



ORIGINAL ARTICLE

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Behaviour of volatile aromatic compounds during distillation of cachaça in a three-body pot still

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Abstract

Why was the work done: The Brazilian spirit cachaça is distilled from fermented sugarcane juice. The equipment and distillation method influence the volatilisation of aromatic compounds, which affects the chemical composition and odour quality of the spirit. Distillation in a three-body pot still increases yield and productivity, but the impact of this process on cachaça aroma has received little attention. The work reported here considers the distribution of volatile compounds during distillation of cachaça in a three-body pot still, together with the composition and odour activity values (OAV).

How was the work done: Fermented sugarcane juice was distilled in a three-body pot still and 13 volatile compounds quantified using gas chromatography with flame ionisation detection and gas chromatography–mass spectrometry. The OAV of the compounds in the heart fraction of cachaça were calculated. The chemical and sensory quality of cachaça from a three-body pot still was compared to single and double-distilled spirit.

What are the main findings: The volatile compounds were classified as 'light', 'intermediate' and 'heavy', according to their volatility and distribution. Initially, aldehydes, esters and higher alcohols predominated, with acetic acid, ethyl carbamate and furfural predominant in the final fractions. Of the analysed compounds, 66% contributed to the aroma of cachaça, notably - with an OAV > 10 - ethyl acetate and 3-methyl-1-butanol.

Why is the work important: Distillation in the three-body pot produced high-quality cachaça, with chemical parameters within the limits of the Brazilian legislation. These results provide a reference for evaluating the quality of cachaça and for research of the odour activity values in distilled beverages.

Keywords

cachaça; distillation; three-body pot still; volatilisation; retort; sugar cane spirit; odour activity values

Introduction

Cachaça is a regulated spirit in Brazil, produced from sugar cane, with an ethanol content between 38% and 48% by volume at 20°C (Brazil 2022). Production involves the fermentation of sugar cane juice by wild or pure strains of yeast, followed by distillation in pot stills or continuous columns. A standard pot still configuration has not been established, and aging is not a legal requirement. Pot stills include the simple model, with a boiler, hat, neck and condenser, and those with rectifier columns or a post-hat dephlegmator (Alcarde 2024). Most producers choose single distillation, although some adopt double distillation (Alcarde 2024), a common practice with malt whiskey in Scotland (Nicol 2014).

To optimise the yield and productivity of the distillation process, a three-body pot still was developed, consisting of a boiler (lower body), a distiller (intermediate body) and a preheater (upper body) (Alcarde 2024). The steam generated during the distillation of the wine in the boiler is forwarded to the distiller, which in contact with the residual wine, causes bubbling, resulting in steam condensation, wine heating and enrichment in ethanol. As the process continues, the wine in the distiller reaches boiling point and the steam is transferred to the preheater, where it exchanges heat with the wine, increasing temperature and condensation. The distillate is collected until the alcohol by volume (ABV) declines to 50% when the boiler heating is stopped and the liquid discharged. The remaining liquid in the distiller is transferred to the boiler, and the preheated wine is transferred to the distiller, where a new batch of wine is added, starting a new distillation cycle.

Although the final cut of the 'heart' fraction has an ABV of 50% (at the condenser outlet), the liquid in the boiler (lower body) is ethanol-free, as this is partially recovered in the distillate with the remainder in the distiller (intermediate body), thereby increasing the distillation yield. In this system it is not necessary to recover the 'tail' fraction and the productivity of the process is higher. Although the evolution of the volatile aromatic compounds during distillation of cachaça using single and double distillation has been reported (Alcarde et al. 2010; 2012), data on distillation using a three-body pot still and impact

on the quality of cachaça has not been reported.

Volatile compounds are influenced by liquid-steam balance during distillation. Volatility depends on the boiling temperature of the pure compound, solubility in water and ethanol and molecular weight (Zanghelini et al. 2024b). Those compounds classified as 'light', with low molecular weight (acetaldehyde and ethyl acetate), are distilled at the beginning of the process, having relatively high volatility. The 'intermediate' compounds (methanol and ethyl lactate) are distilled throughout the process, being soluble in ethanol and totally or partially soluble in water. The compounds considered as 'heavy' (acetic acid and furfural) are preferentially distilled at the end of the process, as they are soluble in water and have higher boiling points (Puentes et al. 2018). Despite having a high boiling point, higher alcohols appear throughout the distillation in pot stills (Léauté 1990).

The evolution of volatile aromatic compounds during distillation in pot stills has been reported for sugar cane spirit (Alcarde et al. 2010; Nóbrega et al. 2011; Alcarde et al. 2012), cognac (Douady et al. 2019) and plum brandy (Balcersek et al. 2017). Nonetheless, not all the volatile compounds present in distilled spirits contribute to the perception of aroma, as they must be at levels above their odour detection threshold (ODT) to be perceptible. The odour activity value (OAV) has been employed to evaluate the odour quality in rum (Franitza et al. 2016a; Nicolotti et al. 2019), whiskey (Poisson and Schieberle 2008), baijiu (Liu and Sun 2018), melon brandy (Zhang et al. 2022) and cognac (Zanghelini et al. 2024b), but not cachaça.

To date, information on the chemical composition, and the aroma profile of cachaça using three-body pot stills is limited. Given that distillation and reflux rates vary depending on the type of pot still used which influences the aromatic composition of the distillate (Balcersek et al. 2017), it is necessary to study the behaviour of volatile compounds and chemical composition of cachaça from distillation in three-body pot stills. This work reports the volatile compounds responsible for the aroma and flavour of cachaça from distillation in a three-body pot still, the composition of the distillate and the OAV of these compounds.

Materials and methods

Production of cachaça using a three-body pot still

This study was conducted at the distillery of the Department of Food Science and Technology at the University of São Paulo. The preparation of the cane juice and fermentation were carried out at a laboratory scale. The juice was extracted from sugar cane variety SP 81-3250 in a Maqtron M-730 stainless steel mill (Joaçaba, SC, Brazil) and adjusted to 18° Brix by diluting with drinking water. Fermentation was performed in 750 L stainless steel tanks using *Saccharomyces cerevisiae* DistilaMax CN strain (Lallemand Biofuels and Distilled Spirits, Milwaukee, WI, USA). Fermentation took place for 40 h at 30°C and was complete once all the fermentable sugars were consumed. The fermented must (10.1% ABV) was distilled in a copper three-body pot still, with each body having a capacity of 150 L (Alambiques Santa Efigênia Ltda, Itaverava, MG, Brazil) (Figure 1).

For the first distillation, 450 L of wine were equally divided into the three bodies (150 L); preheater at the top, the distiller in the middle and the boiler at the bottom. The boiler uses

electrical resistance heating, with the steam from the boiler bubbled into the distiller wine, heating and enriching it with ethanol and other volatile compounds. As the wine in the distiller is boiled, steam is emitted which condenses preheating the wine in the preheater forming the distillate after passage through the cooler.

The first distillation the boiler receives is the whole wine; with the initial three batches enabling the three-body pot still to operate in a quasi-stationary state. The experiment was in quintuplicate, with five consecutive distillations performed.

For each batch the 'head' fraction of the distillate (3 L, corresponding to 2% of the intermediate body volume) was separated. The 'heart' fraction (22 L) was collected until reaching ~50% ABV (measured by an alcoholmeter) in the condenser outlet, when the heating was turned off. The ABV in cachaça from this fraction was ~58%.

With the next batch of distillation, the distilled wine in the lower tank was evacuated, the wine from the distiller transferred to the boiler, with the heated wine in the preheater moved to the distiller body and fresh wine (150 L) in the upper preheater.

