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VANESSA PEDRO DA SILVA

PERFIL VOLATILÔMICO DAS CACHAÇAS PARAIBANAS

JOÃO PESSOA - PB

2025

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Tese apresentada ao Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos, Centro de Tecnologia, Universidade Federal da Paraíba, em cumprimento aos requisitos para obtenção do título de Doutor em Ciência e Tecnologia de Alimentos.

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Aos três dias do mês de julho do ano de dois mil e vinte e cinco, na sala do PPGCTA do Bloco Multimídia, às 13h, reuniu-se a Banca Examinadora composta pela Profa. Dra. Taliana Kênia Alves Bezerra, orientadora do trabalho e presidente da Banca, Prof. Dr. Normando Mendes Ribeiro Filho (Coorientador), Profa. Dra. Marta Suely Madruga (Membro Interno/UFPB), Prof. Dr. Marcelo Barbosa Muniz (Membro Interno/UFPB), Prof. Dr. José Germano Veras Neto (Membro Externo à Instituição/UEPB) e Prof. Dr. Roberto Germano Costa (Membro Externo/UFPB). A reunião teve por objetivo julgar o trabalho da estudante **Vanessa Pedro da Silva**, matrícula nº 20211016623, sob o Título: "PERFIL VOLATILÔMICO DAS CACHAÇAS PARAIBANAS." Os trabalhos foram abertos pela Profa. Dra. Taliana Kênia Alves Bezerra. A seguir foi dada a palavra à estudante para apresentação do trabalho. Cada Examinador(a) arguiu a doutoranda, com tempos iguais de arguição e resposta. Terminadas as arguições, procedeu-se o julgamento do trabalho, concluindo a Banca Examinadora por sua Aprovação. Atendidas as exigências da Resolução nº 15/2019/CONSEPE, que regulamenta o Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos, e nada mais havendo a tratar, foi lavrada a presente Ata, que vai assinada pelos membros da Banca Examinadora e pela doutoranda.

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Ao meu amado filho Julius,

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RESUMO

A cachaça é uma bebida destilada tradicional brasileira, obtida a partir da fermentação e destilação do caldo de cana-de-açúcar, e sua composição química pode estar ligada à sua origem geográfica e às práticas de produção. O objetivo deste estudo foi avaliar a composição química e volátil de cachaças não envelhecidas (tradicionais) e cachaças armazenadas em barris de Amburana (*Amburana cearensis*), produzidas em três mesorregiões do estado da Paraíba, Brasil (Agreste, Sertão Paraibano e Mata Paraibana), a fim de fornecer informações relevantes para a identificação geográfica e o controle de qualidade. Foram analisadas 26 amostras de cachaça não envelhecida (tradicional) e 21 amostras de cachaça armazenadas em barris de Amburana por períodos de quatro meses a quatro anos. Foram determinados parâmetros físico-químicos teor alcoólico, acidez volátil, álcoois superiores, aldeídos, ésteres, furfural, metanol, carbamato de etila e teor de cobre. O perfil volátil por CG-MS e análise espectrofométrica de intensidade de cor, fenólicos totais e antioxidantes. A análise estatística envolveu ANOVA, teste de *Tukey*, mapas de calor e PCA. As cachaças não envelhecidas atenderam em praticamente sua totalidade aos limites estabelecidos pelos padrões de identidade e qualidade; entretanto, nas cachaças armazenadas em barris de Amburana, foram detectados acidez volátil excessiva e altos níveis de carbamato de etila e cobre, sugerindo deficiências de higiene, práticas de destilação ou manutenção de equipamentos. As análises cromatográficas identificaram compostos voláteis, incluindo álcoois, ésteres, cetonas, aldeídos e terpenos, permitindo a diferenciação entre as cachaças de acordo com sua origem geográfica e condições de envelhecimento. As cachaças não envelhecidas da região da Mata Paraibana apresentaram maiores concentrações de álcoois e ésteres em comparação com as do Agreste, enfatizando a influência de fatores edafoclimáticos. Nas cachaças armazenadas em barris de Amburana, compostos como 2-metil-1-butanol, 3-metil-1-butanol, álcool feniletílico e vários ésteres etílicos foram predominantes, contribuindo para notas sensoriais doces, florais, frutadas e amadeiradas; e observado uma ampla variabilidade no conteúdo fenólico total (0,73 a 61,88 mg GAE/L), que se correlacionou positivamente com a intensidade da cor e a atividade antioxidante, demonstrando ainda mais o impacto das condições de armazenamento e reutilização do barril. Em conclusão, o estudo revelou uma grande variabilidade na qualidade química das cachaças paraibanas, devido às diferenças nas práticas de produção e armazenamento. Além disso, foi possível uma separação das cachaças não envelhecidas em mesorregiões influenciadas por fatores edafoclimáticos. O processo de envelhecimento em madeira de Amburana contribuiu positivamente para a

complexidade da bebida, enriquecendo-a com aromas desejáveis que melhoram seu perfil sensorial, no entanto, destaca-se a necessidade de protocolos de higiene, para garantir a consistência da qualidade, padronização das condições de envelhecimento, como reutilização do barril e tempo de armazenamento.

PALAVRAS-CHAVE: compostos voláteis, indicação geográfica, fatores edafoclimáticos, práticas de produção, envelhecimento.

ABSTRACT

Cachaça is a traditional Brazilian distilled beverage, obtained from the fermentation and distillation of sugar cane juice, and its chemical composition may be closely linked to its geographical origin and production practices. The aim of this study was to evaluate the chemical and volatile composition of non-aged (traditional) cachaças and cachaças stored in Amburana (*Amburana cearensis*) barrels, produced in three mesoregions of the state of Paraíba, Brazil (Agreste, Sertão Paraibano and Mata Paraibana), in order to provide relevant information for geographical identification and quality control. 26 samples of unaged cachaça (traditional) and 21 samples of cachaça stored in Amburana barrels for periods of four months to four years were analyzed. Physico-chemical parameters were determined: alcohol content, volatile acidity, higher alcohols, aldehydes, esters, furfural, methanol, ethyl carbamate and copper content. The volatile profile by GC-MS and spectrophotometric analysis of color intensity, total phenolics and antioxidants. Statistical analysis involved ANOVA, Tukey's test, heat maps and PCA. The unaged cachaças practically all met the limits set by the identity and quality standards; however, in the cachaças stored in Amburana barrels, excessive volatile acidity and high levels of ethyl carbamate and copper were detected, suggesting deficiencies in hygiene, distillation practices or equipment maintenance. The chromatographic analyses identified volatile compounds, including alcohols, esters, ketones, aldehydes and terpenes, allowing the cachaças to be differentiated according to their geographical origin and ageing conditions. The unaged cachaças from the Mata Paraibana region had higher concentrations of alcohols and esters compared to those from the Agreste region, emphasizing the influence of edaphoclimatic factors. In cachaças stored in Amburana barrels, compounds such as 2-methyl-1-butanol, 3-methyl-1-butanol, phenylethyl alcohol and various ethyl esters were predominant, contributing to sweet, floral, fruity and woody sensory notes; and a wide variability in total phenolic content was observed (0.73 to 61.88 mg GAE/L), which correlated positively with color intensity and antioxidant activity, further demonstrating the impact of storage conditions and barrel reuse. In conclusion, the study revealed great variability in the chemical quality of cachaças from Paraíba, due to differences in production and storage practices. In addition, it was possible to separate the unaged cachaças into mesoregions influenced by edaphoclimatic factors. The process of aging in Amburana wood has contributed positively to the complexity of the drink, enriching it with desirable aromas that improve its sensory profile. However, there is a need for

