

Amazonian wood chips as a strategy to change the sensory and chemical characteristics of cachaça

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Abstract

The aim of this study was to evaluate the physicochemical properties, temporal profiles, and consumer acceptability of *cachaças* aged with different Amazonian woods, namely, *cumarurana*, *jatobá*, and *louro-vermelho*, compared with traditional oak. *Cachaças* were stored with wood chips in glass containers for 6 months, after which sensory and chemical analyses were conducted. The results revealed that *cachaças* aged with *cumarurana* and *jatobá* had sensory profiles and acceptability similar to those aged with oak, displaying desirable wood and vanilla flavors. In contrast, *cachaça* aged with *louro-vermelho* was less accepted due to the detection of off-flavors. The chemical analysis indicated that higher levels of volatile acidity, higher alcohols, dry extract, esters, and aromatic compounds, such as ethyl hexanoate and ethyl octanoate, positively influenced the sensory acceptance of the *cachaças*. Conversely, a translucent hue and high concentrations of certain alcohols were associated with lower acceptance. The study concludes that *cumarurana* and *jatobá* present promising alternatives to oak for *cachaça* aging, offering new opportunities to diversify flavor profiles and meet consumer demands. The use of Amazonian woods could add value to *cachaça* production, promoting a unique sensory experience while supporting sustainable practices in the beverage industry.

Keywords: physic-chemical analysis; volatile compounds; temporal dominance of sensations; acceptance test.

Practical Application: Amazonian woods in *cachaça* aging create unique flavors, adding diversity and market value.

1 INTRODUCTION

Cachaça, a typical Brazilian beverage, is defined as a sugarcane-derived spirit with an alcoholic strength ranging from 38% to 48% (v/v) at 20°C, distilled from fermented sugarcane juice, and possessing distinctive sensory attributes (Brasil, 2022). Recent legislative measures in Brazil, implemented in 2022, have introduced guidelines regarding the utilization of wood chips in the *cachaça* storage process, heralding an innovative approach for the market. This inclusion facilitates the creation of wood blends, standardization of color and flavor, and the inception of novel *cachaça* varieties. The sensory profile of *cachaça* is predominantly shaped by the yeast employed in fermentation, distillation methods, and optional aging processes (Ratkovich et al., 2023). Notably, *cachaça* ranks as the most-consumed distilled beverage in Brazil and the third globally, trailing only Vodka (Russia) and Soju (Korea) (Conceição et al., 2020).

Throughout the elaboration stages of *cachaça*, numerous chemical and biochemical reactions occur, yielding a plethora of compounds such as esters, alcohols, and carboxylic acids, which significantly influence its sensory characteristics (Oliveira et al., 2020). Every phase of distilled beverage production is

pivotal, with aging playing a particularly crucial role. Wooden barrels mitigate the intense sensory attributes of freshly distilled spirits, such as spiciness, woody undertones, pungency, alcoholic potency, and fruity or floral notes, thereby rendering the resulting aroma and flavor profiles more desirable (Ratkovich et al., 2023). The aging or maturation process typically involves extended storage in wooden barrels or the adoption of wood chips as a means to expedite aging (Abreu-Naranjo et al., 2023). Consequently, *cachaça* assimilates a spectrum of sensory characteristics from wood compounds over the aging duration, thereby altering its color, flavor, and aroma, thus enhancing the quality and value of the distilled beverage (Lima et al., 2022).

The prevalent use of oak wood sourced from Europe or America for barrel production in global distilled beverage aging stems from its permeability, durability, malleability, and ease of handling (Castro et al., 2020; Silveira & Barbeira, 2022). Nevertheless, studies have highlighted the considerable potential of Brazilian wood varieties in producing aged spirits, presenting a viable alternative for *cachaça* producers. Given their ready availability within Brazil, the utilization of native woods not only reduces production costs but also enhances the value of sugarcane spirits through the incorporation of unique wood-derived

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compounds, fostering the creation of products with distinct sensory profiles (Bortoletto et al., 2021).

The Amazon rainforest, a reservoir of Brazilian biodiversity boasting over 11.6 thousand tree species, holds immense potential for beverage storage. Despite this richness, only a limited number of native species are currently employed for this purpose. Although some studies have explored the feasibility of utilizing specific Brazilian woods for *cachaça* aging, a comprehensive assessment of the sensorial quality of stored *cachaça* in these proposed woods remains lacking. Hence, the evaluation of the chemical and sensory characteristics of *cachaças* stored with Amazon and oak wood chips assumes paramount significance, as it holds the potential to yield products with distinctive sensorial attributes, marked by the designation “*cachaça* stored in Amazon wood.” Such labeling not only adds value to *cachaça* but also stimulates its commercial appeal. Therefore, the aim of this study was to comprehensively evaluate the physicochemical properties, temporal profiles, and consumer acceptability of *cachaças* stored with various wood types sourced from the Amazon region—namely, *cumarurana* (CM), *jatobá* (JT), and *louro-vermelho* (LV)—in comparison to oak, a wood traditionally utilized in *cachaça* aging.

2 MATERIAL AND METHODS

2.1 Obtaining cachaça stored with different Amazonian woods

The Amazon species studied were CM (*Dipteryx polyphylla* (Huber) Ducke), JT (*Hymenaea courbaril* L.), and LV (*Nectandra rubra* (Mez) C.K. Allen), which were collected in the management area in Silves, Amazonas, Brazil, at the Monte Verde farm of Precious Woods Amazon Company, which is a company certified by FSC® (Forest Stewardship Council®). Oak (*Quercus* sp) wood was collected from wooden casks in a cooperage of Brodowski, São Paulo, Brazil.

Commercial and freshly distilled cachaça, boasting an alcohol content of 44 g/L, was utilized for this study. The cachaça was sourced from a traditional alembic situated in the state of Minas Gerais, Brazil. The procurement of wooden cubes and subsequent heat treatment procedures followed the methodology outlined by Simioni et al. (2018), which was based on the protocols established by Castro et al. (2015). The latter study involved the immersion of wooden cubes in hydroalcoholic solutions to glean physicochemical insights into Brazilian wood species.

Wooden cubes crafted from both Amazonian and oak woods were procured for this experiment. These cubes underwent meticulous heat treatment before being immersed in glass containers containing *cachaça*. The aging process spanned a duration of 6 months at ambient temperature, shielded from light exposure. Each glass container housed a wooden cube in a ratio of one cube per 250 mL of *cachaça*. The cachaça with the wooden cubes was stored in a dry, dark environment, with a temperature ranging from 18 to 23°C. Following the maturation period, the wooden cubes were removed, and the cachaça underwent comprehensive physicochemical and sensory evaluations.

2.2 Physico-chemical analysis

The physicochemical analyses, as mandated by Brazilian legislation under Normative Instruction No. 13 (Brasil, 2022), were conducted on both unaged *cachaça* and *cachaças* aged with Amazonian woods and oak. These analyses encompassed the determination of various parameters, including dry extract, furfural, copper, methanol, ethanol, higher alcohols, aldehydes, esters, volatile acidity, and secondary compounds. The methodologies employed for these analyses adhered to the Official Methods of Analysis of Distilled Beverages, as outlined in Normative Instruction No. 24 dated 08/09/2005 (Brasil, 2005).

Additionally, assessments of color and volatile compounds were conducted specifically on the aged *cachaças*.

2.2.1 Color measurement

Color parameters were measured using the Konica Minolta Spectrophotometer, model CR CM-5, with illuminant D65, SCE reading mode, 10° angle, and CIE L * C * h color system, which was previously calibrated. The data were presented as the averages of the three measurements.

2.2.2 Identification of volatile compounds in aged cachaças by GC-MS

The identification of volatile compounds in stored *cachaças* was conducted using solid-phase microextraction (SPME) coupled with gas chromatography-mass spectrometry (GC-MS) at the Center for Analysis and Chemical Prospecting (CAPQ/Department of Chemistry/UFLA). Each *cachaça* sample, divided into 5 mL portions, was placed in a 20-mL screw-cap SPME tube (Sigma-Aldrich, Bellefonte, PA, USA), and volatile compounds were extracted via headspace SPME utilizing a DVB/CAR/PDMS (Divinylbenzene, Carboxen, and Polydimethylsiloxane) fiber (Arthur & Pawliszin, 1990). Extraction was carried out at a temperature of 60°C for a duration of 40 min.

For the separation step, a GC-MS QP 2010 Ultra (Shimadzu, Japan) gas chromatograph equipped with an AOC-5000 (Shimadzu, Japan) automatic injector for liquids and gases, and an HP-5 (5% phenyl-95% dimethyl siloxane) column (30 m × 0.25 mm × 0.25 µm) was employed. Injector, interface, and ion detector temperatures were maintained at 250, 240, and 200°C, respectively. The SPME fiber was exposed to the injector for 1 min, operating either in splitless mode or split mode at a 1:20 ratio based on sample peak intensity. Helium (grade 5.0) served as the carrier gas with a flow rate of 1.0 mL/min. The GC oven temperature was initially set at 35°C for 5 min, then ramped at a rate of 3°C/min to 130°C, and then increased at 10°C/min to 260°C.

