanal <sup>b</sup>	11.66	4.48	3.55	2.94	2.03
1450		Furfural <sup>b</sup>	<0.01	nd	nd	nd	nd
1505	979	Benzaldehyde <sup>b</sup>	2.88	0.67	<0.01	<0.01	<0.01
1528	1160	<i>Trans</i> -2-nonenal <sup>b</sup>	1.16	nd	nd	nd	nd
1581		2,6-Nonadienal <sup>b</sup>	0.50	nd	nd	nd	nd
		<i>Ketones</i>	0.10	0.07	0.03	0.05	<0.01
1064		2,3-Pentanedione <sup>b</sup>	<0.01	nd	nd	nd	nd
1279		3-Hydroxy-2-butanone <sup>a</sup>	0.1	0.07	0.03	0.05	<0.01
1638	1063	Acetophenone <sup>b</sup>	<0.01	<0.01	<0.01	<0.01	<0.01
1782		Geranyl acetone <sup>b</sup>	<0.01	<0.01	<0.01	<0.01	<0.01
		<i>Terpenes</i>	0.61	0.12	<0.01	<0.01	<0.01
1245		$\gamma$ -Terpinene <sup>b</sup>	0.20	0.04	<0.01	<0.01	<0.01
1598	1178	4-Terpineol <sup>b</sup>	0.41	0.08	<0.01	<0.01	<0.01
		<i>Acids</i>					
1675		2-Methylbutanoic acid <sup>a</sup>	<0.01	nd	nd	nd	nd
		Co-eluting compounds	–	–	–	–	–
977	c	Propyl acetate <sup>a</sup> + 2,3-butanedione <sup>b</sup>	nq	nq	nq	nq	nq
1134	c	2,3-Hexanedione <sup>b</sup> + methyl 3-methylpentanoate <sup>b</sup>	nq	nq	nq	nq	nq
1317	c	Propyl hexanoate <sup>b</sup> + Propyl tiglate <sup>b</sup>	nq	nq	nd	nd	nd
1492	c	Decanal <sup>b</sup> + 2-Ethyl-1-hexanol <sup>b</sup>	nq	nq	nq	nq	nq
		<i>Ethers</i>	–	–	–	–	–
1084		1,1-Diethoxy-3-methyl-butane <sup>b</sup>	<0.01	nd	nd	nd	nd
1115		1,1-Ethoxyethoxypentane <sup>b</sup>	<0.01	nd	nd	nd	nd

Table 2 (continued)

LRI DB-Wax	LRI DB-5	Compounds identified grouped according to chemical class	Condensate <sub>11/12</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/14</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/19</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/28</sub> ( $\mu\text{g L}^{-1}$ )	Condensate <sub>11/40</sub> ( $\mu\text{g L}^{-1}$ )
		Hydrocarbons	2.30	0.58	0.27	0.25	0.12
1253		Benzocyclobutene <sup>b</sup>	2.30	0.58	0.27	0.25	0.12

nd: not detected; nq: not quantified since co-eluted with another compound in DB-Wax column.

<sup>a</sup> Positively identified compound.

<sup>b</sup> Compound identified by MS and the linear retention index.

<sup>c</sup> LIR in DB-5 column (propyl acetate LRI = 799, 2,3-Butanedione LRI = 723, 2,3-hexanedione LRI = 807, methyl 3-methylpentanoate LRI = 889, propyl hexanoate LRI = 1091, propyl tiglate LRI = 1037, Decanal LRI = 1200, 2-ethyl-1-hexanol LRI = 1031).

were esters and one a terpene, as follows: ethyl crotonoate, ethyl isovalerate, ethyl hexanoate and 4-terpineol. Together, these five compounds contributed about 40% of the total area of the aromagram of condensate<sub>11/12</sub>.

Only one terpene, 4-terpineol, was sensorially detected in the condensate containing the first 23.2% of water recovered (condensate<sub>11/12</sub>), being associated with aromatic notes similar to the fruit and grass. Each compound contributed 5.37% of the total area of the aromagram of the condensate, thus being classified as an important contributor to the aroma of this sample. However, in general, the contribution of the terpenes to the aroma of the two condensates analyzed (Table 3) was not very significant, compared to that of the esters.