hygiene protocols to ensure consistent quality, standardization of aging conditions, such as barrel reuse and storage time.

KEY-WORDS: volatile compounds, geographical indication, edaphoclimatic factors, production practices, ageing.

SUMÁRIO

1. INTRODUÇÃO.....	11
2. OBJETIVOS.....	13
3. REVISÃO DE LITERATURA.....	14
3.1 ARTIGO DE REVISÃO - CACHAÇA PRODUCTION: FROM SUGAR CANE TO SPIRIT.....	15
4. DELINEAMENTO EXPERIMENTAL.....	33
5. RESULTADOS.....	35
5.1 ARTIGO I – CHEMICAL COMPOSITION AND SENSORY DIVERSITY OF COLOURLESS CACHAÇAS FROM PARAÍBA: INFLUENCE OF GEOGRAPHIC ORIGIN AND PRODUCTION PRACTICES.....	36
5.2 ARTIGO II - INFLUENCE OF AMBURANA CEARENSIS BARRELS ON THE SENSORY PROFILE AND COMPLEXITY OF CACHAÇA DURING MATURATION.....	61
6. CONSIDERAÇÕES FINAIS.....	104
7. REFERÊNCIAS.....	105
8. PRINCIPAIS CONTRIBUIÇÕES TECNICO-CIENTÍFICAS.....	107
APÊNDICES.....	111
ANEXOS.....	125

1 INTRODUÇÃO

A cachaça é uma bebida alcoólica típica e exclusivamente produzida no Brasil, obtida pela destilação do mosto fermentado do caldo de cana-de-açúcar, com teor alcoólico entre 38% e 48% em volume a 20°C (BRASIL, 2022). Além de seu valor cultural e histórico, a cachaça possui significativa importância econômica e industrial, sendo derivada de uma das culturas agrícolas mais exploradas no país: a cana-de-açúcar (RIBEIRO-FILHO, 2020).

A produção de cachaça pode ser realizada tanto em pequenas quanto em grandes unidades industriais, utilizando alambiques de cobre ou colunas de inox (BORTOLETTO, 2023), armazenada em tonéis de inox ou submetida à maturação em barris de madeira o que contribui para alterações significativas em sua composição química e sensorial (ALCARDE, et al., 2014; SANTIAGO et al., 2017).

Diferentes madeiras são utilizadas para o envelhecimento/armazenamento de cachaças comerciais, como o carvalho (*Quercus rubra*), a amburana (*Amburana cearensis*), a castanheira (*Bertholletia excelsa*) e o bálsamo (*Sedum dendroideum*), que conferem características químicas e sensoriais distintas à cachaça (BORTOLETTO et al., 2013; SOUZA et al., 2025). A Amburana (*Amburana cearensis*) tem sido amplamente utilizada no envelhecimento de cachaças, sendo uma madeira nativa brasileira (BORTOLETTO et al., 2016). Estão presentes ésteres de ácidos graxos e alguns compostos fenólicos em seu perfil volátil, com predominância de ácido vanílico, cumarina e 4-metilumbelliferona (SANTIAGO et al., 2016), que são importantes para o sabor e o aroma da bebida, conferindo marcadores aromáticos de baunilha, canela e outras especiarias, além de notas florais e frutadas (SOUZA et al., 2025).

A qualidade da cachaça está fortemente relacionada aos compostos voláteis formados durante a produção e envelhecimento, os quais conferem características sensoriais únicas, como aromas florais, frutados e amadeirados (SANTIAGO et al., 2016; SOUZA et al., 2025). Esses compostos podem ser influenciados por diversos fatores, como o tipo de madeira utilizada, o tempo de armazenamento, o volume e a capacidade dos barris (BORTOLETTO et al., 2016; ALCARDE et al., 2014).

O envelhecimento da cachaça em barris de madeira confere qualidade química e aromática a bebida; no entanto a composição volátil e os parâmetros de qualidade, como teor de álcoois superiores, ésteres, acidez volátil e carbamato de etila, variam conforme as práticas de produção adotadas bem como durante o envelhecimento. Estudos constataram alterações no coeficiente de congêneres, representado pela soma de aldeídos totais (em acetaldeído), ésteres

totais (em acetato de etila), álcoois superiores (1-propanol, iso-butanol e isoamílico), furfural e hidroximetilfurfural e acidez volátil (em ácido acético) e no teor de carbamatos (ALCARDE et al., 2014; BORTOLETTO et al., 2015; SOUZA et al., 2025). Além dos aspectos químicos e sensoriais, a origem geográfica exerce papel fundamental na caracterização e valorização da cachaça, influenciada por fatores edafoclimáticos e de produção, que contribuem para a compreensão do *terroir* dessa bebida (SERAFIM et al., 2016).

Nesse contexto, a presente tese teve como objetivo avaliar a conformidade com os parâmetros de qualidade e o perfil de compostos voláteis das cachaças paraibanas não envelhecidas (tradicionais) e as armazenadas em barris de Amburana (*Amburana cearensis*) produzidas em diferentes mesorregiões da Paraíba: Mata Paraibana, Agreste e Sertão Paraibano, levando em consideração os fatores edafoclimáticos e diferentes condições de armazenamento nos barris de Amburana. O estudo visou identificar marcadores químicos associados à origem geográfica, contribuindo para a compreensão do *terroir* das cachaças paraibanas não envelhecidas (tradicionais) e fornecer subsídios técnicos e científicos para a padronização da qualidade das cachaças armazenadas em barris de Amburana, contribuindo para a valorização das cachaças produzidas no estado da Paraíba.

2 OBJETIVOS

2.1 OBJETIVO GERAL

Gerar informações científicas que possam contribuir para a caracterização da origem geográfica das cachaças paraibanas, não envelhecidas (tradicionais) e armazenadas em barris de Amburana (*Amburana cearenses*), através da identificação de compostos voláteis e não-voláteis, contribuindo para a padronização das normas que regulam a produção de cachaças.