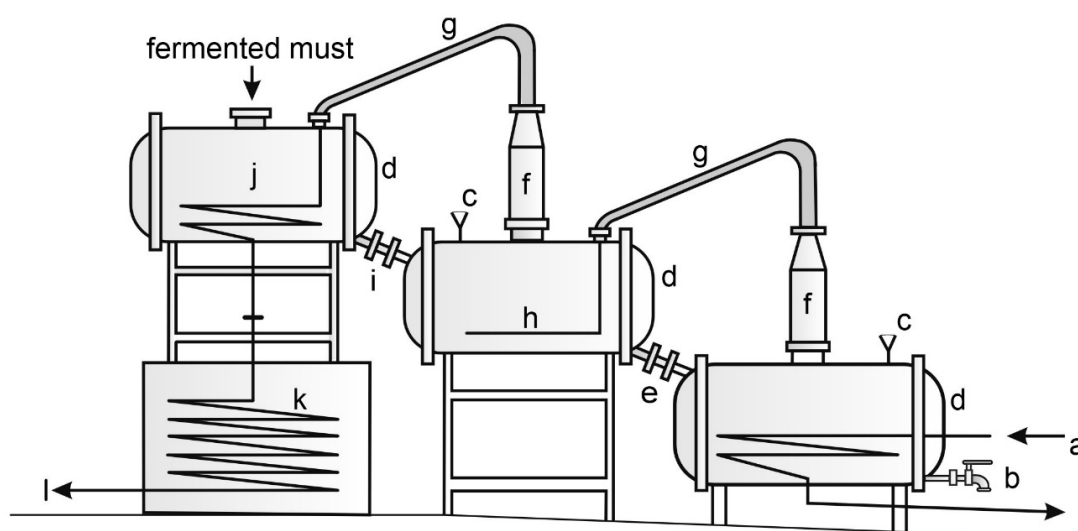


Figure 1. Schematic of a three-body pot still - (a) electrical resistance heating inlet; (b) stillage discharge; (c) pressure equalising valve; (d) liquid level inside the body; (e) flow register from the intermediate body (distiller) to the lower body (boiler); (f) hat; (g) neck; (h) perforated tube; (i) flow register from the upper body (preheater) to the intermediate body (distiller); (j) condensing coil; (k) cooling coil; (l) spirit outlet (Alcarde 2024).

Sampling

To study the distribution of volatile compounds throughout the distillation process of cachaça in the three-body pot still, distillate fractions were collected every 5 min. In all, 15 fractions were collected from each distillation, with an initial fraction from the 'head' and the subsequent fractions from the 'heart' fraction. A total of 75 fractions were collected.

Single and double distillation of cachaça

The chemical and sensory quality of cachaça produced using the three-body pot was compared with cachaça produced using single and double distillation in a pot still (Bortoletto et al. 2015). The single distilled cachaça is the liquid collected after the 'head' fraction (1.5% of the boiler useful volume) with an ABV up to 40% in the condenser outlet. In the double distillation process, the first distillation extracts the ethanol from the wine ('phlegm'), which then undergoes a second distillation. The double distilled cachaça is collected after the 'head' fraction (1% of the boiler useful volume), until the distilled spirit achieves 60% ABV in the condenser outlet. Both single and double distilled cachaça samples were produced in triplicate.

Chemicals and reagents

Ethanol (99.9% (v/v), chromatographic grade) and acetaldehyde, ethyl acetate, ethyl lactate, ethyl carbamate, methanol, acetic acid, furfural, 1-propanol, 2-methyl-1-propanol, 3-methyl-1-butanol, 1-butanol and 2-butanol (all of chromatographic grade with purity > 99%) were purchased from Merck (Darmstadt, Germany). Demineralised water (resistivity 18.2 MΩ-cm at 25°C) was obtained from a Milli-Q purification system (Millipore Waters, Molsheim, France).

Chemical analysis

The volatile compounds as defined by Brazilian legislation for cachaça (Brazil 2022) were analysed. Ethanol was measured using a digital hydrometer DMA-4500 (Anton-Paar GmbH, Graz, Austria). Analysis of acetaldehyde, ethyl acetate, methanol, acetic acid, furfural, higher alcohols (1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol), 1-butanol and 2-butanol used gas chromatography

(GC) in a Shimadzu QP-2010 Plus system (Tokyo, Japan) with a flame ionisation detector (FID). Aliquots (1 µL) were automatically injected into the chromatographic system, equipped with a Stabilwax-DA column (cross-linked carbowax polyethylene glycol, film thickness 30 m × 0.18 mm × 0.18 µm). Analyses were performed at a split ratio of 1:20. Nitrogen was the carrier gas at a flow rate of 1.5 mL/min and pressure of 25.4 kPa. The injector and detector temperatures were at 220°C. The oven temperature program was 40°C for 4 min, with an increase to 120°C at 20°C/min, maintained for 1 min, increasing to 220°C at 25°C/min and held for 4 min (Bortoletto and Alcarde 2013).

Analysis of ethyl lactate was by GC using an Agilent 7890A GC system (Santa Clara, CA, USA) with an FID, equipped with an Agilent CP-Wax 57 CB column (50 m × 0.25 mm × 0.2 µm). Hydrogen was the carrier gas at a constant flow of 4 mL/min. The injector temperature was at 190°C and the injection volume was 1 µL. The initial oven temperature was held at 35°C for 8 min, with an increase of 6°C/min up to 100°C, an increase of 10°C/min up to 200°C which was maintained for 8 min. The temperature of the FID was set at 220°C (Carvalho 2025).

Ethyl carbamate was analysed by GC using a Shimadzu QP-2010 Plus system (Tokyo, Japan) with a mass spectrometry (MS) detector model GCMS-QP2010 Plus, applying electronic impact as the ionisation source with an ionisation energy of 70 eV. A polar phase capillary column (esterified polyethylene glycol) HP-FFAP (50 m × 0.20 mm × 0.33 µm stationary phase film thickness) was used. The injector and detector interface temperatures were 230 and 240°C, respectively. The oven temperature program was 90°C for two min, rising to 150°C at a rate of 10°C/min, followed by an increase up to 220°C at a rate of 40°C/min and then held at this condition for 2 min. The injected volume was 1 µL in automatic splitless mode. Helium was the carrier gas at a flow of 1.2 mL/min (30 cm/s). Selected ion monitoring acquisition was used, monitoring the m/z 62 ions for ethyl carbamate (Alcarde et al. 2012).

As the ethanol content influences the volatilisation of compounds, the samples was adjusted to 40% ABV before analysis. The compounds were quantified by reference to

Table 1. Volatile aromatic compounds and contaminant congeners in cachaça, from distillation in a three-body pot still, quantified using gas chromatography with flame-ionisation detection and gas-chromatography-mass spectrometry.

Compound	RT (min)	LD*	LQ*	Range*	a	b	r ²
Volatile congeners							
Acetaldehyde	0.30	0.071	0.213	7.5–37.5	5.5911	-1.0211	0.9959
Ethyl acetate	1.42	0.058	0.174	12.5–62.5	2.8789	0.9069	0.9997
Ethyl lactate	2.55	0.066	0.198	1.5–7.5	2.4444	0.6158	0.9998
1-propanol	4.44	0.039	0.117	37.5–187.5	2.0469	-0.2449	0.9998
2-methyl-1-propanol	5.23	0.015	0.045	12.5–62.5	1.7259	-0.1719	0.9998
3-methyl-1-butanol	6.71	0.016	0.048	50–250	1.6749	9.1059	0.9998
Acetic acid	19.16	0.531	1.593	37.5–187.5	5.4253	4.1166	0.9997
Contaminant congeners							
Methanol	1.61	0.093	0.279	5–25	4.1398	-0.1626	0.9998
2-butanol	4.03	0.048	0.144	2.5–12.5	1.9171	-5.1088	0.9999
1-butanol	5.98	0.073	0.219	0.75–3.75	1.1163	-1.8591	0.9998
Ethyl carbamate	10.16	0.181	0.543	50–500	64.713	1241.69	0.9991

Concentration - with the exception of ethyl carbamate ($\mu\text{g/L}$), values (*) are as $\text{mg}/100 \text{ mL}$ anhydrous alcohol (99.5–99.9% ethanol). RT = retention time; LD = limit of detection; LQ = limit of quantification; a, b, r^2 are the correlation coefficients of the analytical curves in 40% v/v ethanol.

standard curves from six concentration points. All analyses using GC-FID and GC-MS were in triplicate for each sample. The calculation of the detection and the quantification limits followed Currie (1999) and were based on the signal-to-noise ratio of the chromatograms (Table 1).

Sensory analysis

Sensory assessment of cachaça samples was performed using the Buxbaum model of positive ranking, based on four sensory experiences rated to a maximum of 20 points (Pielech-Przybylska et al. 2016). The panel comprised nine tasters, all experienced in cachaça sensory testing, who in order of sensory evaluation gave scores for colour (0–2), clarity (0–2), aroma (0–4) and taste (0–12). All cachaça samples (single, double and three-body distilled) were diluted to 40% ABV before sensory analysis. The samples (10 mL) were randomly given to tasters in transparent cups marked with random three-digit numbers and covered with Petri dishes. Water was used for cleansing the palate between tests. Evaluation was between 10 and 11 am, at room temperature (22–25°C), and under white light.