Identification of volatile compounds was facilitated by the Automated Mass Spectral Deconvolution and Identification System (AMDIS) v. 2.63 software. Mass spectra of each peak were compared with those in the NIST library using the Mass Spectral Search Program v. 1.7 (NIST, Washington DC, USA), alongside experimentally determined retention indices (RI Exp.) and those reported in the literature (RI Lit.) (Adams, 2007; NIST, 2013). Spectra with a similarity exceeding 80% were considered for analysis. Experimental retention indices were derived from injecting a homologous series of alkanes.

2.3 Sensory evaluation

Temporal profile and acceptance tests were conducted at the Sensory Analysis Laboratory of the Department of Food Science at the Universidade Federal de Lavras. These assessments took place in individual sensory booths, characterized by white lighting, standardized temperature, and adequate ventilation. Prior to testing, tasters received comprehensive instructions on the protocols for sensory analysis, and ample water was provided for palate cleansing between samples.

Furthermore, ethical clearance for this study was obtained from the Ethics and Research with Humans Committee of the Universidade Federal de Lavras under protocol number 32991814.7.0000.5148. Tasters were required to meet specific criteria, including an interest in evaluating the beverage, availability of time, regular consumption of distilled beverages, being at least 18 years of age and having no aversions or allergies to the product.

2.4 Temporal dominance of sensations analysis

The selection of tasters was conducted utilizing the sequential method proposed by Wald (1945). For this purpose, two commercial *cachaças* from different brands (*Cachaça 51*[®] and *Cachaça João Mendes*[®]) were chosen based on the outcomes of a preliminary test, wherein samples exhibiting a significant difference ($p < 0.05$) were identified via triangular tests.

Twenty-two individuals with an interest in evaluating *cachaça* were recruited for participation. Subsequently, their ability to discriminate between samples was assessed through a series of triangular tests (Meilgaard et al., 2006).

Wald's sequential analysis was employed, utilizing predetermined values: $P = 0.30$ (maximum acceptable inability), $PI = 0.70$ (minimum acceptable proficiency), and associated risks of $a = 0.10$ (probability of accepting a candidate lacking sensory acuity) and $b = 0.10$ (probability of rejecting a candidate possessing sensory acuity).

The Wald graph, constructed based on defined parameters, facilitated the selection or rejection of tasters based on their performance in correctly identifying samples across a set number of trials. Ultimately, 12 judges were selected following eight triangular tests, all falling within the acceptance region of the graph. The selected tasters ranged in age from 24 to 50 years and comprised 2 females and 10 males.

Tasters were provided with samples of *cachaça* stored with Amazonian woods and oak to generate an attribute list. They were instructed to taste the samples and record all perceived sensations using Kelly's repertory grid (Moskowitz, 1983). Subsequently, assessors' responses were collected and analyzed under the guidance of a discussion leader. During this process, irrelevant descriptors were eliminated, and synonymous terms were merged. Ultimately, only the most frequently cited attributes were retained for further temporal dominance of sensations (TDS) analysis.

As a result, the attributes and their respective definitions were determined by consensus among the consumers, which

included: vanilla flavor, alcoholic flavor (taste sensation of alcohol, solvent, medicinal, and ethanol), sweetness (basic taste), pungency (burning sensation in the throat), woody flavor (taste sensation similar to wood), spiciness (perception of burning sensation on the tongue), and off-flavor (any different taste that provokes an unpleasant taste sensation). Additionally, tasters established the total duration of sensory analysis (30 s) and the appropriate sample volume for ingestion (5 mL) during this session.

Following the establishment and description of attributes, the tasters participated in two sessions, following the protocol outlined by Albert et al. (2012). In the initial session, tasters were introduced to the TDS method and were familiarized with the SensoMaker software (version 1.91), which was utilized for data acquisition and analysis (Nunes & Pinheiro, 2012). Subsequently, in the second session, tasters engaged in a TDS analysis simulation, during which they evaluated various samples of aged *cachaça*. They were instructed that the dominant taste represents the clearest and most prevalent sensation at any given moment (Pineau et al., 2009).

Subsequently, 12 tasters evaluated *cachaça* stored with Amazonian woods CM, LV, JT, and oak. Samples were assessed in three replicates across three sessions, with four samples evaluated per session. Presented at room temperature, samples were arranged in a monadic order (Macfie et al., 1989) within disposable glass cups sealed with lids to prevent ethanol evaporation, each labeled with a unique three-digit code. Tasters were tasked with consuming a 5 mL sample of *cachaça*, followed by a 2-s delay before swallowing, and then evaluating the sample for a duration of 30 s immediately post-swallowing. Subsequently, they selected dominant attributes using buttons, with the dominant attribute defined as the most prevalent sensation during the 30-s evaluation, permitting the selection of one or more attributes.

2.5 Acceptance test

Acceptance tests were conducted with a cohort of 60 consumers of aged alembic *cachaça*, meeting specific criteria, including a minimum consumption frequency of once a week and an age range between 18 and 60 years old. The gender distribution comprised 29% females and 71% males. Consumers were tasked with evaluating the samples based on appearance and overall liking, using a hedonic 9-point scale ranging from 1 (dislike extremely) to 9 (like extremely) (Stone & Sidel, 2004). Each 5 mL sample was presented in a glass cup labeled with a unique three-digit code, employing a balanced order determined by the Williams Latin Square design (Macfie et al., 1989).

2.6 Data analysis

2.6.1 Physical-chemical data

The experimental design employed was a completely randomized design (CRD), comprising four treatments with four replicates each. The treatments consisted of *cachaça* stored with oak, CM, JT, and LV.

For each component recommended by legislation (dry extract, furfural, methanol, ethanol, higher alcohols, aldehydes, esters, volatile acidity, copper, secondary compounds), the overall mean of all obtained values was computed. These values were then compared against quality standards stipulated by legislation to ascertain the compliance with the *cachaça* samples.

Color parameters were subjected to analysis of variance (ANOVA) followed by Tukey's test in instances where a significant effect ($p < 0.05$) was observed. Principal component analysis (PCA) was performed on the color parameter averages, with a matrix structured with samples as rows and variables as columns. Components corresponding to prominent points in the loading plot were identified within PC1 and PC2.

Regarding volatile compounds, the average chromatogram signal for each sample, prepared via solid-phase microextraction (SPME) and analyzed using gas chromatography-mass spectrometry (GC-MS), was truncated to 3.296 min. Alignment methods were applied to the chromatograms, followed by normalization to the maximum value and centering on the mean. The resulting dataset was organized into a matrix with samples as rows and variables as columns. Subsequently, PCA was conducted on this dataset, with components linked to significant points in the loading plot identified within PC1 and PC2.

2.6.2 Sensorial data

To evaluate the temporal dynamics of perceived attributes in the *cachaças*, dominance rates were computed and plotted over time, yielding TDS curves, following the methodology proposed by Pineau et al. (2009). Dominance rates were derived by dividing the number of mentions of an attribute (across all replications) by the total number of trials (judges \times replications). These curves depict the attributes perceived over evaluation time, alongside the chance line and significance line. The chance line represents the dominance value achievable by chance alone, whereas the significance line denotes the minimum dominance value considered statistically significant at a 95% confidence level. The latter was computed using the confidence interval of a binomial proportion based on a normal approximation (Pineau et al., 2009). TDS sensory data were analyzed through TDS curves, focusing on the dominance rates of significant sensations, that is, attributes reaching the significance level as determined by the curves (Pineau et al., 2009).

Additionally, TDS difference curves were generated to compare *cachaça* samples. These curves were created by subtracting the dominance rates of two samples for each attribute at each time point, with the difference considered significant only when deviating significantly from zero. The significance of the difference was determined as described by Pineau and Schlich (2015).

ANOVA was performed to investigate the effects of different *cachaça* types on appearance and overall liking data. In cases where a significant effect ($p < 0.05$) was detected, Tukey's test was employed for pairwise comparisons.

Furthermore, the TDS parameter maximum dominance rate (DR) was computed from the TDS curves. Subsequently, the DRs obtained solely for significant attributes for each *cachaça*

sample were subjected to PCA, with the average of overall liking serving as a supplementary variable. The maximum dominance rates were organized into a matrix of i lines (samples) and j columns (sensations and overall liking). The data were converted into a correlation matrix, and PCA was conducted using the R software. Scores and loading plots were generated from the first two principal components.

Throughout all physical-chemical and sensory analyses, SensoMaker software version 1.91 was utilized (Nunes & Pinheiro, 2012).

3 RESULTS AND DISCUSSION

3.1 Physico-chemical analysis recommended by the Brazilian legislation

Based on the findings of the physicochemical analysis (refer to Table 1), it was observed that both *cachaças* stored with all evaluated woods and those with no wood complied with the standards stipulated by Brazilian legislation, as outlined in Order No. 539 (Brasil, 2022). However, exceptions were noted for certain components in *cachaças* stored with oak and CM. Specifically, the aldehyde component, represented by acetaldehyde, exceeded the maximum permissible limit set by Brazilian legislation, with concentrations of 36.33 mg/100 mL for oak-stored *cachaça* and 40.52 mg/100 mL for CM-stored *cachaça*, surpassing the established threshold of 30 mg/100 mL. Additionally, the furfural component in oak-stored *cachaça* was found to be 5.72 mg/100 mL, exceeding the allowable limit of 5 mg/100 mL according to Brazilian regulations.