2.2 OBJETIVOS ESPECÍFICOS

- Realizar o mapeamento e coleta nos estabelecimentos produtores de cachaças da Paraíba;
- Avaliar a qualidade química das cachaças paraibanas não envelhecidas (tradicionais) e armazenadas em barris de Amburana, de acordo com os padrões de identidade e qualidade da PORTARIA MAPA Nº 539/2022;
- Avaliar e identificar os compostos voláteis aromáticos importantes na caracterização das cachaças, que possam ser marcadores de origem geográfica, das cachaças paraibanas não envelhecidas (tradicionais);
- Avaliar e identificar os compostos voláteis e não-voláteis que caracterizam o envelhecimento em madeira das cachaças paraibanas armazenadas em barris de Amburana.

3 REVISÃO DE LITERATURA

Revisão de literatura apresentada em formato de artigo, de acordo com a Norma Complementar nº 01/2024 do PPGCTA.

3.1 ARTIGO DE REVISÃO - CACHAÇA PRODUCTION: FROM SUGAR CANE TO SPIRIT.

O artigo foi publicado no periódico *Journal of the Institute of Brewing*, em 2023, sob o título *Cachaça Production: from sugar cane to spirit*. <https://doi.org/10.58430/jib.v129i4.40>

REVIEW ARTICLE

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Cachaça Production: from sugar cane to spirit

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Abstract

Why was the work done: Cachaça, the oldest distilled beverage in the Americas, has great historical and cultural significance. A review of cachaça production is important to preserve tradition, standardise industry processes, promote innovation and quality. This review offers a comprehensive overview of current knowledge and advancements in cachaça production, covering regulation, process control, product quality and future developments.

What are the main findings: The production of Cachaça works within well defined regulations, with its production encompassing both field and industrial practice. The review focuses on sugar cane cultivation, fermentation, distillation, and ageing in wooden barrels. It underscores the significance of regional factors such as climate, soil, and sugar cane variety in shaping the sensory profile of cachaça. While a range of sugar cane cultivars have been developed and grown in different environments, further research on the adaptation of sugar cane crop is necessary. The fermentation of cachaça is spontaneous utilising the microbiota in the sugar cane juice. However, the use of wild sugar cane yeasts, complemented with commercial strains is increasingly used by producers. With regard to distillation, production of the spirit in copper still pots and ageing in tropical wood barrels remain prevalent in the industry.

Why is the work important: This review contributes to ongoing efforts to enhance the quality of cachaça. Whilst the importance of traditional production methods is recognised, this review embraces advancements in technology coupled with insights to future perspectives.

Keywords:

sugar cane, cachaça, production, tropical wood, barrel, ageing, quality

Cachaça: from sugar cane to spirit

Cachaça is a Brazilian spirit produced from sugar cane juice which is fermented and distilled (Bortoletto 2023). Cachaça is only produced in Brazil and has an alcohol content of 38-48% (v/v) (Brazil 2022a). The production of cachaça includes both field and industrial practice. Field practices include planting of sugar cane, harvesting and transportation, with industrial practice involving reception, extraction of juice, fermentation, distillation, standardisation, and ageing. Overall, the primary processes in Cachaça production are fermentation and distillation.

During fermentation, yeasts transform the sugars in sugar cane juice into wine with the formation of ethanol, carbon dioxide and other metabolites. The composition of sugar cane juice reflects the cultivars and adaptation to the environment. The juice contains around 10^3 yeast cells/mL that play a role in fermentation. The environmental yeast are diverse and include *Saccharomyces*, *Schizosaccharomyces*, *Pichia*, *Debaryomyces*, *Kloeckera*, *Zygosaccharomyces* and *Candida* (Rosa et al. 2009). Cachaça fermentation is performed over several fermentation cycles using open, batch fermenters. Although the primary yeast - *Saccharomyces cerevisiae* - is prevalent during fermentation batch fermentations are easily contaminated by environmental microorganisms. Therefore, producers acid wash the yeast inoculum to limit bacterial contamination. The distillation process uses a column or copper still pots (Lima et al. 2022; Bortoletto 2023) and the distilled product contains alcohols, aldehydes, acids, ketones, and esters (Alcarde et al. 2012). However, freshly distilled cachaça exhibits undesirable bitterness and harshness, which are reduced by maturation through ageing (Alcarde et al. 2014).

Cachaça is aged in stainless steel or wooden barrels. The former do not contribute any ageing characteristics to cachaça, whereas wooden barrels promote chemical changes in cachaça including flavour and colour (Alcarde et al. 2014; Bortoletto 2023). Although, oak barrels predominate for ageing of cachaça, other tropical woods have been used including umburana, wild peanut, jequitibá, ararua, jequitibá rosa, cherry tree, purple ipê and chestnut tree (Bortoletto et al. 2021). The use of tropical woods must maintain quality and

authenticity standards of cachaça, although each wood contributes different physiochemical and sensorial characteristics to cachaça (Silva et al. 2009; Bortoletto et al. 2021).

To ensure the yield, productivity and quality of sugar cane cachaça (and brandy), production is regulated by Ordinance Nº 539, which defines the physiochemical, sensorial characteristics and quality standards (Brazil 2022). Over time, the cachaça sector has improved the technologies for sugar cane production (handling, varieties, harvesting and transport), and cachaça production (extraction, fermentation, distillation and ageing). However, the production processes requires improvement to enhance process control, yield, productivity, and product quality. This review considers cachaça production and considers its technical improvement from sugar cane to a high quality spirit with consideration of contradictions, critical comments, and perspectives.

Legislation

During the 19th century, coffee growing had a negative effect on the production and market for cachaça (Rosa et al. 2009). In 1992, the Brazilian government created 'Pró-Cachaça', an incentive program to encourage cachaça producers to return and invest in technologies enabling large scale production (Lima et al. 2022). In 1997, legislation was put in place defining sugar cane spirits such as 'cachaça', 'caninha', or 'aguardente de cana' (Brazil 1997). In 2002, decree 4072 reserved the term 'cachaça' for sugar cane spirits made in Brazilian territory (Brazil, 2002). In 2005, the Ministry of Agriculture, Livestock and Supply approved the technical regulation that defines the quality standards to produce cachaça (Brazil 2005) and in 2013 cachaça was recognised as a Brazilian spirit (Medeiros et al. 2017).