Data analysis

Statistical analyses were performed using analysis of variance (one-way ANOVA) using the Statistical Analysis System software, version 9.3. The mean of the results was compared using the Tukey's test ($p < 0.05$). To determine the degree of

differentiation between the behaviour and the final content of volatile aromatic compounds in cachaça, principal component analysis (PCA) was performed using the free software PALaeontological STatistics (PAST®), version 4.03c (Hammer et al. 2001). The real contribution of volatile compounds to aroma was estimated by OAVs, calculated from the concentration in the samples divided by their perception thresholds from the literature (Niu et al. 2020; Zanghelini et al. 2024b). A volatile aromatic compound with an OAV >1 is considered odorous.

Results and discussion

Concentration and profile of volatile aromatic compounds during distillation

The content of volatile aromatic compounds during distillation in a three-body pot still are reported in Table 2 and summarised in a heatmap (Figure 2). This shows the relationship between compounds and distillation time (blue = higher intensity, red = lower intensity) and highlights temporal patterns of the different distillate fractions. Ethyl acetate and 3-methyl-1-butanol exhibited the highest content in the initial fractions of the distillate (T5), with acetic acid and ethyl carbamate present at greater intensity in the final fractions of the distillate (T75). Furfural exhibited a similar trend but without visual contrast in the heatmap (Figure 2). Esters and higher alcohols are the main volatile compounds responsible for the sensory

Table 2. Volatile aromatic compounds in cachaça across distillation in a three-body pot still.

Distillation time (min)	Concentration*										
	Ethanol	Acetaldehyde ¹	Ethyl acetate ²	1-propanol	2-methyl-1-propanol	3-methyl-1-butanol	Furfural	Acetic acid	Methanol	Ethyl carbamate	Ethyl lactate
5	67.0 ± 1.6 ^a	17.3 ± 0.7 ^a	92.6 ± 3.9 ^a	47.2 ± 2.1 ^a	65.5 ± 3.1 ^a	282.7 ± 13.1 ^a	0.14 ± 0.01 ^a	7.7 ± 0.3 ^a	1.69 ± 0.07 ^a	6.1 ± 0.3 ^a	0.44 ± 0.02 ^a
10	66.5 ± 1.5 ^a	9.5 ± 0.4 ^b	52.6 ± 2.2 ^b	41.1 ± 1.9 ^{ab}	49.9 ± 2.3 ^b	270.7 ± 12.7 ^a	0.27 ± 0.01 ^b	12.7 ± 0.5 ^b	1.32 ± 0.06 ^b	9.5 ± 0.5 ^b	0.56 ± 0.03 ^{ab}
15	66.1 ± 1.6 ^a	6.7 ± 0.3 ^c	31.7 ± 1.4 ^c	36.6 ± 1.6 ^b	51.6 ± 2.2 ^b	260.3 ± 12.2 ^{ab}	0.31 ± 0.01 ^b	13.8 ± 0.6 ^{bc}	1.27 ± 0.06 ^b	16.8 ± 0.8 ^c	0.69 ± 0.03 ^b
20	64.5 ± 1.5 ^{ab}	3.5 ± 0.15 ^d	19.1 ± 0.9 ^d	35.8 ± 1.5 ^b	48.4 ± 2.1 ^b	252.4 ± 11.8 ^b	0.42 ± 0.02 ^c	14.9 ± 0.7 ^c	1.12 ± 0.05 ^c	19.8 ± 0.9 ^c	0.88 ± 0.04 ^{bc}
25	63.3 ± 1.4 ^{ab}	1.8 ± 0.08 ^e	7.7 ± 0.3 ^e	31.3 ± 1.4 ^c	46.3 ± 2.0 ^b	241.1 ± 12.0 ^b	0.44 ± 0.02 ^c	15.6 ± 0.8 ^c	1.05 ± 0.05 ^c	18.6 ± 0.9 ^c	1.01 ± 0.05 ^c
30	62.2 ± 1.4 ^{ab}	0.9 ± 0.04 ^e	2.3 ± 0.11 ^f	25.3 ± 1.2 ^{cd}	40.1 ± 1.8 ^{bc}	237.2 ± 11.2 ^b	0.47 ± 0.02 ^c	16.7 ± 0.8 ^{cd}	0.94 ± 0.05 ^{cd}	21.3 ± 1.0 ^{cd}	1.22 ± 0.06 ^d
35	61.0 ± 1.3 ^b	< LQ	0.4 ± 0.02 ^f	20.1 ± 0.9 ^d	35.1 ± 1.6 ^c	225.2 ± 10.8 ^{bc}	0.50 ± 0.02 ^{cd}	17.2 ± 0.8 ^{cd}	0.83 ± 0.04 ^d	23.6 ± 1.1 ^d	1.44 ± 0.07 ^e
40	60.1 ± 1.2 ^b	< LQ	< LQ	16.2 ± 0.7 ^e	30.5 ± 1.3 ^c	201.1 ± 10.1 ^c	0.51 ± 0.02 ^{cd}	18.3 ± 0.8 ^{cd}	0.76 ± 0.04 ^d	25.6 ± 1.2 ^d	1.66 ± 0.08 ^f
45	58.5 ± 1.1 ^{bc}	< LQ	< LQ	14.2 ± 0.6 ^{ef}	26.7 ± 1.2 ^{cd}	166.3 ± 7.7 ^d	0.53 ± 0.03 ^{cd}	19.9 ± 0.9 ^e	0.71 ± 0.03 ^d	25.0 ± 1.3 ^d	1.81 ± 0.09 ^e
50	57.4 ± 1.0 ^c	< LQ	< LQ	11.1 ± 0.5 ^f	20.7 ± 0.9 ^d	154.2 ± 6.4 ^d	0.55 ± 0.03 ^{cd}	21.1 ± 1.0 ^e	0.65 ± 0.03 ^{de}	26.6 ± 1.1 ^d	1.83 ± 0.08 ^e
55	56.6 ± 1.1 ^c	< LQ	< LQ	7.7 ± 0.3 ^g	15.2 ± 0.7 ^{de}	141.7 ± 6.9 ^{de}	0.57 ± 0.03 ^d	23.4 ± 1.2 ^e	0.62 ± 0.04 ^e	27.3 ± 1.2 ^d	1.86 ± 0.08 ^e
60	55.5 ± 0.9 ^{cd}	< LQ	< LQ	5.0 ± 0.2 ^{gh}	10.7 ± 0.4 ^e	133.6 ± 6.1 ^e	0.59 ± 0.03 ^d	25.6 ± 1.1 ^{ef}	0.55 ± 0.03 ^{ef}	29.0 ± 1.4 ^{de}	1.81 ± 0.07 ^e
65	54.1 ± 0.8 ^d	< LQ	< LQ	4.1 ± 0.20 ^h	8.7 ± 0.4 ^e	127.4 ± 5.8 ^e	0.61 ± 0.03 ^d	28.9 ± 1.3 ^f	0.52 ± 0.02 ^f	30.2 ± 1.5 ^{de}	1.65 ± 0.08 ^f
70	52.4 ± 0.7 ^{de}	< LQ	< LQ	3.1 ± 0.13 ^h	5.7 ± 0.3 ^{ef}	119.9 ± 5.1 ^{ef}	0.62 ± 0.03 ^d	33.4 ± 1.6 ^f	0.48 ± 0.02 ^g	32.5 ± 1.6 ^e	1.57 ± 0.07 ^{ef}
75	50.1 ± 0.7 ^e	< LQ	< LQ	2.0 ± 0.08 ^h	4.1 ± 0.2 ^f	111.3 ± 4.6 ^f	0.63 ± 0.03 ^d	39.3 ± 1.7 ^g	0.44 ± 0.02 ^g	34.6 ± 1.5 ^e	1.42 ± 0.06 ^e

Concentration - with the exception of ethanol (% v/v) and ethyl carbamate (µg/L), *mg/100 mL anhydrous alcohol, . LQ = limit of quantification. ¹ Acetaldehyde LQ < 0.55 mg/100 mL anhydrous alcohol, ² Ethyl acetate LQ < 0.42 mg/100 mL anhydrous alcohol. Means (x ± standard deviation) followed by different lowercase letters are significantly different (p < 0.05) as analysed by (ANOVA) and the Tukey's test.