Cachaça samples, both with no wood and those stored with oak, CM, JT, and LV, exhibited varying levels of aldehydes, with values of 77.30, 36.33, 40.52, 29.98, and 18.80 mg/100 mL, respectively. This observation suggests that the maturation process contributed to the reduction of aldehyde concentrations in the *cachaças*, consequently enhancing their quality.

Acetaldehyde, the primary aldehyde found in *cachaças*, can constitute up to 90% of the total aldehyde concentration. Its presence serves as a significant indicator of distilled beverage maturation, as its content diminishes under anaerobic conditions (Cardoso, 2020). This phenomenon likely explains the observed reduction in aldehyde content across all evaluated *cachaça* samples in this study.

Considering acute aldehyde toxicity, which occurs with oral intake of 1930 mg/kg acetaldehyde, the maximum exposure in our study was 24.3 mg, translating to a maximum of 0.4 mg/kg per taster, significantly below the threshold for acute intoxication.

In a study by Caruso et al. (2008), it was found that 31 out of 60 *cachaça* samples exceeded the established maximum limit of 30 mg/100 mL for acetaldehyde, with concentrations reaching up to 120 mg/100 mL, surpassing those observed in our study.

The evaluation of the physicochemical profile of *cachaças* stored in oak and *amburana* barrels by Santiago et al. (2014) revealed inadequate acetaldehyde concentrations in the "head"

Table 1. Physico-chemical analysis recommended by the Brazilian legislation of *cachaças* stored with oak, *jatobá* (JT), *cumarurana* (CM), and *louro-vermelho* (LV) wood cubes.

Analysis	Oak	JT	LV	CM
ethanol ¹	38.85 ± 0.02	38.05 ± 0.021	38.41 ± 0.06	41.31 ± 0.07
dry extract ²	0.88	0.60	0.33	0.70
volatile acidity ³	65.83 ± 0.03	67.38 ± 0.02	50.91 ± 0.08	56.43 ± 1.71
higher alcohols ^{3*}	202.24 ± 2.27	196.71 ± 4.4	194.17 ± 1.68	195.85 ± 2.72
furfural ³	5.72 ± 0.14	2.53 ± 0.06	1.13 ± 0.01	3.66 ± 0.04
aldehydes ³	36.33 ± 0.30	29.98 ± 0.01	18.80 ± 0.03	40.52 ± 0.19
esters ³	35.49 ± 1.16	21.39 ± 0.29	13.89 ± 0.02	26.91 ± 1,03
copper ⁴	3.93 ± 0.05	2.66 ± 0.03	1.51 ± 0.02	2.30 ± 0.06
methanol ³	0.75 ± 0.01	0.58 ± 0.02	0.79 ± 0.04	0.69 ± 0.04
2-butanol ³	5.02 ± 0.02	5.10 ± 0.07	3.54 ± 0.02	3.45 ± 0.02
1-butanol ³	2.05 ± 0.01	2.15 ± 0.03	1.73 ± 0.01	1.71 ± 0,00
secondary compounds ³	345.62 ± 3.91	317.70 ± 4.85	278.95 ± 1.46	323.37 ± 5.70

¹% v/v; ²g/L; ³mg/100 mL-1 anhydrous alcohol; ⁴mg/L; *3-methyl-1-butanol + 2-methyl-1-propanol +1-propanol.

fraction of the *cachaças*, reaching 80.88 mg/100 mL, surpassing the aldehyde values found in our study. High aldehyde levels are often found in the “head” fraction and at the beginning of the “heart” fraction (Cardoso, 2020).

The elevated aldehyde concentrations observed in our study are likely attributable to the utilization of the early “heart” fraction for *cachaça* aging or potentially due to oxidation or the activity of contaminating bacteria (Barbosa et al., 2022; Cardoso, 2020).

Regarding furfural, no wood-stored *cachaça* and *cachaças* stored with oak, CM, JT, and LV displayed furfural values of 0.28, 5.72, 3.66, 2.53, and 1.13 mg/100 mL, respectively, with only oak-stored *cachaça* exceeding the established maximum limit of 5 mg/100 mL.

In terms of toxicity, oral ingestion of 65 mg/kg of furfural results in acute toxicity, yet our study presented a maximum exposure of 30 mg, equating to a maximum of 0.05 mg/kg per taster, well below the threshold for acute intoxication.

Zacaroni et al. (2011) suggested that higher furfural levels may originate from the thermal treatment of barrels or the burning of sugarcane before harvesting or distillation in the presence of residual sugar. Miranda et al. (2008) observed that the furfural content increased with aging duration in oak barrels, albeit without surpassing Brazilian legislation standards.

In our study, the elevated furfural content could be attributed to the use of thermo-treated oak barrel wood cubes, which underwent secondary thermal processing at 200°C for 120 min. Furfural serves as a marker of *cachaça* aging due to its aromatic nature, likely originating from Maillard and caramelization reactions involving wood-derived compounds that are extracted and incorporated into the beverage (Barbosa et al., 2022).

3.2 Color analysis

The color analysis aimed to compare the hue, lightness, and saturation of *cachaças* stored in Amazonian woods with those stored in oak barrels. Regarding the lightness parameter

(L*), indicative of luminosity, no significant differences were observed among the samples (Table 2). Similarly, the chroma, which gauges color saturation or intensity, showed no discernible variation across the samples.

However, concerning the hue angle, reflective of the predominant tone, it was noted that *cachaça* stored in oak barrels did not deviate from those stored in CM and JT woods but differed notably from LV-stored *cachaça*. Furthermore, *cachaça* stored in JT wood exhibited a darker hue compared with CM. Consequently, it was observed that *cachaças* stored in oak, CM, and JT displayed a yellowish-orange hue, whereas LV-stored *cachaça* diverged, featuring a translucent light blue hue that was distinct from the others.

Cardoso (2020) notes that both the type and hue of wood, along with the duration of aging, exert a significant influence on the coloration of *cachaças*. The organic components inherent to wood, such as tannins and resins, contribute to the progressive darkening or intensification of the yellow hue in sugarcane spirits.

In a study by Castro et al. (2015), the characterization of extracts derived from hydroalcoholic solutions and cubes of JT, CM, and LV woods revealed noteworthy variations in tannin content, with JT wood exhibiting notably higher levels compared with the other woods. Conversely, LV wood showcased the lowest tannin content, potentially influencing the color profile of the *cachaças* examined in this study. Notably, *cachaça* stored in LV wood exhibited a lighter hue, contrasting with the deeper coloring of JT-stored *cachaça*, which notably deviated even from CM-stored *cachaça*, displaying a more pronounced color intensity.

Consequently, the aging process of *cachaças* fosters the development of a yellowish tint, attributable to the chemical constituents accrued during maturation, which contribute to the intricate aromas and flavors cherished by consumers (Ratkovich et al., 2023; Silveira & Barbeira, 2022). Consequently, *cachaça* stored in CM and JT woods, exhibiting hues akin to those stored in oak, demonstrates considerable promise as a viable alternative to traditional oak aging methods.

3.3 Principal component analysis: physical-chemical analyses

To explore the similarities and disparities in the physico-chemical profiles of *cachaças* stored in Amazonian woods and oak, we conducted an exploratory analysis using PCA (Figure 1), where the scores represent the *cachaças* and the loadings represent the physical-chemical analyses. The two principal components captured 89.94% of the total data variance, with PC1 contributing 63.64% and PC2 contributing 26.3%.

Observations revealed that *cachaças* stored in oak and JT exhibited similar loadings in PC1, showcasing higher concentrations of dry extract, furfural, copper, esters, aldehydes, isoamyl alcohol, n-butyl alcohol, sec-butyl alcohol, as well as elevated acidity and total secondary components.

Throughout the aging process, there was a noticeable increase in dry extract within the *cachaças*, potentially attributable to the presence of up to 40% phenolic compounds derived from lignin and tannic substances, alongside the degradation of hemicellulose (Guimarães et al., 2020). These processes contribute to the formation of compounds pivotal in flavor, aroma, and color modification, such as phenolic compounds, sugars, organic acids, and volatile oils (Corbion et al., 2023).

Table 2. Colorimetric parameters of *cachaças* stored with Amazon and oak woods.

	L	Chroma	Hue
Oak	10.69 ± 0.49 a	1.17 ± 0.85 a	91.20 ± 10.35 ab
LV	10.43 ± 0.16 a	0.73 ± 0.01 a	244.95 ± 2.76 c
CM	10.40 ± 0.55 a	0.74 ± 0.50 a	109.41 ± 9.04 b
JT	11.06 ± 0.93 a	1.34 ± 0.67 a	85.57 ± 0.63 a

*Different vertical letters indicate significant differences between the samples according to Tukey's test; JT: *jatobá*; LV: *louro-vermelho*; CM: *cumarurana*.