The technical regulation (ordinance 13/2005) boosted cachaça production. However, in 2022, a new regulation was published that establishes the identity and quality standards for sugar cane spirit including 'cachaça' and 'aguardente de cana' (Brazil 2022b). This focussed on the quality parameters that assure the identity and quality of cachaça. This was an important step in boosting the production

of cachaça. Currently cachaça is produced in all Brazilian states and is the second most consumed alcoholic beverage in the country. The sugar cane sector contributes to the preservation of the diverse genetic resources, cultural heritage, historical legacy in Brazil. Cachaça helps shape the socio-economic landscape of the country, is a versatile ingredient in culinary practice and an ingredient in the preparation of the national drink, 'caipirinha'.

Styles

Cachaça is an alcoholic beverage with an ABV (alcohol by volume) of 38 to 48%. Cachaça is classified into four categories: 'cachaça', 'cachaça adoçada' (sweetened cachaça) and 'aged'. Aged cachaça is divided into 'cachaça descansada' (short aged) and 'aged cachaça' (premium cachaça and extra premium cachaça) (Brazil 2022b).

Sugar cane and its cultivars

'Cachaça is produced from the wort of sugar cane juice with proper sensorial characteristics' (Brazil 2022a). Sugar cane is a tropical crop that grows well in hot climates at temperatures between 18 and 35°C (Rossato et al. 2013). Sugar cane cultivars result from crossbreeding of species or other cultivars and adapt to different soils, climate and environment. As a result, these factors influence the chemical composition and maturation of the sugar cane plants (Rosa et al. 2009; Lima et al. 2022). Sugar cane cultivars contain 75 to 89% water and 11-25% soluble solids (including carbohydrates, amino acids, minerals, vitamins, and lipids). Sugar cane juice contains glucose, fructose, and sucrose as the primary sugars (Rosa et al. 2009; Martini et al. 2010). Poor adaptation of sugar cane cultivars results in a low concentration of soluble solids and can negatively affect the yield and quality of cachaça (Vilela et al. 2021).

Sugar cane used for cachaça production is typically manually cropped and harvested, and is distinct from sugar cane destined for biofuel or sugar where mechanical harvesting is used (Rosa et al. 2009; Rossato et al. 2013). Large scale sugar cane plantations have embraced the adoption of new and improved sugar cane cultivars. In contrast, smaller producers have maintained the cultivation of older genotypes characterised by low yields and productivity. This is attributed to the limited

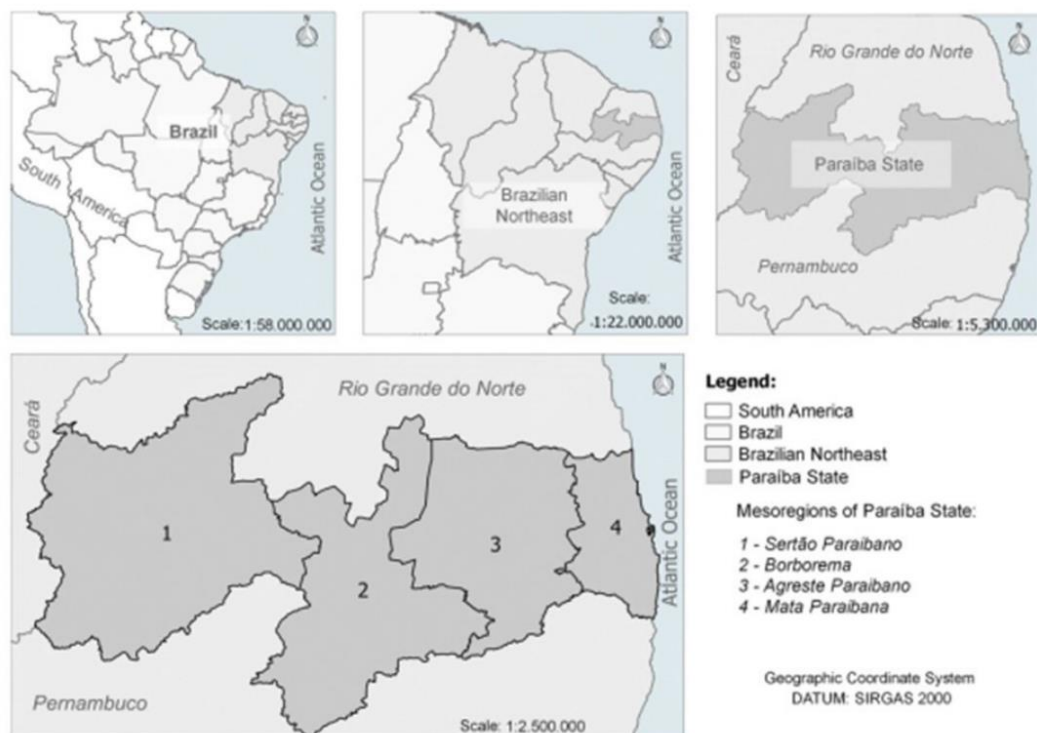
adaptability with heightened susceptibility to diseases, insects, and pests exhibited by older genotypes (Ravaneli et al. 2006; Rosa et al. 2009). Recently, small scale sugar cane producers have undergone a transformative shift thanks to the emergence and advancement of novel sugar cane cultivars. These cultivars have enhanced adaptability to varying weather conditions, efficient straw removal, resistance against lodging, low fibre content, resilience to pests, insects, diseases and exhibit enhanced productivity (Ravaneli et al. 2006). This has significantly influenced small scale producers and their practices in cultivating sugar cane, such that they use new sugar cane cultivars to guarantee the quality and yield of cachaça (Martini et al. 2010).

In Brazil, three research institutes - Planalsucar/Ridesa (RB), Copersucar Sugar cane Technology Centre (CTC) and Instituto Agronômico de Campinas (IAC) - have breeding programmes for the development of new sugar cane cultivars. Of these, RB cultivars cover 68% of the sugar cane growing area in Brazil, with the RB92579 cultivar the most cropped sugar cane in the Northeast region due to easy adaptation and high productivity (Martini et al. 2010; Alcarde et al. 2012). In the Paraíba state, the RB867515 cultivar is used by small and large producers due to its adaptation to growth conditions, yield and productivity. Further, this cultivar shows excellent microbiological and physical-chemical characteristics (Martini et al. 2010; Alcarde et al. 2014).

With the exception of Amapá and Roraima, sugar cane is produced in all Brazilian states. Paraíba, is divided into four mesoregions, with sugar cane cropping in two mesoregions, Agreste Paraibano (3), and Mata Paraibana (4) (Figure 1). The Mata Paraibana mesoregion is characterised by hot and humid tropical climate that contains a large area for sugar cane cultivation due to its weather and relief. However, most of the sugar cane production is destined for bioethanol and sugar production (Medeiros-Silva et al. 2019; Vilela et al. 2021). The Agreste mesoregion is characterised by medium to low temperatures with irregular rainfall and produces most of the cachaça in Paraíba (Medeiros-Silva et al. 2019).

Figure 1.