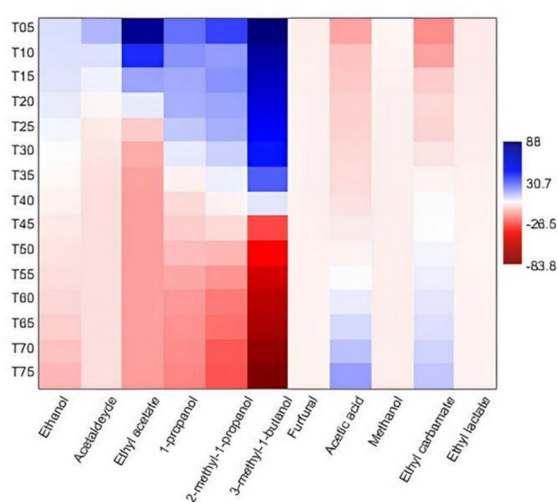


Figure 2. Heatmap showing the correlation between compounds and distillation time (blue/higher intensity, red/lower intensity) and temporal patterns of the different distillate fractions of cachaça.

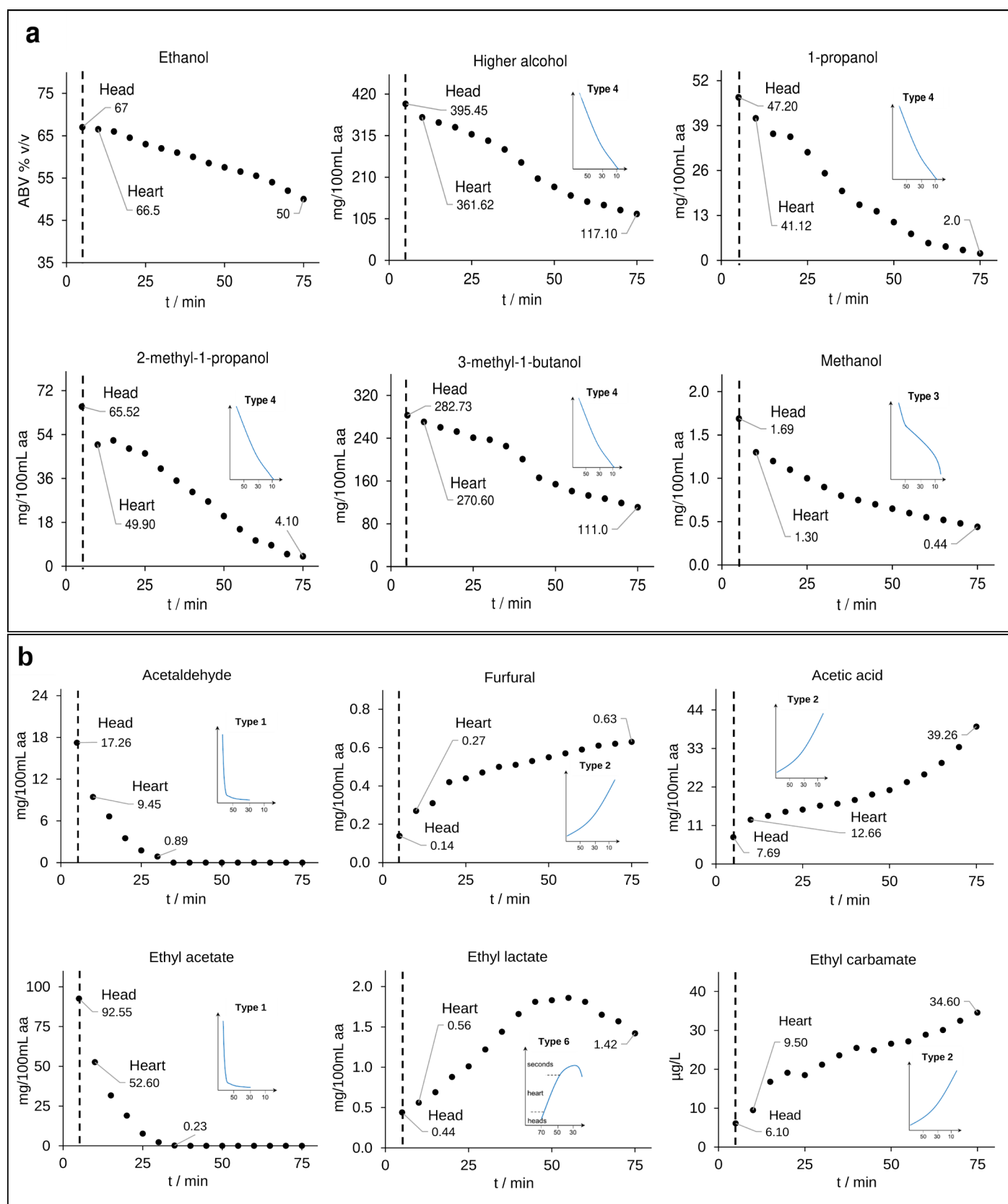
characteristics of distilled beverages, with 3-methyl-1-butanol the main higher alcohol in cachaça (Hernández-Gómez et al. 2009).

Due to their preferential solubilities in ethanol, the distillation of higher alcohols occurs throughout the distillation process in pot stills (Alcarde et al. 2010). Ethyl acetate has a high degree of volatilisation, accumulating in the 'head' fraction of the distillate. Given that acetic acid and ethyl carbamate

have higher boiling points and affinity for water, they are distilled to a higher concentration in the final fractions of the distillate (Léauté 1990). Balcerek et al (2017) reported that during the distillation process of plum brandies, aliphatic aldehydes, esters and higher alcohols (1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol) were the main compounds found in the 'head' fraction, while furfural, acetic acid and ethyl carbamate were present at higher levels in the 'tail' fraction. A similar result for the volatile aromatic compounds during distillation was obtained here (Table 2)

Figure 3 shows the concentration of volatile aromatic compounds in cachaça from the three-body pot still as a function of distillation time. During the distillation process, the volatile aromatic compounds showed different types of behaviour, according to their volatilities in the ethanol-water matrix. Following the classification (types 1 to 8) proposed by Zanghelini et al (2024b), based on the profiles described by Cantagrel (1989) and Douady et al (2019) for cognac distillation, Zanghelini et al (2024b) grouped 'light', 'intermediate' and 'heavy' compounds according to the profile of concentration plotted as a function of the ABV of the distillate. The light compounds are highest in concentration in the first fractions of distillate and are more present in the head fractions, with

Figure 3. Volatile aromatic compounds during the distillation of cachaça in a three-body pot still. Comparison with the classification of Zanghelini et al (2024b), based on Cantagrel (1989) and Douady et al (2019) (small inset plots). Vertical dashed lines indicate the cut point between the 'head' (5 min) and 'heart' fractions of the distillate



hardly any or lower presence in the tails. The intermediate compounds, whose volatility is similar to ethanol, are present in all distillate fractions. The heavy compounds increased

concentration across the distillation, peaking in the final fractions of distillate.

Both acetaldehyde and ethyl acetate showed high

volatility at the beginning of the distillation and about halfway through the process were below the limit of quantification in the boiler (Table 2). The concentration peaks in the 'head' fraction were peculiar to light compounds, classified as 'type 1' by Zanghelini et al (2024b). These compounds exhibit high volatility throughout the distillation process, with their extraction at the beginning of the process and, consequently, are lost from the boiler (Douady et al. 2019; Zanghelini et al. 2024b).

Higher alcohols (1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol) followed the pattern of ethanol, having an affinity for this alcohol in the steam phase and low solubility in water, despite having relatively high boiling points (Léauté 1990). These compounds were found at higher concentrations in the 'head' fraction of the distillate, with a gradual reduction during distillation (Figure 3a), characteristic of the profile of type 4 compounds (Zanghelini et al. 2024b). Similar results were reported by Puentes et al (2018) with the distillation of Armagnac, and Satora et al (2017) for the distillation of plum spirits. With the distillation of sugar cane spirit, Alcarde et al (2010) observed a similar behaviour, suggesting that the distillation of higher alcohols in pot stills reflects the process regardless of the raw material used or the type of distilled beverage produced. Zanghelini et al (2024b) categorised the short chain alcohols from Charentaise distillation of cognac, such as propanol and isobutanol, as type 4 compounds, with volatilities close to that of ethanol. Balcerek et al (2017) also found that the higher alcohols (1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol) were higher in the 'head' fraction of plum brandy distilled in alembic stills than the 'heart' fraction and lower still in the 'tail' fraction.

Although the absolute volatility of methanol is higher in pure water, its relative volatility increases as the concentration of ethanol in the mixture increases. This behaviour is related to its log *P* value, a well established measurement of the hydrophilicity of a compound, which with methanol is -0.7 , between that of ethanol (-0.31) and of water (-1.38) (Da Porto et al. 2010). In the present study, methanol exhibited a higher concentration at the beginning of distillation, characteristic of type 3 behaviour (Zanghelini et al. 2024b).