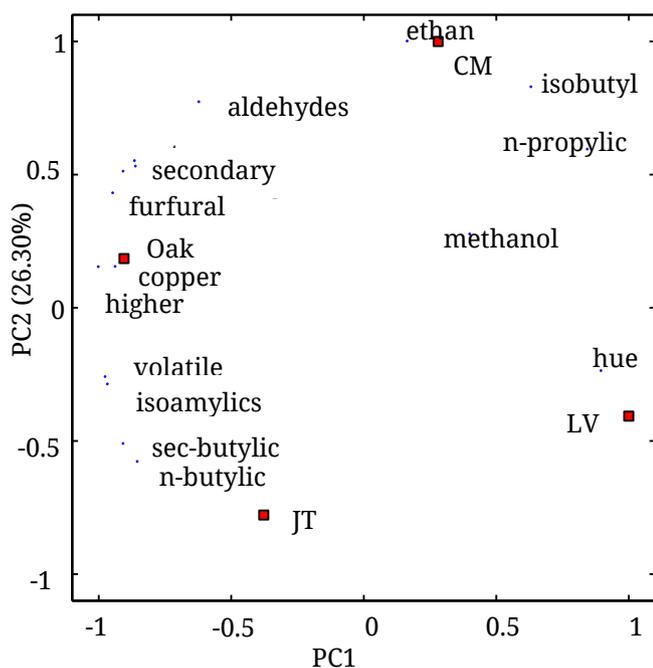


Figure 1. Principal component analysis of physical-chemical analysis of *cachaças* stored with oak and the Amazonian woods *jatobá* (JT), *cumarurana* (CM), and *louro-vermelho* (LV).

Esters, higher alcohols, aldehydes, and organic acids constitute the secondary compounds in *cachaças* responsible for the characteristic aroma and flavor of these beverages, playing a significant role in shaping the sensorial profile of the distillates (Bortoletto et al., 2021).

During wood toasting, the degradation of hemicellulose yields furanic aldehydes like furfural and 5-hydroxymethyl-furfural (5-HMF), contributing to the color and distinct aroma of aged spirits (Santiago et al., 2020). Additionally, copper, at normal levels, is desirable in *cachaças* as it aids in the elimination of certain undesirable odors (Cardoso, 2020).

Throughout fermentation, yeast generates higher alcohols like isoamyl, isobutyl, and n-propyl alcohols, imparting aroma and conferring distinctive scents to distilled beverages like sugarcane spirits (Bortoletto et al., 2018). Despite higher values in *cachaças* stored in oak and JT, n-butyl and sec-butyl alcohols, considered contaminants per Brazilian legislation, remained within permissible limits across all samples (Brasil, 2022).

Cachaças stored in JT and oak share similarities in PC1, characterized by elevated copper and higher alcohol concentrations. Conversely, *cachaça* stored in CM exhibited higher ethanol levels, whereas LV demonstrated higher hue angle values, consistent with its translucent hue (light blue) as indicated in the attributes, and their respective definitions were determined by consensus among the consumers. Furthermore, CM and LV *cachaças* exhibited the highest levels of isobutyl and n-propyl alcohols.

Finally, the PCA revealed no significant difference in methanol levels between *cachaças* stored in oak, LV, and CM, whereas *cachaça* stored in JT exhibited the lowest methanol levels among the samples.

3.4 Volatile compounds

Figure 2 depicts the PCA conducted on volatile compounds detected in *cachaças* stored in Amazonian woods and oak, analyzed via GC-MS. The analysis is divided into three parts: Figure 1A presents the score plot representing the new variable space defined by the first (PC1) and second (PC2) main components, where the four *cachaça* samples are projected based on the average chromatogram of the three replicates obtained. Figures 1B and 1C illustrate principal components 1 and 2, respectively, constructed from the chromatogram data, together accounting for 87.76% of the total data variance.

According to this PCA, 12 compounds significantly contributed to differentiating the samples. Table 3 presents these volatile compounds detected by GC-MS analysis, along with their odor characterizations. The predominant volatiles identified were: 1,1-diethoxyethane, 3-methyl-1-butanol, 2-methyl-1-butanol, ethyl hexanoate, phenylethyl alcohol, ethyl octanoate, ethyl decanoate, ethyl dodecanoate, ethyl tetradecanoate, nonanal, furfural, and ethyl succinate.

Among the alcohols identified in this study, 2-methylpropanol, 1-propanol, 3-methyl-1-butanol, 2-methyl-1-butanol, and phenylethyl alcohol were detected, likely originating from alcoholic fermentation by yeast (Ratkovich et al., 2023).

The concentration and proportion of esters depend on various factors, primarily yeast type and quantity, aeration, and agitation during fermentation, temperature, and broth volume.

Table 3. Volatile compounds identified in *cachaças* stored with Amazonian woods and oak which most influenced the PCA analysis.

Volatile compounds	Odor
3-Methyl-1-butanol	Whiskey (B), alcohol, banana, sweet (C)
2-Methyl-1-butanol	Alcohol, banana, medicinal, solvent (A)
Ethyl hexanoate	Green apple (A, D) fruity, sweet I
Phenyl ethyl alcohol	Floral (F)
Ethyl octanoate	Apple, sweetish, fruity (A), sweet (D), green frutal, limon (E)
Ethyl decanoate	Grape (B), pleasant, soap (D), brandy (G)
Ethyl dodecanoate	Herbal leaf (B), soapy (D)
Ethyl tetradecanoate	-
Nonanal	Citrus-like, soapy (H)
Furfural	Husk (A), sweet fruit, flower(E)
Diethyl succinate	-
1,1-Diethoxyethane	Refreshing, fruity, green (I)

(A) Meilgaard (1975); (B) Acree e Heinrich (1997); (C) Angelino (1991); (D) Siebert et al. (2005); (E) Janzanti (2004); (F) Souza et al. (2006); (G) Pino et al. (2012); (H) Czerny et al. 2008; (I) Williams (1974).

Low ester concentrations contribute to fruity aromas, whereas high concentrations can yield undesirable flavors in *cachaça* (Cardoso, 2020). The identified esters in this study included ethyl acetate, ethyl hexanoate, ethyl octanoate, ethyl decanoate, ethyl dodecanoate, ethyl tetradecanoate, and ethyl succinate.

Another compound class found in *cachaça* is acetals, such as 1-ethoxy-1-pentoxo-ethane, with high levels contributing to the beverage's final aroma. These compounds form when aldehydes react with alcohols, reducing the free aldehyde content and mitigating their pungent odor (Lima et al., 2022; Paolini et al., 2022; Qiao et al., 2023).

Aldehydes can originate from alcoholic fermentation processes with partial oxygenation. Additionally, aldehydes can undergo further oxidation, producing acetic acid during acetic fermentation (Cardoso, 2020). In this study, acetaldehyde, nonanal, and furfural were identified. Acetaldehyde, particularly abundant in the "head" fraction of distillation, contributes to either a pungent or diluted fruit aroma, depending on concentration (Caetano et al., 2021).

Returning to the PCA (Figure 2), it is important to note that alignment issues affected the peaks of 3-methyl-butanol and 2-methyl-butanol compounds in PC1 and PC2, as