Paraíba state in Brazil with mesoregion divisions (Adapted from Medeiros-Silva et al. 2019).



Serafim et al (2016) sampled cachaça from five Brazilian states, produced by spontaneous fermentation and distilled in copper stills pot. The authors reported that there were chemical differences in cachaças which were not aged in wooden barrels. Vilela et al. (2021) examined thirty-eight cachaça samples from Paraíba state and identified differences in their chemical profile. The differences of these cachaças were associated with the characteristics of each region, including weather, soil, environment, sugar cane cultivars, and the type of wild yeast native to each region.

Juice extraction, wort composition, and microbiota

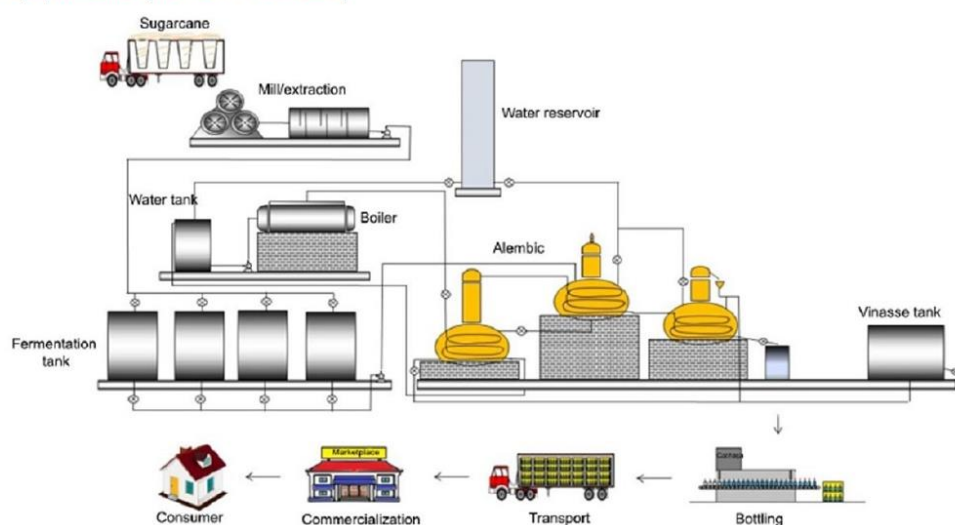
Sugar cane harvesting should be during the optimal harvesting period or 'maturation index' (MI), corresponding to the highest sucrose concentration in the culms (Rosa et al. 2009). The sugar cane cultivars can reach maturation at different times

and are classified as early (< 12 months), average ($\approx$ 12 months) or late (> 12 months). These three periods are driven by the type of sugar cane cultivar and its adaptation to the environment (Martini et al. 2010). Not all producers use the maturation index for harvesting but identify a soluble solids concentration >18°Brix as suitable (Rosa et al. 2009). Sugar cane juice is ground (mechanically crushed) to extract 65-75% (at a small-scale), or up to three times for a 90% extract (Rosa et al. 2009; Martini et al. 2010). Increasingly grinding is being replaced by milling and then grinding (Figure 2).

Yeasts are inoculated into sugar cane juice (or 'must') (Martini et al. 2010; Bortoletto 2023) that supports microbial growth and fermentation through sugars (glucose, fructose, and sucrose), nitrogenous material (amino acids, peptides, proteins, nitrogenous ions, nucleic acids), vitamins, organic acids, lipids, inorganic elements (potassium,

Figure 2.

Cachaça production (from Silva et al. 2020)



phosphorus, magnesium, zinc, copper, iron, manganese) (Briggs et al. 2004). Sugar cane juice contains about 10^3 yeast/mL, which multiply during fermentation. Although *Saccharomyces cerevisiae* is prevalent, other yeasts have been identified including *Schizosaccharomyces*, *Pichia*, *Debaryomyces*, *Kloeckera*, *Zygosaccharomyces* and *Candida* (Rosa et al. 2009; Monjito et al. 2014). With the correct yeast handling practice, the cell number increases to 30×10^6 /mL in 48 hours (Monjito et al. 2014).

Sugar cane juice also inevitably contains bacteria, contaminants which convert sugar and ethanol to lactic and acetic acid (Borges et al. 2014). These acids are thought to be associated with the formation of volatile compounds although there is little evidence for this (Rosa et al. 2009). In contrast, *Saccharomyces* yeast produce acetic, lactic, citric and succinic acids (Briggs et al. 2004). Researchers and producers have been considering options for inactivating wort microbiota as these can impact on the quality of cachaça. This would improve fermentation control and the quality of cachaça.

Fermentation

In the production of cachaça (Figure 2), fermentation is performed by yeasts which convert sugars into

ethanol, carbon dioxide and secondary metabolites. *Saccharomyces cerevisiae* is widely used for cachaça production due to its resistance to toxicity, high fermentative capacity, resistance to stress, and the formation of desirable compounds (Briggs et al. 2004; Martini et al. 2010; Duarte et al. 2013; Ribeiro-Filho et al. 2021). Cachaça yeasts are indigenous to the sugar cane plant, and reflect the cultivars, weather, soil, and environment (Vilela et al. 2021). Typically, cachaça fermentation is spontaneous using the yeast from sugar cane (Vicente et al. 2006; Martini et al. 2010; Portugal et al. 2017). However, producers are increasingly using commercial yeasts to inoculate the juice (Duarte et al. 2013; Monjito et al. 2014). Although commercial yeasts enable faster fermentation, yeasts from different environments are important in the quality and diversity of cachaça.

The sugar cane plant is rich in yeast from the eleventh internode to the top with reducing sugars and elevated acidity (Martini et al. 2010; Borges et al. 2014). Strains of *Saccharomyces cerevisiae* have been isolated from sugar cane with favourable characteristics in adaptation/survival to environmental factors including pH, temperature, oxygen, osmotic stress, nutritional limitation (carbon, amino acids and inorganic elements) and ethanol yield (Alcarde et al. 2014). Commercial yeasts are an attractive alternative to better

control fermentation (Rosa et al. 2009; Alcarde et al. 2014; Paredes et al. 2018). Despite this, some producers prefer to continue use the yeasts from sugar cane juice microbiota.

Yeast strains for cachaça production have been isolated according to fermentation rate, stress tolerance, sugar consumption, flocculation, no hydrogen sulphide, low acetic acid, ethanol tolerance, high ethanol formation and desired aroma compounds (Vicente et al. 2006; Nova et al. 2009; Paredes et al. 2018). Further, a mix of yeasts (*Saccharomyces cerevisiae* and non-*Saccharomyces*) were used to assess fermentation performance, flavour and aroma. It was observed that *Pichia caribbica* and *Saccharomyces cerevisiae* improved fermentation, increasing ethanol content and the aroma profile (Duarte et al. 2013; Borges et al. 2014). Similarly, *Saccharomyces cerevisiae* and *Meyerozyma caribbica* were reported to increase the content of esters and higher alcohols (Amorim et al. 2016).