The alcohols, aldehydes and ketones also failed to present much odoriferous impact on the chromatographic effluents of condensate<sub>11/12</sub> – together these odoriferous classes contributed about 9% of the total area of the aromagram.

Table 3 shows that 18 odoriferous volatiles were perceived by the sensory panel in the condensate containing 100% of the water recovered from the juice (condensate<sub>11/40</sub>), of which 13 were esters, 3 aldehydes, 1 terpene and 1 ketone. With the exception of octanal, which was only perceived in condensate<sub>11/40</sub>, all the odoriferous volatiles detected in

this condensate were also detected in condensate<sub>11/12</sub>, but they were perceived with lower aroma intensity in condensate<sub>11/40</sub>, which is compatible with the fact that they were present in lower concentrations in this condensate. These results indicate that the recovery of all the water and volatiles evaporated from the cashew apple juice up to the point where it reached 40 °Brix just contributed to diluting the material recovered, reducing the odoriferous power of the sample.

#### 4. Conclusions

The results of this study indicate that a more efficient recovery of the volatiles important to the cashew apple aroma and flavor can be obtained condensing the first 23% of the water evaporated off the juice during the concentration process; in this study when the beverage was concentrated from 10.6 to approximately 12 °Brix. The recovery of the volatiles after this point appears to only exert the effect of diluting the recovered material, not contributing to any improvement in the sensory quality of the condensate. The esters were the compounds found in greater concentrations in the material recovered by condensation. They were also the volatiles with the greatest odoriferous impact on the material recovered,

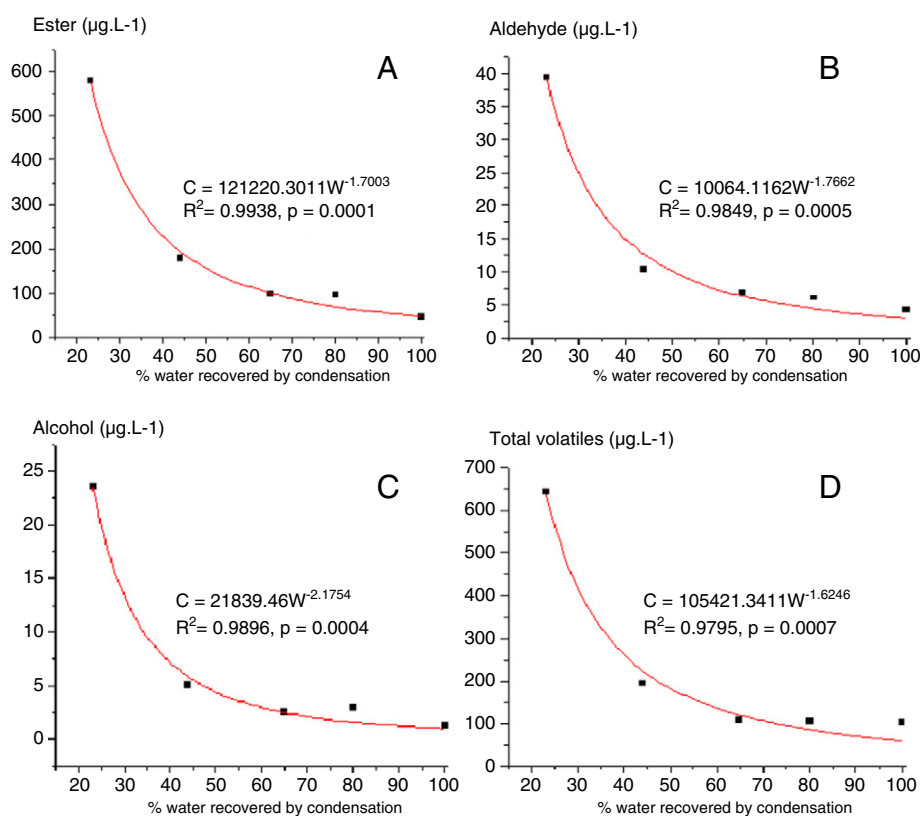


Fig. 2. Percentage of water recovered in the condensates during the concentration of the cashew apple juice from 11 to 40 °Brix versus the concentrations of esters (A), aldehydes (B) alcohols (C) and total volatiles (D) in each condensate. C = concentration of each chemical class, W = % of water in the condensate.