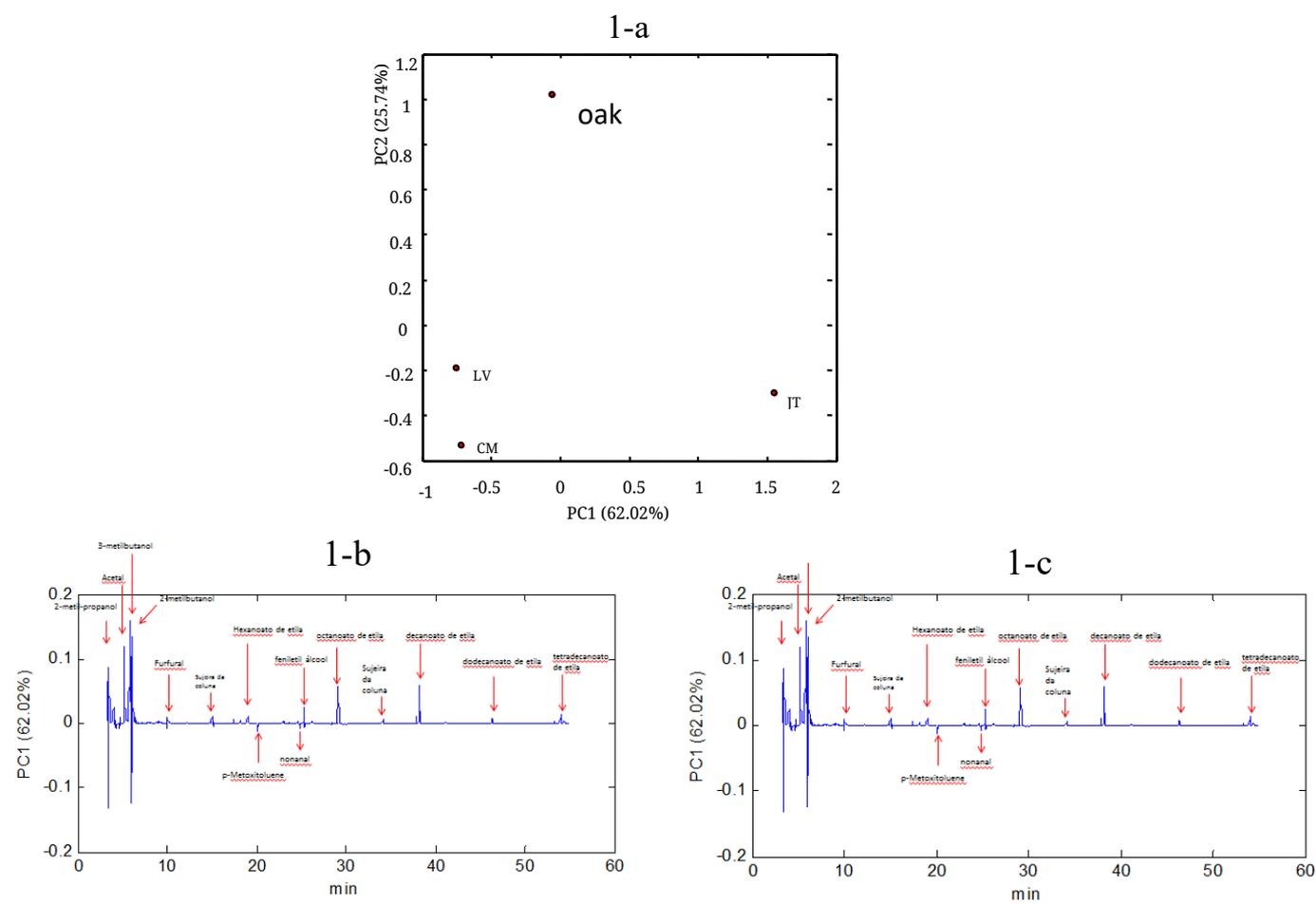


Figure 2. Principal component analysis of scores (1a) and loadings (1b and 1c) for volatile compounds from *cachaças* stored with Amazonian woods and oak. Samples: JT: *jatobá*; LV: *louro-vermelho*; CM: *cumarurana*.

well as 1,1-diethoxyethane in PC2. Consequently, these compounds were not considered in the PCA discussion, except for 1,1-diethoxyethane in PC1.

PC1 (Figure 1B), representing approximately 62% of data variance, was primarily associated with volatile compounds such as 1,1-diethoxyethane, furfural, ethyl hexanoate, phenylethyl alcohol, ethyl octanoate, ethyl decanoate, ethyl dodecanoate, and ethyl tetradecanoate. The sample with the highest abundance of these compounds, located in the IV quadrant of the score plot, corresponds to *cachaça* stored in JT. Consequently, JT *cachaça* is characterized by refreshing aromas reminiscent of herbal leaves, green apple, green fruit, lemon, fruity notes, grapes, floral sweetness, and pleasant woodiness.

PC2 (Figure 1C), accounting for 25.74% of the data variance, was mainly linked to volatile compounds such as nonanal, phenylethyl alcohol, ethyl succinate, ethyl decanoate, ethyl dodecanoate, and ethyl tetradecanoate. Samples with the highest abundance of these compounds, located in quadrants III and IV of the score plot, correspond to *cachaças* stored in LV, CM, and JT. These samples are characterized by citrusy, herbal, floral, grape, pleasant, and soapy notes.

PC2 is also associated with volatile compounds such as furfural, ethyl hexanoate, and ethyl octanoate, with the sample exhibiting the highest abundance of these compounds positioned in quadrant II of the score plot, corresponding to *cachaça* stored in oak. Oak-stored *cachaça* presents aromatic qualities similar to JT-stored *cachaça*, including green apple, green fruit, lemon, fruity, floral sweetness, and woodiness.

In previous studies, Cardeal et al. (2008) and Pino et al. (2012) identified compounds like 3-methyl-1-butanol, 2-methyl-1-butanol, ethyl octanoate, and ethyl hexanoate in *cachaças*, both stored with wood and without wood. Additionally, Santiago (2016) identified volatile compounds, including 1,1-diethoxyethane, 3-methyl-1-butanol, 2-methyl-1-butanol, ethyl decanoate, ethyl dodecanoate, and ethyl octanoate, in *cachaças* stored in oak and Brazilian native woods like jatobá.

In summary, the GC-MS analysis detected 31 compounds in the *cachaças* evaluated, and a supplementary table with theoretical and experimental retention index information, along with match data, is provided. Moreover, ethanol, acetaldehyde, 1-propanol, ethyl acetate, and 2-methyl-1-propanol were identified in all *cachaças* but were not included in the PCA due to a signal cutoff at 3.296 in the chromatogram.

3.5 Sensory analysis

TDS curves of all *cachaças* are illustrated in Figure 3, and both dominant and non-dominant attributes were reported for each *cachaça* sample. In this figure, we also showed the TDS difference curves comparing *cachaça* stored with oak with *cachaças* stored with Amazonian woods. TDS showed that there was a variation in the sensory profile in all types of *cachaças*.

The sensory profile of oak-stored *cachaça* was characterized by a notable presence of alcoholic and woody flavors, perceived significantly after 17 and 20 s, respectively. Notably, while both flavors were perceived significantly, the dominance of

the alcoholic flavor was higher compared with the wood flavor. Additionally, vanilla notes became significant toward the end of the evaluation period.

In contrast, *cachaças* stored in Amazonian woods exhibited distinct sensory profiles. LV *cachaça* was described with significant dominance of alcoholic flavor from approximately 18 s, and off-flavor became prominent toward the end of ingestion. CM *cachaça* showed a significant perception of the alcoholic flavor and spicy attributes from 15 and 20 s, respectively, with alcoholic flavor dominance surpassing the spicy notes. In JT *cachaça*, alcoholic flavor predominated after approximately 20 s.

Significant sensory differences were observed between oak-stored *cachaça* and those stored in Amazonian woods (Figure 3). Oak-stored *cachaça* displayed higher dominance rates of wood flavor and vanilla attributes toward the end of the ingestion period (17–30 s) compared with *cachaças* stored in Amazonian woods. These attributes are commonly associated with the aging process and sensory characteristics of *cachaça* (Ratkovich et al., 2023).

LV *cachaça* exhibited wood flavor dominance at the beginning (~6 s) and off-flavor dominance from 11s, contrasting with oak-stored *cachaça*. CM *cachaça* showed wood flavor predominance at the beginning (~5 s), similar to LV, and higher dominance of spicy attributes between 22 and 28 s compared with oak-stored *cachaça*. JT *cachaça* did not exhibit any attribute with higher dominance than oak-stored *cachaça*.

Alcoholic flavor was dominant in all samples, likely due to incomplete volatilization caused by aging in glass recipients containing wooden cubes. Additionally, *cachaça*'s inherently high alcohol content contributes to the natural perception of alcoholic flavor even after aging.

In terms of acceptance, *cachaças* received appearance ratings ranging from 7 to 8 ("like moderately" and "like very much"), except for LV *cachaça*, which scored lower (6 to 7, "like slightly" and "like moderately"). The *cachaças* stored in oak ($7.58 \pm 1.41b$), CM ($7.45 \pm 1.42b$), and JT ($7.45 \pm 1.59b$) did not differ statistically from each other in terms of appearance. In contrast, LV ($6.57 \pm 2.03a$) *cachaça* was the least accepted and differed significantly from the others, possibly due to its translucent hue (light blue) compared with the yellowish/orange color of other samples, which studies suggest positively influences the acceptance of stored *cachaças* (Odello et al., 2009; Simioni et al., 2018).

Overall liking scores ranged from 6 to 7 ("like slightly" and "like moderately") for all samples, except for LV *cachaça*, which scored an average of $5.81 \pm 2.24a$ ("neither like nor dislike" to "like slightly"), significantly differing from the *cachaça* stored in oak ($6.74 \pm 1.71b$), which obtained the highest overall liking score. The *cachaças* CM ($6.38 \pm 1.67ab$) and JT ($6.58 \pm 1.99ab$) did not show significant differences from the other samples. Consistently, LV *cachaça* was the least accepted sample, aligning with the results for the appearance attribute.

Previous studies by Serafim et al. (2013) found similar acceptance trends for *cachaças* stored in oak and other woods, supporting the findings of this study for oak-stored, JT, and CM *cachaças*.