Secondary metabolites produced during fermentation

Yeast metabolites are either non-volatile or volatile. Sugars (including mono-, di-, tri-, and polysaccharides), inorganic salts, nucleotides, amino acids, proteins, polyphenols, small peptides contribute to the flavour of the beverage. Whereas volatile compounds (esters and higher alcohols) are responsible for aroma. Although the raw material can influence flavour formation, yeast metabolism generates a multitude of compounds, which, together with the partition coefficient of compounds during distillation, influence the aroma profile of cachaça and. Yeast compounds can be grouped into six groups including organic acids, higher alcohols, carbonyl compounds, sulphur compounds, phenolic compounds, and volatile esters (Saerens et al. 2010). Higher alcohols and esters are important aroma compounds. Higher alcohols are generated from amino acid metabolism and esters from higher alcohols and acetyl-CoA (Briggs et al. 2004).

Distillation

During distillation, compounds are separated according to their boiling point (Alcarde et al. 2012). Distillation can be performed as a continuous, semi-

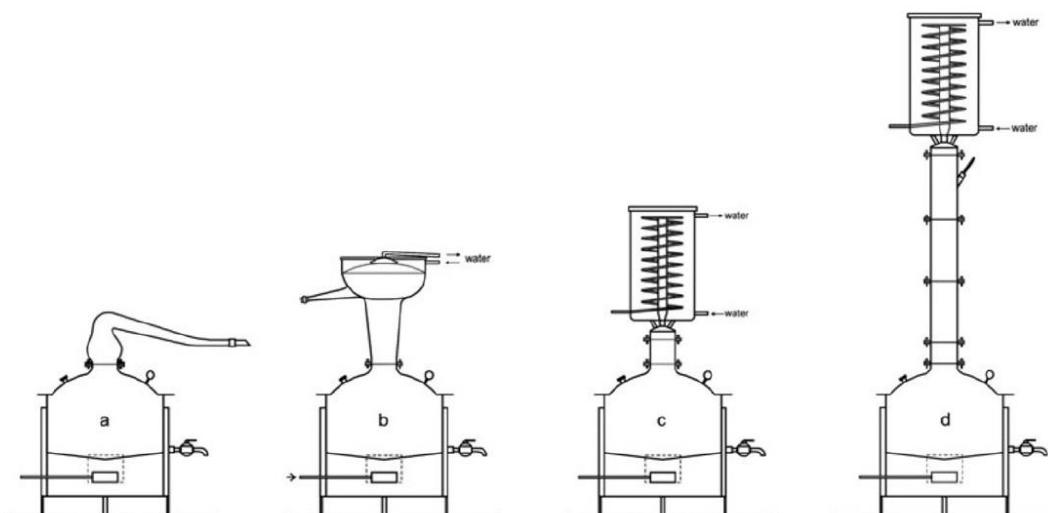
continuous or discontinuous batch process (Rosa et al. 2009; Alcarde et al. 2012;). The continuous process uses fractional distillation with stainless steel columns that are used for large scale production of cachaça or for bioethanol (Rosa et al. 2009; Bortoletto 2023). Discontinuous distillation or 'simple' or 'double distillation' with a copper still pot (alembic) are used in the small scale production of cachaça (Figure 3). A copper still can result in an increase in the concentration of copper in cachaça, an issue which is managed by effective cleaning. Cachaça distillation is controlled by alcohol content and is separated into three fractions - 'head', 'heart' and 'tail' (Alcarde et al. 2012; Serafim et al. 2013; Portugal et al. 2017).

The heads fraction contains desirable volatile compounds, a high ethanol content but more undesirable compounds (including methanol) (Alcarde et al. 2009; Nova et al. 2009; Serafim et al. 2013; Rota et al. 2013). The hearts fraction is used for cachaça, and represents 80-85% of the distillate, with ethanol and other volatile compounds reflecting the requirements of legislation (Alcarde et al. 2009; Serafim et al. 2013; Borges et al. 2014; Bortoletto et al. 2021). The tails fraction contains a low level of ethanol and desirable compounds, with a high levels of acetic acid, furfural, and hydroxymethylfurfural (Nova et al. 2009; Rota et al. 2013; Granato et al. 2014; Bortoletto and Alcarde 2015; Portugal et al. 2017). Further, during distillation or double distillation, the undesirable compound ethyl carbamate (from the reaction of ethanol and cyanide) is retained in the tails fraction (Alcarde et al. 2012). It should be noted that continuous distillation in stainless steel columns does **not** result in separation into the three fractions (Nova et al. 2009; Alcarde et al. 2012).

Post processing, cachaças from copper still pots or stainless steel columns are described on tasting as aggressive, alcoholic, hard, bitter with negative sensory characteristics (Rosa et al. 2009; Alcarde et al. 2014). The use of copper still pots minimise some of these characteristics as copper removes sulphur compounds, generated during fermentation from sulphur containing amino acids (Alcarde et al. 2014; Silvello et al. 2021). During the first three to six months of maturation in stainless steel or wooden barrels, some of the highly volatile compounds are lost, which improves the sensory characteristics (Alcarde et al. 2014; Silvello et al. 2021).

Figure 3.

Copper pot stills used to produce Cachaça: (a) hot head; (b) head cooler; (c) dephlegmator; and (d) rectifier (adapted from Alcarde et al. 2012).



Discontinuous double distillation can also be used to reduce the hard and bitter characteristics of unaged cachaça (Alcarde et al. 2012). Double distillation uses a second distillation, using (1) hearts fraction, (2) a mix of head and tails fraction) or (3) a mix of 50% (heads and tails) + 50% of freshly fermented wort. Double distillation reduces acidity, aldehydes, esters, methanol, higher alcohols, copper and ethyl carbamate and is good for ageing (Alcarde et al. 2012). Indeed, double distillation generates a more standardised beverage, improving its sensorial quality and consistency (Alcarde et al. 2012; Rota et al. 2013).

Increasing the number of distillations results in a more neutral spirit with fewer congeners (acetic acid, aldehyde, ethyl acetate, propyl alcohol and furfural) and contaminants (methanol, butan-2-ol, copper and ethyl carbamate). Moving from double distillation to five rounds of distillation, creates a soft and flavourless product, which can be developed by ageing in wooden barrels. However, for traditional unaged cachaça, a simple or double distillation can generate a product with the desirable and complex flavour profile of cachaça. This process takes five hours and further research is required establish the optimal duration and configuration of copper still pots for aroma formation during distillation.