Ethyl lactate showed type 6 behaviour, increasing from the beginning of distillation, with a peak concentration in the 'heart' fraction, and a decrease in the final fractions (Figure 3b). According to Zanghelini et al (2024b), type 6 or intermediate compounds have lower volatility than ethanol at the beginning of the distillation process, a parameter that steadily increases as the concentration of ethanol in the boiler is reduced. Consequently, these compounds are distilled throughout the process and are present in higher contents in the 'heart' fraction.

Some compounds have lower volatility than ethanol at the beginning of distillation, but with a gradual increase in the ethanol concentration as that in the still boiler decreases, leading to a higher concentration in the 'tail' fraction (Douady et al. 2019; Zanghelini et al. 2024b). This was observed for furfural (type 2 profile, Zanghelini et al. 2024b), with higher concentrations in the final fractions of the distillate (Figure 3b). It has been reported for a variety of distilled beverages that the concentration of furfural was higher towards the end of the distillation process (Matias-Guiu et al. 2016; Awad et al. 2017; Balcerek et al. 2017; Xiang et al. 2020; Zanghelini et al. 2024b). Acetic acid and ethyl carbamate also showed increasing concentrations across the fractions during distillation (Table 2), with a type 2 profile and peak concentration in the last fractions of the distillation. Alcarde et al (2012) observed similar results for ethyl carbamate in double distilled cachaça. According to Matias-Guiu et al (2016), high molecular weight compounds soluble in water tend to be distilled in the 'tail' fraction.

In a three-body pot still distillation, heating the wine in the boiler generates hydroalcoholic steam rich in light compounds, which bubbles into the wine in the distiller, heating it and enriching it in ethanol, which in turn generates a more concentrated hydroalcoholic steam than from the boiler (Alcarde 2024). Accordingly, the higher alcohol content in the distillation system influences the absolute and relative volatilities of volatile compounds (Esteban-Decloux et al. 2022), resulting in a better separation and distribution of these compounds in the distillate due to their specific volatilities at higher concentrations of ethanol in the generating liquid. This was also observed by Zanghelini et al (2024b) in double distilled cognac.

Classifying the volatile aromatic compounds during distillation into light; intermediate and heavy compounds demonstrates that their behaviour reflects their absolute and relative volatilities (Esteban-Decloux et al. 2022). Furthermore, the classification of compounds by Zanghelini et al (2024b) share various features (structural and physical chemical properties); volatilisation of influenced by chemical group, chain length and conformation, molecular weight and log P value.

Concentration of compounds and implications for legal limits and health

The concentration of volatile compounds in the 'head' and 'heart' fractions of cachaça, as well as their legal limits, are reported in Table 3. The distillation process conducted in a three-body pot still conferred cachaça with a higher ABV (~58%) compared to that produced using a simple distillation process (Bortoletto et al. 2015). The alcohol content of cachaça was reduced with drinking water, to the legal ABV (48 to 38%).

Figure 3 shows that the 'head' fraction of the distillate had a higher content of acetaldehyde, ethyl acetate and higher alcohols (1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol) but with a higher concentration of the contaminant methanol. The 'final fractions' of the distillate contained higher concentrations of acetic acid, furfural and ethyl lactate, as well as the contaminant ethyl carbamate. These compounds are typically associated with the 'tail' in the final stage of distillation. In the 'heart' fraction of the distillate, representing cachaça, the concentration of all volatile aromatic compounds and contaminants were within the Brazilian legal limits (Brazil 2022).

The volatile acidity of cachaça, normally represented by 90% acetic acid (Moreira et al. 2012), contributes to the aroma and flavour of the spirit. However, excess acetic acid (> 100 mg/100mL anhydrous alcohol), is considered a sensory defect with an undesirable and aggressive flavour (Alcarde et al. 2011; Franco et al. 2023). Cachaça, distilled in a three-body pot still produced a relatively low acidity (~22 mg/100

Table 3. Concentration of volatile aromatic compounds and contaminants in the 'head' and 'heart' fractions of cachaça distilled in a three-body pot still.

Compound	Fraction*		Legal limit for cachaça (Brazil, 2022)
	Head	Heart	
Ethanol (% ABV)	67.0 ± 1.0	58.7 ± 0.8	38–48
Aromatic volatile compounds			
Acetic acid	7.7 ± 0.3	22.0 ± 1.1	< 150
Acetaldehyde	17.3 ± 1.0	6.1 ± 0.3	< 30
Ethyl acetate	92.6 ± 3.3	28.3 ± 1.2	< 200
1-propanol	47.2 ± 2.0	23.4 ± 1.0	–
2-methyl-1-propanol	65.5 ± 3.0	30.5 ± 1.3	–
3-methyl-1-butanol	282.7 ± 11.3	189.5 ± 8.1	–
Higher alcohols ¹	395.5 ± 16.3	243.4 ± 10.4	< 360
Furfural	0.14 ± 0.01	0.36 ± 0.02	< 5
Congeners ²	513.1 ± 20.9	300.0 ± 13.1	200-650
Ethyl lactate	0.4 ± 0.02	1.4 ± 0.05	–
Contaminants			
Methanol	1.69 ± 0.06	0.8 ± 0.03	< 20
1-butanol ³	< LQ	< LQ	< 3
2-butanol ⁴	< LQ	< LQ	< 10
Ethyl carbamate	6.1 ± 0.2	38.5 ± 1.3	< 210

*mg/100 mL anhydrous alcohol, other than ethanol content (% ABV) and ethyl carbamate (µg/L).

LQ = limit of quantification. ¹ Sum of alcohols (1-propanol, 2-methyl-1-propanol, 3-methyl-1-butanol).

² Sum of congeners (acetic acid, acetaldehyde, ethyl acetate, higher alcohols, and furfural).

³ 1-butanol LQ < 0.37 mg/100 mL anhydrous alcohol, ⁴ 2-butanol LQ < 0.24 mg/100 mL anhydrous alcohol

mL anhydrous alcohol), against an ideal volatile acidity of 50 mg/100mL anhydrous alcohol for a balanced sensory quality. The typical volatile acidity of cachaça from simple stills is about 80 mg/100mL anhydrous alcohol (Pereira et al. 2025).

At low concentrations (< 10 mg/100mL anhydrous alcohol), acetaldehyde provides a fruity character to distilled beverages; but in excess (> 30 mg/100mL anhydrous alcohol), it contributes a pungent odour that compromises sensory quality (Lachenmeier and Sohnius 2008). Further, acetaldehyde is 'possibly carcinogenic in humans' (Group 2B) as defined by the International Agency for Research on Cancer (1999). Further, it can impact the central nervous system (Fernandes et al. 2007), causing nausea, vomiting, restlessness, sweating, mental confusion, decreased blood pressure, increased heart rate and headache (Alcarde et al. 2011). Although acetaldehyde is produced by yeast as an intermediate in the production of ethanol, high concentration in cachaça are indicative of spontaneous oxidation or bacterial activity (Moreira et al. 2012). Zanghelini et al (2024b) found high contents of acetaldehyde in the 'head' fraction of the distillate during the production of cognac. With high volatility across the process, the concentration of acetaldehyde gradually decreased during distillation with exhaustion from the boiler. In this study, the separation of the 'head' fraction from the final distillate resulted in cachaça with a low acetaldehyde content (~6 mg/100 mL anhydrous alcohol).

Esters are formed by yeast during fermentation (Reis et al. 2018), and by esterification of organic acids with alcohols (Franitza et al. 2016b; Kelly et al. 2023). These compounds are important contributors to the aroma of cachaça and are qualitatively similar in distilled spirits, contributing to their pleasant fruity aroma (Moreira et al. 2012). At low concentrations (< 200 mg/100 mL anhydrous alcohol), esters offer pleasant floral and fruit aromas, whereas at higher concentrations, they have a negative effect on sensory quality, causing a cloying taste in spirits (Nykänen and Suomalainen 1983).

Ethyl acetate is the main ester in cachaça, accounting for up to 80% (Corrêa et al. 2021), followed by ethyl lactate, which is considered an aroma stabiliser in distillates (Zanghelini et al. 2024a). Brazilian law

does not specify limits for ethyl lactate in cachaça. Together, these two esters represent 95% of the total esters in cachaça (Moreira et al. 2012). Distillation in a three-body pot still produced cachaça with a concentration of esters (~28 mg/100 mL anhydrous alcohol) within the ideal range (< 200 mg/100 mL anhydrous alcohol) for the sensory quality of the distilled spirit.