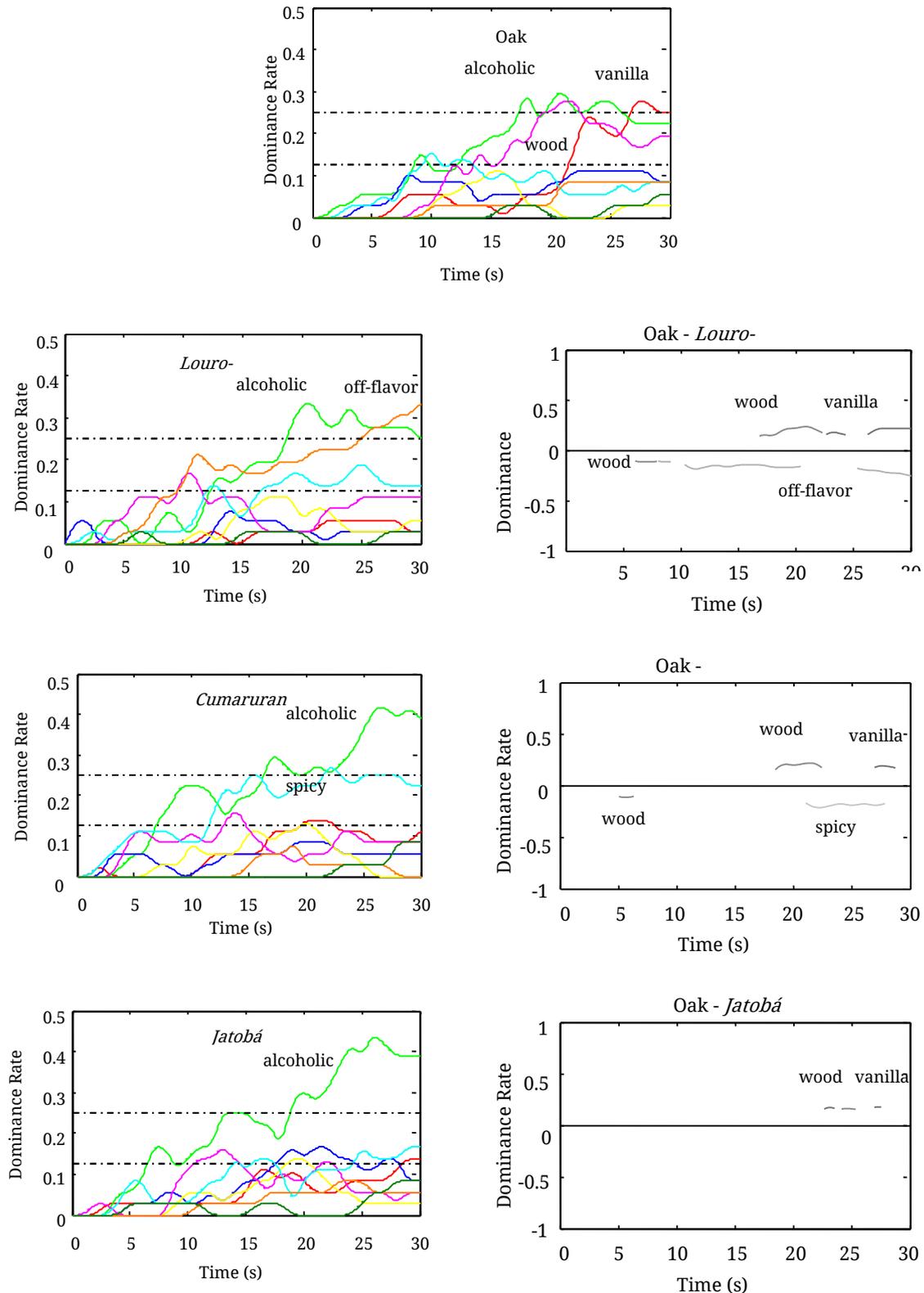


Figure 3. TDS of *cachaças* stored with oak, *louro-vermelho*, *cumarurana* and *jatobá* and TDS difference curves comparing *cachaças* stored with oak with the other *cachaças*.

It is evident that all samples, except for *cachaça* stored with LV, exhibited satisfactory sensory acceptability. *Cachaças* stored with CM and JT demonstrated comparable acceptability to oak-stored *cachaça*. This favorable reception could be attributed

to detecting wood flavor and vanilla attributes in oak-stored *cachaça*, coupled with the absence of the off-flavor attribute in CM and JT samples. Consequently, the alcoholic flavor attribute, consistently perceived across all samples via TDS testing,

appeared to exert minimal influence on sample acceptability, given its uniform perception in all *cachaças* analyzed.

The diminished acceptability of *cachaça* stored with LV may be linked to the significant detection of the off-flavor attribute in TDS analysis. Moreover, despite the wood flavor attribute predominating at the onset of LV *cachaça* evaluation compared with oak-stored *cachaça*, it did not positively impact sample acceptability, aligning with findings by Simioni et al. (2018). Notably, CM *cachaça* also exhibited initial wood flavor dominance, yet its acceptability was akin to oak, JT, and LV *cachaças*, according to overall liking grades.

To ascertain the drivers of liking for the samples, PCA was conducted using the maximum dominance rates of each significant attribute and overall liking data as supplemental variables.

According to the PCA scores and loading plots (Figure 4), oak-stored *cachaça* stood out in acceptance, likely due to the high dominance rates of the vanilla and wood flavor attributes. Similarly, CM and JT *cachaças* garnered acceptance, possibly owing to the high dominance rates of the spicy and alcoholic flavor attributes. In the TDS curves, the alcoholic flavor attribute was consistently perceived across all samples and could be deemed a neutral attribute, devoid of substantial influence on acceptance. Janzanti (2004) noted that the “initial burning” attribute positively influenced the acceptance of stored *cachaças*, akin to the spicy attribute in JT and CM *cachaças*.

Odello et al. (2009) and Yokota (2005) have reported a positive correlation between the preference for stored *cachaça* and various sensory attributes, including the intensity of yellow color, the initial and aftertaste of wood, sweetness, aroma of wood, vanilla, fruit, and viscosity. Consequently, it is notable

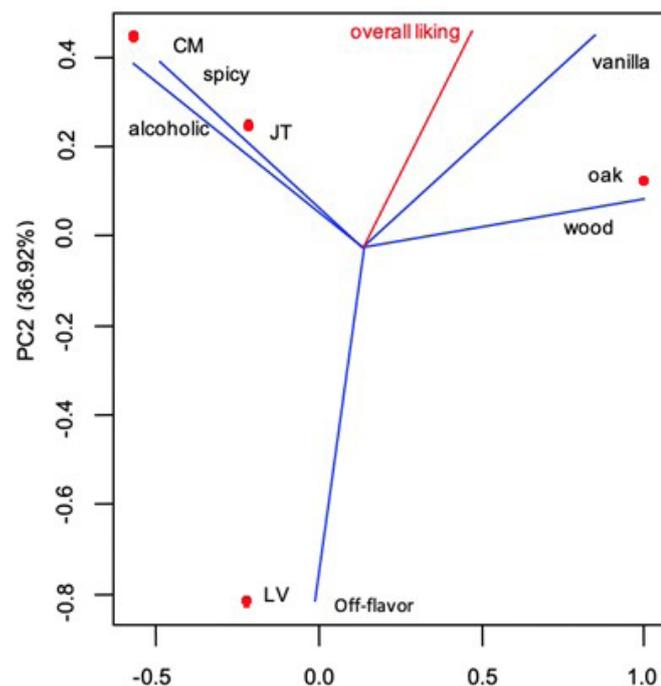


Figure 4. PCA scores and loadings for the maximum dominance rates from *cachaças* TDS curves. Overall liking was considered a supplementary variable. Samples: JT: *jatobá*; LV: *louro-vermelho*; CM: *cumarurana*.

that the attributes of wood flavor and vanilla, coincidentally highlighted in this study, were positively associated with the acceptance of stored *cachaça*.

In terms of the physical-chemical analysis, it was observed that higher concentrations of dry extract, furfural, copper, esters, aldehydes, isoamyl alcohol, n-butyl and sec-butyl alcohols, increased acidity, and total secondary components positively contributed to the overall liking of *cachaças* stored with JT and oak. This is evidenced by the positive weights assigned to these components in PC1 and PC2, respectively, as depicted in Figure 2. Additionally, volatile compounds such as ethyl hexanoate, ethyl octanoate, and furfural were also positively associated with the aroma profiles of *cachaças* stored with JT and oak, which exhibited aromas reminiscent of green fruits, husk, floral notes, and sweetness (Table 3). Therefore, it is plausible to infer that the wood flavor and vanilla attributes identified in oak-stored *cachaça* may be linked to these aromatic profiles.

Conversely, less acceptable samples, particularly *cachaça* stored with LV, displayed lower acceptance rates and were characterized by the presence of off-flavor attributes (Figure 4). Furthermore, the physical-chemical analysis revealed that *cachaça* stored with LV exhibited a higher hue angle, indicative of a translucent hue (light blue), along with elevated concentrations of isobutyl alcohol and n-propyl alcohol. It is noteworthy that *cachaça* stored with CM also exhibited higher concentrations of these alcohols, along with elevated ethanol levels.

Analysis of volatile compounds revealed that ethyl decanoate, ethyl dodecanoate, and nonanal significantly influenced the acceptance of *cachaças* stored with LV, JT, and CM, as indicated by their negative weights in PC2. These compounds imparted aromas resembling herbal leaf, grape, floral, pleasant notes, brandy, soapy, and citrus-like scents, which were associated with the off-flavor attribute identified in LV *cachaça* through TDS curves.

Previous studies have explored alternative woods to oak for aging *cachaça*. Alcarde et al. (2010) found that Brazilian woods such as ipê-roxo, amendoim, cabreúva, cerejeira, and pereira exhibited sensory acceptance comparable to that of *cachaça* aged in oak casks. Similarly, Faria et al. (2003) noted that amendoim and pereira could serve as viable alternatives to oak. Therefore, the use of jatobá wood for *cachaça* aging has been previously considered, demonstrating good acceptability. The present study enhances these findings through modern sensory and statistical techniques such as TDS analysis.