Ageing

Whilst ageing is a standard process step in the production of cachaça, it is not mandatory (Rosa et al. 2009) but significantly improves flavours (Rosa et al. 2009; Caetano et al. 2022). However, as noted above, freshly distilled cachaças can be hard and bitter. Accordingly, a short storage period (three to 12 months) in steel or wooden barrels is recommended to improve the sensory characteristics of cachaça (Alcarde et al. 2014; Silvello et al. 2021). Further, the poor sensory characteristic of fresh or young cachaças can be mollified by the addition of sucrose (6 g/L to young or 30 g/L to fresh). In the latter case, the addition of sucrose requires labelling as 'sweetened' cachaça (Brazil 2022b).

Cachaça is classified as 'cachaça', 'sweetened cachaça', 'aged cachaça', 'premium cachaça', and 'extra premium cachaça' (Brazil 2022b). 'Aged' cachaça must contain 50% of cachaça which has been aged for at least 12 months in wooden 700 L barrels, whereas 'premium cachaça' contains 100% aged spirit. The 'extra premium' cachaça is 100% aged spirit but aged for three years or more.

Like whiskey, cachaça is aged using oak barrels (Rosa et al. 2009; Castro et al. 2020). There are several oak

species including peduncular oak (*Quercus robur* L, *Quercus pedunculata* Ehrh), sessile oak (*Quercus petraea* L, *Quercus sessiliflora* Sm), American white oak (*Quercus alba* L) and North American red (*Quercus rubra*) (Chatonnet and Dubourdieu 1998). Ageing in oak barrels contributes attractive compounds including cumurins, scopoletin, gallic acid, ellagic acid, and vanillin (Chatonnet and Dubourdieu 1998; Bortoletto et al. 2016; Castro et al. 2020). However, as oak is not a native Brazilian wood, its use has become expensive through importation costs (Silva et al. 2009).

As a result other Brazilian tropical wood species have been used for cachaça ageing including castanheira-do-Pará (Pará or Brazil nut tree), umburana/ amburana (*Amburana cearenses*), balsamo (*Myroxylon balsamum*), Jatoba (*Hymenaea courbaril*), wild peanut, araruva, jequitibá rosa, peroba rosa (*Aspidosperma polyneuron*), cherry tree, ipê (Brazilian walnut), chestnut tree, grápiá, pau-pereira, and freijó (Bortoletto and Alcarde 2013; Bortoletto et al. 2016; Bortoletto et al. 2021). The use of tropical woods contributes a different physicochemical composition and sensory characteristics to cachaça (Alcarde et al. 2010; Bortoletto et al. 2021). Ageing cachaça using tropical wooden barrels opened a new market to the diversity of tropical wooden barrels and possibilities for blending, resulting in different flavours and sensory character (Bortoletto et al. 2021).

During ageing, compounds in cachaça interact with cellulose, hemicellulose, and lignin in the wood, undergoing decomposition facilitated by alcohol and water. Lignin degradation is responsible for the generation of compounds with low molecular weight including phenolic compounds (Carvalho et al. 2020; Castro et al. 2020; Silvello et al. 2021) which have been widely studied due to antioxidant properties (Bortoletto 2023). Phenolic compounds are secondary metabolites obtained from plants, bacteria, or fungi with high antioxidant capacity (Carvalho et al. 2020) and are divided into flavonoids (isoflavonoids, anthocyanidins, flavonoids, flavonols, flavanone, and flavones), and non-flavonoids (hydroxycinnamic and hydroxybenzoic acids, stilbenoids, lignoids and coumarins). In foods, phenolic compounds contribute colour, astringency, aroma and oxidative stability (Carvalho et al. 2020; Castro et al. 2020).

Ageing cachaça in tropical wooden rather than oak barrels results in a similar spectrum of compounds but with differences in concentration (Zacaroni et al. 2011). Castanheira (Pará or Brazil nut) barrels contribute phenolic compounds such as gallic acid, and ellagic acid (Zacaroni et al. 2011; Bortoletto et al. 2016). Barrels made from the timber tree umburana contribute curamin, eugenol, cinnamon laurel, catechin and vinylic acid (Alcarde et al. 2014). Barrels made from balsamo contribute phenolic compounds such as ellagic acid and vanillin (Alcarde et al. 2010; Bortoletto et al. 2016). However, barrels made from Brazilian walnut (ipê) contribute syringic acid, vanillic acid and coniferaldehyde (Alcarde et al. 2014; Bortoletto et al. 2016). Similarly, the phenolic acids from peroba wood are vanillin and syringic acid (Santiago et al. 2017). Gallic acid and syringaldehyde are the main phenolic compounds found by ageing in jatobá barrels (Bortoletto et al. 2016; Santiago et al. 2017).

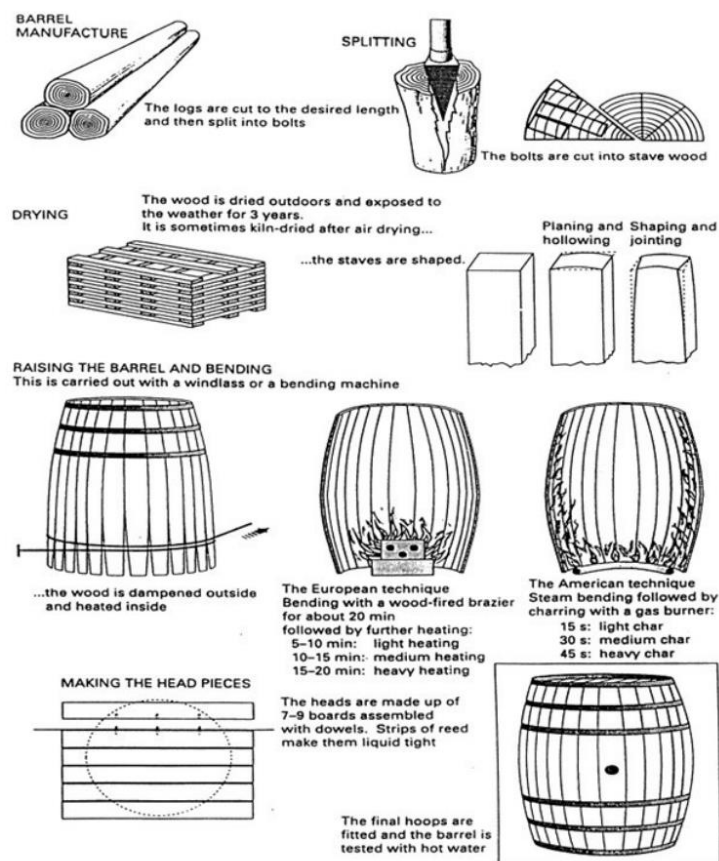
An evaluation of tropical wooden barrels by Bortoletto et al (2016) reported that cachaça aged in oak provide a sweet, smooth taste with a vanilla aroma and a yellow colour. A similar colour was observed in cachaças aged in barrels made from umburana, cabreúva and jatobá. In contrast, barrels made from feijó, peanut wood, araruva, and jequitibá did not influence the colour of aged cachaça (Alcarde et al. 2010; Santiago, et al. 2017). Balsamo barrels contributed a sharp and pleasant aroma with a reddish-yellow colour (Bortoletto et al. 2016; Santiago et al. 2017). Ageing in barrels from peanut wood, pear tree, jatobá, and eucalyptus contributed similar characteristics to oak barrels including colour, together with polysaccharides and lignin, which improve the sensory character (Souza et al. 2012).