Higher alcohols are formed from sugars during fermentation, transamination of amino acids or reduction of aldehydes by yeast (Guymon 1974; Nykänen and Nykänen 1991). The primary higher alcohols in cachaça are 1-propanol, 2-methyl-1-propanol and 3-methyl-1-butanol (Procópio et al. 2011), which influence the flavour of distilled beverages (de-la-Fuente-Blanco et al. 2016; Gonzalez and Morales 2017). Higher alcohols are quantitatively the most important volatile aromatic compounds in fruit distilled spirits (Madrera and Mangas Alonso 2005) and contribute more to the odour than to the quality of distilled beverages (Calleja and Falqué 2005). However, the 'vegetal' and 'herbaceous' odours imparted by six carbon higher alcohols may cause defects (Calleja and Falqué 2005). Concentrations below 300 mg/100 mL anhydrous alcohol are considered favourable to the sensory quality of distilled beverages, but above 400 mg/100 mL anhydrous alcohol, impair the sensory perception (Rapp and Versini 1995; Gantumur et al. 2022). In the 'heart' fraction of the distillate samples, the concentration of higher alcohols was ~243 mg/100 mL anhydrous alcohol, lower than the values for cachaça distilled in simple pot stills (Alcarde et al. 2011; Bortoletto et al. 2015). This can be explained by the fermented must (wine) undergoing two distillations in a three-body pot still (first batch in the distiller and second batch in the boiler), rather than one distillation in the simple pot stills.

Furfural is formed during distillation as a product of the Maillard reaction through dehydration of residual pentoses from fermentation activated by heat and related to the pyrogenation of organic matter during distillation of beverages (Hernández-Gómez et al. 2005; Balcersek et al. 2017). The burning of sugar cane for harvesting and the presence of bagasse residues in the fermented juice during distillation contribute to the formation of furfural in cachaça (Alcarde 2024). As it is potentially toxic, the concentration of furfural is limited in distilled

spirits (Ramírez-Guizar et al. 2020; Zanghelini et al. 2024a). However, furfural (< 5 mg/100mL anhydrous alcohol) can contribute to the bouquet of the distilled spirits, with a smoked and nutty aroma, as reported in cognac (Zanghelini et al. 2024a). The concentration of furfural in the 'heart' fraction of the distillate in this study was 0.36 mg/100 mL anhydrous alcohol, lower than the maximum legal limit for cachaça of 5 mg/100 mL anhydrous alcohol (Brazil 2022).

The 'congener coefficient' for cachaça, or the sum of acetic acid, acetaldehyde, ethyl acetate, higher alcohols and furfural is between 200-650 mg/100 mL anhydrous alcohol (Brazil 2022). The total content of congeners in cachaça using a three-body pot still was ~300 mg/100 mL anhydrous alcohol. These compounds are responsible for the bouquet of cachaça, and their concentration determines the sensory quality of the beverage. The major volatile aromatic compound in the congener coefficient in this work was 3-methyl-1-butanol. At appropriate concentrations, it imparts whiskey, malt, alcoholic, wine, banana and sweet aromas (Moreira et al. 2012). Cachaça distilled in stainless steel columns (27 samples) had a congener coefficient of 240-357 mg/100mL (mean 298) of anhydrous alcohol whereas Cachaça distilled in copper pot stills (23 samples) had a 'congener coefficient' of 251-517 mg/100mL (mean 364) anhydrous alcohol (Alvarenga et al. 2023). Serafim and Franco (2015) reported a mean congener coefficient of 411 mg/100mL anhydrous alcohol for cachaça (44 samples) distilled in stainless steel columns and an average congener coefficient of 415 mg/100mL anhydrous alcohol for cachaças (61 samples) distilled in copper pot stills.

Methanol is formed by the enzymatic degradation of pectins and is present in fruit and stone fruit spirits (Hernández-Gómez et al. 2005; Balcerek et al. 2017). As a contaminant in distilled beverages with high toxicity, its concentration is subject to legal control (Cabarroglu and Yilmaztekin 2011). Methanol can cause headaches, dizziness, vomiting and, in extreme cases, blindness, coma and death (Abud et al. 2019). Methanol is oxidised to methanal (formaldehyde) and formic acid, which can cause acidosis and affect the respiratory system (Moreira et al. 2012). With its volatilisation behaviour (Figure 3a), it is concentrated in the initial fractions of the distillate, presenting higher concentrations in the 'head' fraction. The concentration of methanol in

the 'heart' fraction of the distillate (0.77 mg/100 mL anhydrous alcohol) from the three-body pot still (Tables 2, 3) is well below the maximum limit for cachaça (20 mg/100 mL anhydrous alcohol), and lower than that reported by Bortoletto et al (2015) in cachaça produced in simple pot stills. Further the contaminants, n-butanol and sec-butano, were not detected in cachaça produced from a three-body pot still in this work.

The formation of ethyl carbamate in distilled beverages is through enzymatic degradation of cyanogenic glycosides in raw materials, resulting in cyanide ions, which are oxidised to cyanate, which then reacts with ethanol in the presence of copper ions forming ethyl carbamate (Aresta et al. 2001). Other routes for the formation of ethyl carbamate are from the precursors urea, citrulline and N-carbamyl phosphates (Hesford and Schneider 2001; Alcarde et al. 2012). Ethyl carbamate is carcinogenic according to the International Agency for Research on Cancer (Group 2A) (Baan et al. 2007) with a maximum legal limit in cachaça of 210 µg/L (Brazil 2022).

The absence of a hat (the upper part of a pot still), faster distillation, higher temperatures and flows, result in the greater transport of ethyl carbamate to cachaça (Lima et al. 2012). However, Balcerek et al (2017) did not observe the influence of different distillation methods on ethyl carbamate in plum distillates. Further, a reduction in ethyl carbamate in cachaça produced in pot stills with reflux systems in the hat and double distillation have been reported (Alcarde et al. 2013; Bortoletto et al. 2015). Here, the concentration of ethyl carbamate in the 'heart' fraction from the three-body pot still was 38.5 µg/L. After dilution (to 38% ABV), ethyl carbamate was ~25 µg/L, a value considerably lower than the legal limit of 210 µg/L (Brazil 2022). This is lower than the ethyl carbamate levels found in commercial cachaça by Bortoletto and Alcarde (2015, 2016) where 39% (n = 268) and 24% (n = 376) of the samples were above the legal limit. More recently, Pereira et al (2024) reported an improving picture with 5% of 391 commercial cachaça samples containing ethyl carbamate above the maximum permitted limit.

Principal component analysis (PCA) was used to analyse the differentiation between the volatilisation and volatile aromatic compounds -