Based on the outcomes derived from both the TDS test and the sensory acceptance evaluation, it is apparent that *cachaças* stored with oak, JT, and CM were favored over others, whereas those with LV, featuring a predominant off-flavor attribute, were less preferred. The presence of attributes such as vanilla, wood flavor, spicy notes, and alcoholic flavor positively influenced the acceptance of *cachaças*. Conversely, higher levels of dry extract, furfural, copper, esters, aldehydes, isoamyl alcohol, n-butyl and sec-butyl alcohols, increased acidity, and total secondary components, along with the volatile compounds ethyl hexanoate, ethyl octanoate, and furfural, likely contributed positively to the overall liking of the *cachaças*. These compounds imparted

aromas reminiscent of green apple, fruitiness, sweetness, green frutal, lemon, husk, and floral notes. Conversely, factors such as higher hue angle values, indicative of a translucent hue (light blue), elevated concentrations of isobutyl alcohol and n-propyl alcohol, and the presence of volatile compounds such as ethyl decanoate, ethyl dodecanoate, and nonanal in high concentrations, likely contributed negatively to the overall liking of the *cachaças*, particularly with aromas evocative of brandy, soapiness, and citrus-like scents.

Therefore, the evaluated *cachaças* exhibited distinct sensory profiles, offering unique characteristics that can be marketed under the designation “*cachaça* stored in Amazonian wood.” This labeling strategy adds value to *cachaça*, facilitating its commercialization in domestic and international markets. Furthermore, it opens avenues for the development of differentiated *cachaças* tailored to meet the preferences of new consumer segments. Additionally, the prospect of utilizing Amazonian rainforest wood for crafting aging barrels represents a novel milestone in industrialization, offering a new source of raw material for cask production.

4 CONCLUSION

The evaluated *cachaças* complied with Brazilian regulatory standards, with aldehyde levels initially exceeding limits but decreasing during storage, thereby enhancing quality.

Cachaças stored with cumarurana (CM) and jatobá (JT) exhibited higher acceptability and potential for use, akin to oak-stored *cachaça*, likely due to the presence of wood flavor and vanilla attributes, and the absence of off-flavors. Conversely, louro-vermelho (LV) *cachaça*, with detected off-flavors, showed lower acceptability.

Elevated levels of volatile acidity, higher alcohols, dry extract, esters, furfural, and aldehydes, along with specific volatile compounds like ethyl hexanoate, ethyl octanoate, and furfural, likely contributed positively to *cachaças*' acceptability, with sweetish and husky aromas being emphasized. In contrast, LV *cachaça*, exhibiting lower acceptability, displayed characteristics such as a higher hue angle, lower concentrations of volatile acidity, furfural, aldehydes, esters, copper, 2-butanol, and secondary compounds compared with others.

Overall, Amazonian woods CM and JT showed greater potential for *cachaça* aging than LV, although all could substitute oak. The use of Amazon wood chips presents a viable strategy for enhancing *cachaças*' sensory attributes and standardizing their flavor, color, and aroma, thus adding value to the final product.

REFERENCES

- Abreu-Naranjo, R., Yordi, E. G., Radice, M., Scalvenzi, L., & Pérez-Martínez, A. (2023). Preliminary study regarding the optimisation of the accelerated ageing of sugar cane spirit by applying ultrasound-assisted extraction and white oak chips (*Quercus alba*). *Food Analytical Methods*, *16*, 1120-1130. <https://doi.org/10.21203/rs.3.rs-2061069/v1>
- Acree, T., & Heinrich, A. (1997). *Gas chromatography – olfactometry (GCO) of natural products*. Kovats retention indices sorted by DB5. Cornell University. Retrieved from <http://nysaes.cornell.edu/flavornet>
- Adams, R. P. (2007). *Identification of essential oil components by gas chromatography/mass spectrometry*. Allured Publishing Corporation.
- Albert, A., Salvador, A., Schlich, P., & Fiszman, S. (2012). Comparison between temporal dominance of sensations (TDS) and key-attribute sensory profiling for evaluating solid food with contrasting textural layers: Fish sticks. *Food Quality and Preference*, *24*(1), 111-118. <https://doi.org/10.1016/j.foodqual.2011.10.003>
- Alcarde, A. R., Souza, P. A., & Belluco, A. E. S. (2010). Aspectos da composição química e aceitação sensorial da aguardente de cana-de-açúcar envelhecida em tonéis de diferentes madeiras. *Ciência e Tecnologia de Alimentos*, *30*(Suppl. 1), 226-232. <https://doi.org/10.1590/S0101-20612010000500035>
- Angelino, S. A. G. F. (1991). Volatiles in beer. In H. Maarse (Ed.), *Volatile compounds in foods and beverages* (pp. 581-615). Marcel Dekker.
- Arthur, C. L., & Pawliszin, J. (1990). Solid phase microextraction with thermal desorption using fused silica optical fibers. *Analytical Chemistry*, *62*(19), 2145-2148. <https://doi.org/10.1021/ac00218a019>
- Barbosa, R. B., Santiago, W. D., Alvarenga, G. F., Oliveira, R. E. S., Ferreira, V. R. F., Nelson, D. L., & Cardoso, M. G. (2022). Physical-chemical profile and quantification of phenolic compounds and polycyclic aromatic hydrocarbons in *cachaça* samples aged in oak (*Quercus* sp.) barrels with different heat treatments. *Food Bioprocess Technology*, *15*, 1977-1987. <https://doi.org/10.1007/s11947-022-02853-w>
- Bortoletto, A. M., Silvello, G. C., & Alcarde, A. R. (2018). Good manufacturing practices, hazard analysis, and critical control point plan proposal for distilleries of *cachaça*. *Scientia Agricola*, *75*(5), 432-443. <https://doi.org/10.1590/1678-992X-2017-0040>
- Bortoletto, A. M., Silvello, G. C., & Alcarde, A. R. (2021). Aromatic profiling of flavor active compounds in sugarcane spirits aged in tropical wooden barrels. *Brazilian Journal of Food Technology*, *24*, e2019071. <https://doi.org/10.1590/1981-6723.07119>
- Brasil (2005). Ministério da Agricultura, Pecuária e do Abastecimento. Instrução normativa nº 24, de 8 de setembro de 2005: Padrões oficiais para análise físico-química de bebidas e vinagre. *Diário Oficial da União*.
- Brasil (2022). Ministério da Agricultura, Pecuária e Abastecimento. Portaria nº 539, de 26 de dezembro de 2022: Estabelece os padrões de identidade e qualidade da aguardente de cana e da *cachaça*. *Diário Oficial da União*.
- Caetano, D., Lima, C. M. G., Sanson, A. L., Silva, D. F., Hassemer, G. S., Verruck, S., Silva, G. A., Afonso, R. J. C. F., Coutrim, M. X., & Gregório, S. R. (2021). Descriptive screening and lexicon development of non-aged artisanal *cachaça* sensorial profile using principal component analysis and Kohonen artificial neural networks. *Journal of Sensory Studies*, *36*(3), e12645. <https://doi.org/10.1111/joss.12645>
- Cardeal, Z. L., de Souza, P. P., Gomes da Silva, M. D. R., & Marriott, P. J. (2008). Comprehensive two-dimensional gas chromatography for fingerprint pattern recognition in *cachaça* production. *Talanta*, *74*(4), 793-799. <https://doi.org/10.1016/j.talanta.2007.07.021>
- Cardoso, M. G. (2020). *Produção de aguardente de cana* (4th ed.). Ed. UFPA.
- Caruso, M. S. F., Nagato, L. A. F., & Alaburda, J. (2008). Avaliação do teor alcoólico e componentes secundários de *cachaças*. *Revista do Instituto Adolfo Lutz*, *67*(1), 28-33. <https://doi.org/10.53393/rial.2008.67.32786>