**Table 3**  
Aroma volatiles detected by the sensory panel in condensate<sub>11/12</sub> and condensate<sub>11/40</sub> (n<sub>1</sub> = 4 panelists; n<sub>2</sub> = 4 repetitions).

Compounds	LRI Osme <sup>a</sup>	Aroma intensity <sup>b</sup>		% Aromagram area <sup>c</sup>		Aroma quality as described by the sensory panel
		Cond <sub>11/12</sub>	Cond <sub>11/40</sub>	Cond <sub>11/12</sub>	Cond <sub>11/40</sub>	
Esters				76.27	83.32	
Ethyl propanoate	929	2.2	–	0.82	–	Sweet/fruit
Ethyl 2-methylpropanoate	944	2.6	1.4	2.23	1.93	Pungent/fruit
Propyl acetate	960	3.4	1.8	2.89	2.11	Sweet, fruit
Methyl butanoate	982	1.5	1.1	2.39	1.28	Fruit, grass, tutti-fruity
Ethyl acrylate	989	2.2	–	3.44	–	Fruit, green, citric
Methyl 2-methylbutanoate	994	3.0	2.4	1.73	10.40	Fruit, cashew, sweet
2-Methylpropyl acetate	1001	3.3	–	3.85	–	Grass, cashew, fruit
Ethyl butanoate	1016	4.7	2.5	9.07	4.60	Cashew, pineapple, strawberry
Ethyl 2-methylbutanoate	1028	3.7	3.4	4.26	22.10	Cashew, pineapple, lemon
Ethyl isovalerate	1048	4.4	2.9	6.21	12.72	Fruit, grass, sweet
Ethyl 2-methyl-2-propenoate	1112	2.7	–	3.38	–	Grass, fruit
Isoamyl acetate	1128	1.6	–	2.17	–	Fruit, banana, sweet
Propyl 3-methyl-butanoate	1161	1.1	–	0.86	–	Flower, fruit, grass
Ethyl crotonoate	1171	5.1	3.5	10.25	8.60	Cashew, fruit, sweet
Ethyl 3-methyl-pentanoate	1183	1.5	1.1	2.32	2.16	Fruit, sweet, grass
Ethyl hexanoate	1227	4.3	3.3	7.06	7.04	Fruit, flower
Ethyl cis-3-hexenoate	1290	2.9	0.9	2.33	1.62	Grass, citric, pungent
Ethyl octanoate	1426	2.7	0.8	2.74	1.44	Grass, fruit, earth
Ethyl 2-octenoate	1537	3.2	2.5	4.10	7.32	Earth, grain, mold
Phenyl ethyl acetate	1747	1.4	–	4.17	–	Pungent, cheese, mold
Aldehydes				3.23	6.03	
Hexanal	1063	3.0	1.4	2.08	2.89	Grass, vanilla
Octanal	1270	–	1.5	–	2.76	Lemon, grass
Benzaldehyde	1498	1.3	0.6	1.15	0.38	Grass, earth, grain
Alcohols				3.33		
2-Methyl-1-propanol	1078	1.2	–	1.31	–	Green, mold, earth
Hexanol	1366	1.9	–	2.02	–	Grass, eucalyptus
Terpenes				5.37	3.01	
4-Terpeneol	1594	4.3	2.1	5.37	3.01	Fruit, grass
Ketones				2.91	7.60	
2,3-Butanedione	967	3.1	3.6	2.91	7.60	Fruit, cashew, sweet
Hydrocarbons				1.26		
Cardene	1231	1.2	–	1.26	–	Pineapple, cashew
Co-eluting compounds				3.23		
2,3-Hexanedione + Methyl 3-methylpentanoate	1134	3.3	–	3.23	–	Sweet, green, fruit
NI				4.40	–	
NI	974	2.4	–	4.40	–	Fruit, vanilla, strawberry

NI, not identified compound.

<sup>a</sup> Linear retention index calculated from the time corresponding to the maximum intensity of the odor peak (DB-Wax column).

<sup>b</sup> Average intensity at the consensual aromagram (0 = none; 10 = extreme).

<sup>c</sup> Percentage of the total aromagram area.

being associated by the sensory panel with the fruity and cashew-like aroma notes present in the condensates.

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