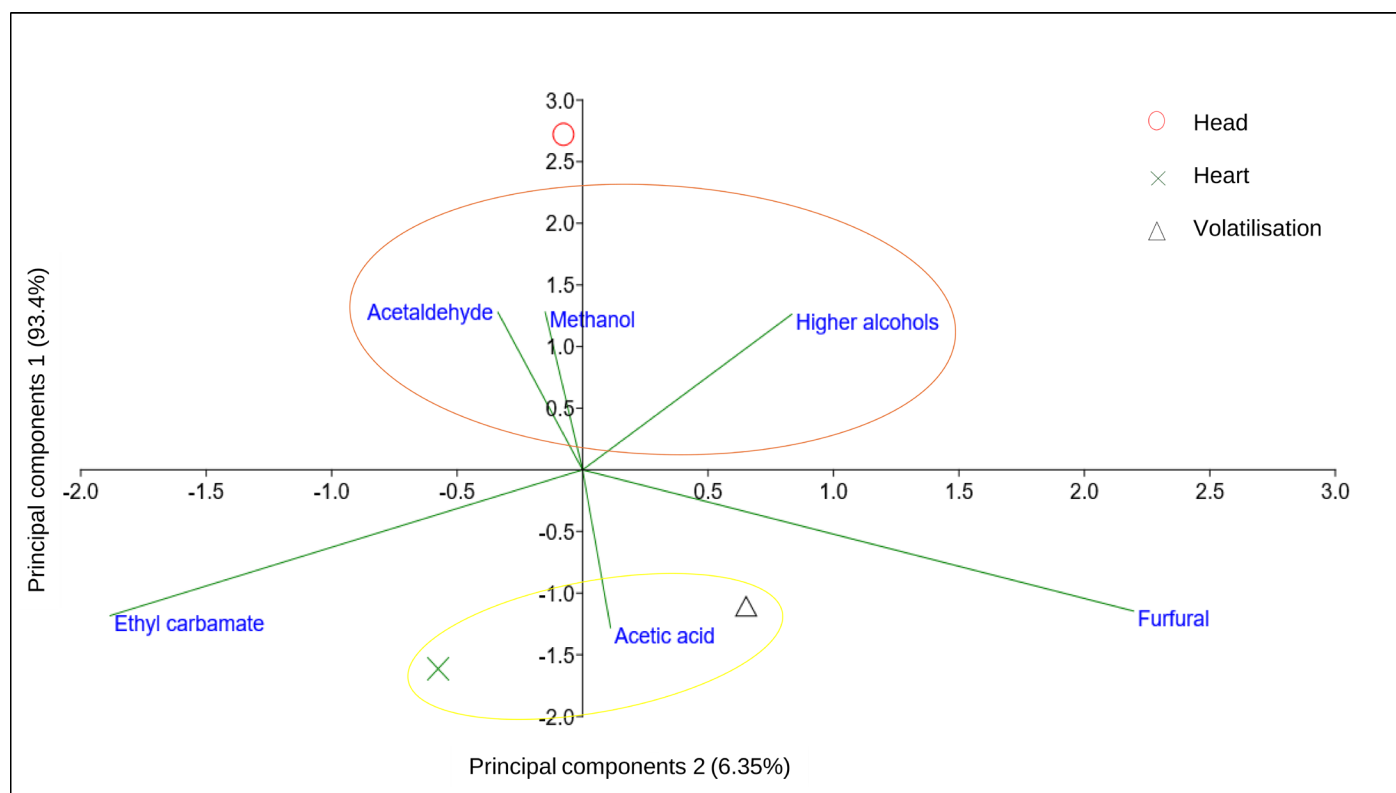
acetaldehyde, acetic acid, furfural, higher alcohols, methanol and ethyl carbamate in the 'head' and 'heart' fractions of cachaça distilled in a three-body pot still (Table 3). The principal components 1 (PC1) and 2 (PC2) explained 99% of the variance, demonstrating the robustness of the model. Table 4 shows the volatile aromatic compounds with the highest correlation with PC1 and PC2, along with the variance explained for each compound, and its contribution to the corresponding principal component. PC1 was influenced by acetaldehyde, methanol, higher alcohols and acetic acid, whereas PC2 was more influenced by ethyl carbamate and furfural.

In Figure 4, the samples of cachaça were plotted in a plane defined by PC1 and PC2, which explains the variance between the volatilisation behaviour of the compounds and the 'head' and 'heart' fractions of the distillate from a three-body pot still. These results show that the 'head' fraction was separated from the 'heart' fraction, while the volatilisation behaviour of the compounds and the 'heart' fraction of the distillate were grouped. Acetaldehyde, methanol and higher alcohols were the volatile aromatic compounds that contributed the most to PC1, with higher concentrations in the 'head' fraction. Ethyl carbamate was related to the 'heart' fraction, whereas furfural related to volatilisation behaviour, both significantly contributing to PC2.

Table 4. Factor loadings and variances from principal component analysis (PCA) of aromatic volatiles in cachaça distilled in a three-body pot still.

Principal components	Explained variance (%)	Cumulative proportion (%)	Correlated variable and factor loadings
PC1	93.64	93.64	Methanol (0.421) Higher alcohols (0.415) Acetic acid (-0.421) Acetaldehyde (0.420)
PC2	6.35	99.99	Ethyl carbamate (-0.620) Furfural (0.723)

Figure 4. Principal component analysis of volatile aromatic compounds in cachaça distilled in a three-body pot still.



Although both ethyl carbamate and furfural increased towards the end of the distillation, the PCA revealed that their patterns of contribution did not coincide along the same axis of variation. According to [Figure 3b](#), furfural accumulated at the end of distillation, a behaviour that was reflected in its projection on PC2. This multivariate analysis demonstrated how the compounds volatilised during distillation: some as markers of the 'head' fraction (acetaldehyde, methanol and higher alcohols), ethyl carbamate related to the 'heart', with furfural related to the overall volatilisation behaviour and as a marker of the 'final fraction'. Although associated with PC1, acetic acid with higher levels in the final fractions was the opposite to acetaldehyde, methanol and higher alcohols.

Odour active value and sensory characteristics of the heart fraction

Volatile aromatic compounds form the characteristic flavour of cachaça (Bortoletto and Alcarde 2015), but only at concentrations above their odour detection threshold (ODT) (Duarte et al. 2017). The contribution of volatile aromatic

compounds present in the 'heart' fraction can be estimated by their odour activity value (OAV). Since the ODT of compounds depends on the ethanol concentration in hydroalcoholic mixtures, it is important to calculate OAVs in a similar matrix (Lee et al. 2001; Le Berre et al. 2007; Fiches et al. 2014; Ickes and Cadwallader 2017). There is a lack of reported ODTs for compounds in cachaça, and those used here were obtained from other distilled beverages or measured in hydroalcoholic solutions with ABV similar to distilled spirits (Willner et al. 2013; Gao et al. 2014; Zhao et al. 2018; Niu et al. 2020; Zanghelini et al. 2024b).

The OAV of volatile aromatic compounds, concentration in cachaça, together with the ODT (from the literature) and odour descriptions is shown in [Table 5](#). Compounds with the greatest influence on the aroma of cachaça were, in descending order with OAVs > 1, ethyl acetate, 3-methyl-1-butanol, 2-methyl-1-propanol, 1-propanol, acetaldehyde and acetic acid. These compounds are also important in the aroma of baijiu (Niu et al. 2020), cognac (Uselmann and Schieberle 2015; Zanghelini et al. 2024b) and pear brandy (Willner et al. 2013).

Table 5. Volatile aromatic compounds, odour detection thresholds and odour activity values in cachaça distilled in a three-body pot still.

Volatile aromatic compound	Aroma description ¹	ODT ² (mg/L)	'Heart' ³ (mg/L)	OAV ⁴
Acetic acid	Vinegar, pungent, sour	75.5 ^A	87.8 ± 4.2	1.16
Acetaldehyde	Green, pungent, sweet, spicy, cocoa-like, fruity, ethereal, aldehydic	19.2 ^A	24.4 ± 1.4	1.27
Furfural	Smoky, nutty, bread-like, caramelised, sweet, woody, almond	44.0 ^D	1.4 ± 0.08	0.03
Ethyl acetate	Fruity, nail polish, ether-like, pineapple-like, fruity	4.0 ^B	113.0 ± 4.8	28.26
Ethyl lactate	Fruity, green	10.0 ^B	5.7 ± 0.20	0.57
1-propanol	Alcoholic, fermented, chemically smelly, bread-like, musty	40.0 ^B	93.5 ± 4.1	2.34
2-methyl-1-propanol	Malty, alcoholic, chemically smelly, slightly alcoholic-like, fruity	28.3 ^D	122.12 ± 5.2	4.32
3-methyl-1-butanol	Malty, cocoa, chemically smelly, spicy, alcoholic, fruity, fusel oil	56.1 ^C	757.8 ± 32.4	13.51
Methanol	Alcoholic	20.0 ^B	3.1 ± 0.01	0.15

ODT = odour detection threshold; OAV = odour activity value (concentration of the compound in the 'heart' fraction/ODT). ¹ Good Scents Company database (<https://thegoodscentscompany.com>), ² ODT in hydroalcoholic solutions at 40% ABV, ^A Uselmann and Schieberle (2015); ^B Berger et al (2007); ^C Poisson and Schieberle (2008); and at 46% ABV ^D Gao et al (2014). ³ Average concentration in the 'heart' (~58% ABV after dilution to 40% ABV. ⁴ OAV in the 'heart' fraction diluted to 40% v/v ethanol; **OAV > 1**.

Volatile aromatic compounds impact on the sensory acceptance of cachaça by consumers, with the results reported here suggesting that higher alcohols are important. Two of these, 3-methyl-1-butanol and ethyl acetate, making the greatest contribution. Since studies on volatile aromatic compounds in cachaça have been limited to quantification, no data is available on OAVs or their influence on the sensory perception of this distilled spirit. The results of this present study showed that 66% of the analysed components contributed to the aroma of cachaça distilled in a three-body pot still.

It is important to note that ODTs vary according to the method used to measure them and the chemical interactions that occur between the volatile aromatic compounds in a hydroalcoholic solution. Indeed, the perceptive interactions between volatile aromatic compounds may intensify or weaken the olfactory impact, such that they could be detected even if their OAVs are lower than 1 or, conversely, not be perceptible if their OAVs are higher than 1 (Qin et al. 2022).