- Castro, J. P., Perígolo, D. M., Bianchi, M. L., Mori, F. A., Fonseca, A. S., Alves, I. C. N., & Vasconcellos, F. J. (2015). Uso de espécies amazônicas para envelhecimento de bebidas destiladas: Análises física e química da madeira. *CERNE*, 21(2), 319-327. <https://doi.org/10.1590/01047760201521021567>
- Castro, M. C., Bortoletto, A. M., Silvello, G. C., & Alcarde, A. R. (2020). Maturation related phenolic compounds in cachaça aged in new oak barrels. *Journal of the Institute of Brewing*, 127(1), 70-77. <https://doi.org/10.1002/jib.629>
- Conceição, V. S. da Rocha, A. M., Silva, M. S., Soares, P. M., & Lopes, J. M. (2020). The geographical indication of cachaça: An instrument for regional development and innovation. *Brazilian Journal of Development*, 6(6), 35137-35155. <https://doi.org/10.34117/bjdv6n6-160>
- Corbion, C., Smith-Ravin, J., Marcelin, O., & Bouajila, J. (2023). An overview of spirits made from sugarcane juice. *Molecules*, 28(19), 6810. <https://doi.org/10.3390/molecules28196810>
- Czerny, M., Christlbauer, M., Christlbauer, M., Fischer, A., Granvogel, M., Hammer, M., et al. (2008). Re-investigation on odour thresholds of key food aroma compounds and development of an aroma language based on odour qualities of defined aqueous odorant solutions. *European Food Research Technology*, 228, 265-273. <https://doi.org/10.1007/s00217-008-0931-x>
- Faria, J. B., Ferreira, V., Lopez, R., & Cacho, J. (2003). The sensory characteristic defect of cachaça distilled in absence of copper. *Alimentos e Nutrição*, 14(1), 1-7.
- Guimarães, B. P., Neves, L. E. P., Guimarães, M. G., & Ghesti, G. F. (2020). Evaluation of maturation congeners in beer aged with Brazilian woods. *Journal of Brewing and Distilling*, 9(1), 1-7. <https://doi.org/10.5897/JBD2019.0053>
- Janzantti, N. S. (2004). *Compostos voláteis e qualidade de sabor da cachaça* (Doctoral dissertation, Universidade Estadual de Campinas).
- Lima, C. M. G., Benoso, P., Pierozan, M. D., Santana, R. F., Hassemmer, G. S., Rocha, R. A., Dalla Nora, F. M., Verruck, S., Caetano, D., & Simal-Gandara, J. (2022). A state-of-the-art review of the chemical composition of sugarcane spirits and current advances in quality control. *Journal of Food Composition and Analysis*, 106, 104338. <https://doi.org/10.1016/j.jfca.2021.104338>
- Macfie, H. J., Bratchell, N., Greenhoff, K., & Vallis, L. V. (1989). Designs to balance the effect of order of presentation and first-order carry-over effects in hall tests. *Journal of Sensory Studies*, 4(2), 129-148. <https://doi.org/10.1111/j.1745-459X.1989.tb00463.x>
- Meilgaard, M. C. (1975). Flavor chemistry of beer: Part II: Flavor and threshold of 239 aroma volatiles. *MBAA Technical Quarterly*, 12(3), 151-168.
- Meilgaard, M. C., Civille, G. V., & Carr, B. T. (2006). *Sensory evaluation techniques* (4th ed.). CRC Press.
- Miranda, M. B., Martins, N. G. S., Belluco, A. E. S., Horii, J., & Alcarde, A. R. (2008). Chemical profile of aguardente – Brazilian sugar cane alcoholic drink – aged in oak casks. *Ciência e Tecnologia de Alimentos*, 28(Suppl.), 84-89. <https://doi.org/10.1590/S0101-20612008000500014>
- Moskowitz, H. R. (1983). *Product testing and sensory evaluation of foods: Marketing and R&D approaches*. Food & Nutrition Press.
- National Institute of Standards and Technology (NIST) (2013). *NIST Chemistry WebBook*. National Institute of Standards and Technology. Retrieved from <http://webbook.nist.gov/chemistry/>
- Nunes, C. A., & Pinheiro, A. C. M. (2012). *SensoMaker Version 1.9* [Software]. Universidade Federal de Lavras.
- Odello, L., Braceschi, G. P., Fortunato Seixas, F. R., da Silva, A. A., Galinaro, C. A., & Franco, D. W. (2009). Avaliação sensorial de cachaça. *Química Nova*, 32(7), 1839-1844. <https://doi.org/10.1590/S0100-40422009000700027>
- Oliveira, R. E. S., Cardoso, M. G., Santiago, W. D., Barbosa, R. B., Alvarenga, G. F., & Nelson, D. L. (2020). Physicochemical parameters and volatile composition of cachaça produced in the state of Paraíba, Brazil. *Research, Society and Development*, 9(7), e504974409. <https://doi.org/10.33448/rsd-v9i7.4409>
- Paolini, M., Tonidandel, L., & Larcher, R. (2022). Development, validation and application of a fast GC-FID method for the analysis of volatile compounds in spirit drinks and wine. *Food Control*, 136, 108873. <https://doi.org/10.1016/j.foodcont.2022.108873>
- Pineau, N., & Schlich, P. (2015). Temporal dominance of sensation (TDS) as a sensory profiling technique. In J. Delarue, J. B. Lawlor, & M. Rogeaux (Eds.), *Rapid Sensory Profiling Techniques and Related Methods* (pp. 67-82). Woodhead Publishing.
- Pineau, N., Schlich, P., Cordelle, S., Mathonnière, C., Issanchou, S., Imbert, A., Rogeaux, M., Etiévant, P., & Koster, E. (2009). Temporal dominance of sensations: Construction of the TDS curves and comparison with time-intensity. *Food Quality and Preference*, 20(6), 450-455. <https://doi.org/10.1016/j.foodqual.2009.04.005>
- Pino, J. A., Tolle, S., Gök, R., & Winterhalter, P. (2012). Characterisation of odour-active compounds in aged rum. *Food Chemistry*, 132(3), 1436-1441. <https://doi.org/10.1016/j.foodchem.2011.11.133>
- Qiao, L., Wang, J., Wang, R., Zhang, N., & Zheng, F. (2023). A review on flavor of Baijiu and other world-renowned distilled liquors. *Food Chemistry*, 20, 100870. <https://doi.org/10.1016/j.foodchem.2023.100870>
- Ratkovich, N., Esser, C., Machado, A. M. R., Mendes, B. A., & Cardoso, M. G. (2023). The spirit of cachaça production: An umbrella review of processes, flavour, contaminants and quality improvement. *Foods*, 12(17), 3325. <https://doi.org/10.3390/foods12173325>
- Santiago, W. D. (2016). *Perfil físico-químico, compostos voláteis, carbamato de etila e compostos fenólicos de cachaças armazenadas em tonéis recém-confeccionados de carvalho e madeiras nativas brasileiras* (Doctoral dissertation, Universidade Federal de Lavras).
- Santiago, W. D., Borges, C. N., Barbosa, R. B., Mendonça, H. A., Nelson, D. L., & Cardoso, M. G. (2020). Investigation of Brazilian cachaças regarding their standardization and quality. *Research, Society and Development*, 9(7), e387974117. <https://doi.org/10.33448/rsd-v9i7.4117>
- Santiago, W. D., Cardoso, M. G., Gomes, M. S., Rodrigues, L. M. A., Cardoso, R. R., & Brandão, R. M. (2014). Correlação entre extrato seco total, composição fenólica total e intensidade de cor de cachaças envelhecidas em tonéis de carvalho (*Quercus sp*) e amburana (*Amburana cearensis*) em um período de 12 meses. *Revista e-xacta*, 7(2), 9-15. <https://doi.org/10.18674/ex-acta.v7i2.1259>
- Serafim, F. A. T., Seixas, F. R. F., Silva, A. A., Galinaro, C. A., Nascimento, E. S. P., Buchviser, S. F., Odello, L., & Franco, D. W. (2013). Correlation between chemical composition and sensory properties of Brazilian sugarcane spirits (cachaças). *Journal of the Brazilian Chemical Society*, 24(6), 973-982. <https://doi.org/10.5935/0103-5053.20130125>
- Siebert, T. E., Smyth, H. E., Capone, D. L., Neuwöhoner, C., Pardon, K. H., Skouroumounis, G. K., et al. (2005). Stable isotope dilution analysis of wine fermentation products by HS-SPME-GCMS. *Analytical Bioanalytical Chemistry*, 381(4), 937-947. <https://doi.org/10.1007/s00216-004-2992-4>
- Silveira, A. L., & Barbeira, P. J. S. (2022). A fast and low-cost approach for the discrimination of commercial aged cachaças using synchronous fluorescence spectroscopy and multivariate classification. *Journal of the Science of Food and Agriculture*, 102(11), 4918-4926. <https://doi.org/10.1002/jsfa.11857>

- Simioni, S. C. C., Tovar, D. M., Rodrigues, J. F., Souza, V. R., Nunes, C. N., Vietoris, V., & Pinheiro, A. C. M. (2018). Temporal dominance of sensations and preferences of Brazilians and Slovaks: A cross-cultural study of cachaças stored with woods from the Amazon rainforest. *Journal of the Science of Food and Agriculture*, 98(11), 4058-4064. <https://doi.org/10.1002/jsfa.8922>
- Souza, M. D. C. A., Vásquez, P., Del Mastro, N. L., Acree, T. E., & Lavin, E. H. (2006). Characterization of cachaça and rum aroma. *Journal of Agriculture and Food Chemistry*, 54(2), 485-488. <https://doi.org/10.1021/jf0511190>
- Stone, H., & Sidel, J. (2004). *Sensory evaluation practices*. Elsevier Academic Press.
- Yokota, S. R. C. (2005). *Avaliação sensorial descritiva de cachaça envelhecida entre 18 e 24 meses por 4 painéis de julgadores* (Master's thesis, Universidade Federal de Viçosa).
- Wald, A. (1945). Sequential tests of statistical hypotheses. *The Annals of Mathematical Statistics*, 16(2), 117-186. <https://doi.org/10.1214/aoms/1177731118>
- Williams, A. A. (1974). Flavour research and the cider industry. *Journal of the Institute of Brewing*, 80(5), 455-470. <https://doi.org/10.1002/j.2050-0416.1974.tb06795.x>
- Zacaroni, L. M., Cardoso, M. G., Saczk, A. A., Santiago, W. D., Anjos, J. P., Masson, J., & Nelson, D. L. (2011). Caracterização e quantificação de contaminantes em aguardentes de cana. *Química Nova*, 34(2), 320-324. <https://doi.org/10.1590/S0100-40422011000200016>