Accordingly, OAVs should be used with caution to estimate the olfactory impact of different volatile aromatic compounds in liquids with a similar matrix. The OAV values reported here represent indicators of the odour of the compounds and are the basis for further research on odour activity values in cachaça

Comparison with other distillation methods

Table 6 details the concentration of aromatic volatile and contaminant compounds in cachaça from single distillation, double distillation and distillation using a three-body pot still. The double distilled cachaça had the highest ethanol content, with the single distilled cachaça the highest content of congeners (acetic acid, acetaldehyde, ethyl acetate, ethyl lactate, higher alcohols and furfural). Double distilled cachaça from the three-body pot still had similar concentrations of acetaldehyde, ethyl acetate, ethyl lactate and higher alcohols. The double distilled cachaça exhibited the lowest concentrations of acetic acid and total congeners.

Table 6. Aromatic volatile compounds and contaminants in cachaça using different distillation methods.

Compound	Cachaça samples*			Legal limit (Brazil, 2022)
	Single distillation	Three-body pot still	Double distillation	
Ethanol (% ABV)	45.0 ± 0.6 ^c	58.7 ± 0.8 ^b	69.1 ± 0.9 ^a	38–48 ⁵
Aromatic volatile compounds				
Acetic acid	38.5 ± 1.6 ^a	22.0 ± 1.1 ^b	11.8 ± 0.4 ^c	< 150
Acetaldehyde	9.7 ± 0.4 ^a	6.1 ± 0.3 ^b	5.7 ± 0.3 ^b	< 30
Ethyl acetate	39.9 ± 1.9 ^a	28.3 ± 1.2 ^b	23.8 ± 1.1 ^b	< 200
1-propanol	30.3 ± 1.6 ^a	23.4 ± 1.0 ^b	20.5 ± 0.9 ^b	–
2-methyl-1-propanol	44.8 ± 2.0 ^a	30.5 ± 1.3 ^b	29.9 ± 1.4 ^b	–
3-methyl-1-butanol	250.2 ± 10.8 ^a	189.5 ± 8.1 ^b	174.1 ± 7.4 ^c	–
Higher alcohols ¹	325.3 ± 14.4 ^a	243.4 ± 10.4 ^b	224.5 ± 9.7 ^b	< 360
Furfural	0.96 ± 0.04 ^a	0.36 ± 0.02 ^b	0.11 ± 0.01 ^c	< 5
Congeners ²	414.4 ± 18.3 ^a	300.2 ± 13.1 ^b	265.9 ± 11.5 ^c	200–650
Ethyl lactate	3.3 ± 0.1 ^a	1.4 ± 0.05 ^b	1.1 ± 0.04 ^b	–
Contaminants				
Methanol	2.7 ± 0.11 ^a	0.8 ± 0.03 ^b	0.6 ± 0.02 ^b	< 20
1-butanol ³	0.19 ± 0.01	< LQ	< LQ	< 3
2-butanol ⁴	< LQ	< LQ	< LQ	< 10
Ethyl carbamate	65.6 ± 3.1 ^a	38.5 ± 1.3 ^b	5.9 ± 0.2 ^c	< 210

Means ($x \pm$ standard deviation) with different superscript letters are significantly different ($p < 0.05$) by ANOVA and Tukey's test. *mg/100 mL anhydrous alcohol, except for ethanol (% ABV) and ethyl carbamate ($\mu\text{g/L}$). LQ = limit of quantification. ¹ Sum of alcohols (1-propanol, 2-methyl-1-propanol, 3-methyl-1-butanol). ² Sum of congeners (acetic acid, acetaldehyde, ethyl acetate, higher alcohols, and furfural). ³ 1-butanol LQ < 0.37 mg/100 mL anhydrous alcohol. ⁴ 2-butanol LQ < 0.24 mg/100 mL anhydrous alcohol. ⁵ Commercial spirit after dilution with water.

With the contaminants, single distilled cachaça contained the highest level of methanol and ethyl carbamate, whereas double distilled cachaça had the lowest concentration of ethyl carbamate (Table 6). Similar conclusions for the reduction of ethyl carbamate by double distillation were made by others (Nóbrega et al. 2011; Alcarde et al. 2012; Bortoletto and Alcarde 2016; Pereira et al. 2024).

The alcohol content influences the volatility of compounds during distillation (Esteban-Decloux et al. 2022). The higher the concentration of ethanol in the liquid, and the consequent alcohol vapours during distillation, provides a better separation of the distillate compounds, especially those in the 'head' and 'tail' fractions (Alcarde 2024). This explains why cachaça distilled in three-body pot stills or by double distillation has a lower concentration of congeners and contaminants than cachaça from single distillation. Alvarenga et al (2023) also observed that cachaça distilled in multiple-stage columns had a lower congener coefficient (298 mg/100mL anhydrous alcohol) than cachaça distilled in single stage pot stills (364 mg/100mL anhydrous alcohol). Columns provide a higher ABV in the liquid under distillation.

The results obtained by the single distilled cachaça (Table 6) are in accordance with other reports. The chemical profiles of cachaça produced by simple distillation in copper pot stills had average concentration (mg/100mL anhydrous alcohol) of 17.7 for acetaldehyde, 59.7 for ethyl acetate, 201.3 for higher alcohols and 85.2 for acetic acid (Alvarenga et al. 2023). Serafim and Franco (2015) determined an average concentration in cachaça (mg/100mL anhydrous alcohol) of 2.9 for acetaldehyde, 86.0 for ethyl acetate, 273.0 for higher alcohols and 53.1 for acetic acid for a single distillation in copper pot stills. The differences in chemical composition of cachaça using different methods impacted on their sensory assessment (Table 7). Cachaça from the three-body pot still and double distillation achieved maximum value (2.0) for colour and clearness. The cachaça from a three-body pot still had the highest score for aroma (3.8/4.0) and taste (10.6/12.0). It received the highest cumulative score (18.4/20.0) from the sensory panel and was characterised as pleasant and balanced in aroma and taste. The sample obtained by single distillation

Table 7. Sensory assessment of cachaça obtained from different distillation methods.

	Cachaça		
	Single distillation	Three-body pot still	Double distillation
Colour (max 2 points)	1.9 ± 0.1 ^b	2.0 ± 0.0 ^a	2.0 ± 0.0 ^a
Clarity (max 2 points)	1.9 ± 0.1 ^b	2.0 ± 0.0 ^a	2.0 ± 0.0 ^a
Aroma (max 4 points)	3.3 ± 0.1 ^b	3.8 ± 0.2 ^a	2.8 ± 0.4 ^c
Taste (max 12 points)	9.3 ± 0.4 ^c	10.6 ± 0.3 ^a	10.2 ± 0.4 ^b
Total (max 20 points)	16.4 ± 0.2 ^c	18.4 ± 0.2 ^a	17.0 ± 0.2 ^b

Means ($x \pm$ standard deviation) with different superscript letters are significantly different ($p < 0.05$) by ANOVA and Tukey's test.

had the lowest total score (16.4/20.0) and the panellists described its aroma and taste as pungent, probably reflecting its high content of acetic acid. Conversely, the double distilled cachaça was characterised as smooth in taste, most likely due to its lower acidity. It also received a better score (10.2/12.0) for taste than the single distilled cachaça (9.3/12.0). Nevertheless, the double distilled cachaça received the lowest score for aroma (2.8/4.0), possibly reflecting the lowest content of congeners.

Conclusions

In this study, distillation in a three-body pot still was used to produce cachaça. The outcomes of this work will contribute to a more complete understanding of the behaviour of volatile aromatic compounds during distillation. The compounds were classified as light, intermediate and heavy, according to their volatility and the specific concentration profile of each component throughout the distillation process. The configuration of the equipment and the distillation method in the three-body pot led to the production of good quality cachaça, with all chemical parameters within the limits of the prescribed Brazilian legislation. Of the volatile aromatic compounds, ethyl acetate and 3-methyl-1-butanol contributed the most to the aroma of cachaça, both with OAVs > 10. These results can be used as a reference for the odorous quality of volatile compounds and provide the basis for further studies on OAVs in cachaça.

Author contributions

Tiago Coelho: Investigation, formal analysis, writing (original draft, review and editing).

André Alcarde: Conceptualisation, funding acquisition, methodology, formal analysis, supervision, writing (original draft, review and editing), visualisation.

Conflict of interest

The authors declare that there are no conflicts of interest.

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