Wooden barrels

No two wooden barrels are the same, reflecting the background of the tree (genetic and topographical), the part of the tree from which the wood was taken and subsequent variations in the production process (Gollihue et al. 2021). The manufacture of wooden barrels is outlined in Figure 4. France and the United States are the largest producers and exporters using oak from French forests (*Quercus petraea*, *Q. robur*) and American oak (*Q. alba*) (Cadahia et al. 2009; Carpena et al. 2020). The wood used in barrels for

Figure 4.

Barrel manufacture for spirit ageing (adapted from Mosadale e Puech 1998).



spirit maturation must meet requirements for porosity, permeability, and chemical composition (Cadahía et al. 2009). Cylinder cuts are used to form the staves, which are dried, cut to shape and are heated to be more malleable (Mosadale and Puech 1998). After assembly of the barrel by the Cooper, the internal surface is charred or 'toasted' to be light, medium or heavy using different methods in Europe and the USA (Figure 4).

Toasting changes the structure, and composition of wood, which impacts on the spirit during ageing (Mosadale and Puech 1998; Bortoletto et al. 2021). The toasting process is divided into three types: 1) 'light' which contributes a yellow colour to the beverage 2) 'medium' which results in a red colour and 3) 'heavy' gives a dark brown colour. Barrels toasted using heavy toasting result in smoky aromas which may not be desirable (Mosadale and Puech 1998; Bortoletto et al. 2016; Bortoletto et al. 2021).

Flavour and aroma compounds

Cachaça contains both volatile (aromas), and non-volatile (flavour) compounds (Portugal et al. 2017; Bortoletto 2023). Cachaça flavour and aroma is influenced by numerous factors including sugar cane cultivar, yeast microbiota, fermentation conditions, distillation method and – where applied – ageing (barrel wood and toasting) (Bortoletto and Alcarde 2013; Granato et al. 2014, Bortoletto et al. 2016). Cachaça spirit contains a multitude of secondary compounds including aldehydes, esters, acids, alcohols, and phenols (see Table 1). Even at low concentrations compounds influence flavour and aroma through complex interactions which can be both synergistic and antagonistic.

Table 1.

Volatile compounds identified in cachaça aged in different types of wooden barrel.

CLASS	COMPOUNDS	DESCRIPTORS	WOOD
Ethyl esters	ethyl pentanoate; ethyl hexanoate; ethyl heptanoate; ethyl octanoate; ethyl nonanoate; ethyl decanoate; ethyl undecanoate; ethyl dodecanoate; ethyl tridecanoate; ethyl tetradecanoate; ethyl pentadecanoate; ethyl hexadecanoate; ethyl octadecanoate; diethylsuccinate; diethyl malate	Alcohol, apple, banana	Cerejeira, Castanheira
Acetate esters	ethyl butyrate; propyl acetate; isobutyl acetate; hexyl acetate; isoamyl acetate; heptyl acetate; octyl acetate; nonyl acetate; dacyl acetate; dodecyl acetate; tetradecyl acetate; hexadecyl acetate	Perfume, rancid	Amendoim, Araruva
Propyl esters	propyl octanoate; propyl dodecanoate; propyl hexadecanoate	Sweet	Carvalho, Cerejeira
Decanoate esters	pentyl decanoate; heptyl decanoate	Vinegar	Cabreúva, Castanheira, Cerejeira
2-Methylpropyl (isobutyl) Esters	2-methylpropyl octanoate; 2-methylpropyl decanoate; 2-methylpropyl dodecanoate; 2-methylpropyl tetradecanoate	Soap	Castanheira, Cerejeira
3-Methylbutyl (isopentyl) esters	3-methylbutyl acetate; 3-methylbutyl hexanoate; 3-methylbutyl octanoate; 3-methylbutyl decanoate; 3-methylbutyl dodecanoate	Orange peel Papaya	Castanheira
Phenylethyl esters	pentyl acetate; phenethyl acetate; phenethyl butanoate; phenethyl hexanoate; phenethyl octanoate	Roses	Cabreúva
Propan-2-yl (isopropyl) esters	propan-2-yl dodecanoate; propan-2-yl tetradecanoate; propan-2-yl hexadecanoate	Solvent, sugar cane	Amburana
Aldehydes	acetaldehyde; furfural; heptanal; octanal; nonanal; decanal; undecanal; dodecanal; tridecanal; tetradecanal; pentadecanal; hexadecanal; octadecanal	Honey	Grápia, Carvalho, Cerejeira, Ipê, Jequitibá
Fatty alcohols	propanol; pentanol; hexanol; heptanol; octanol; nonanol; decanol; undecanol; dodecanol; tridecanol; tetradecanol; pentadecanol; hexadecanol; 2-methyl-1-butanol; 3-methyl-1-butanol	Butter, citrus	Carvalho
n-Alcohols	propane-1,2-diol; heptan-2-ol; octan-2-ol; nonan-2-ol; decan-2-ol; undecan-2-ol; dodecan-2-ol; tridecan-2-ol; pentadecan-2-ol; furfuryl alcohol	Coconut, fruity	Castanheira, Cerejeira
Acids	acetic acid; propionic acid; isobutyric acid; butyric acid; hexanoic acid; heptanoic acid; 2-ethyl caproic acid; octanoic acid; nonanoic acid; decanoic acid; benzoic acid, ellagic acid. vanillic acid, gallic acid	Vinegar, pungent, acidic, dairy-like	Bálsamo, Ipê, Carvalho, Jequitibá, Amburana
Phthalates	pentyl decanoate; heptyl decanoate; 2-methylpropyl butyl benzene-1,2-dicarboxylate; bis(2-methylpropyl) benzene-1,2-dicarboxylate; dibutyl benzene-1,2-dicarboxylate	Waxy, wood	Cabreúva, Carvalho
Fatty acid ester	heptan-2-one; octan-2-one; nonan-2-one; decan-2-one; undecan-2-one; dodecan-2-one; tridecan-2-one; pentadecan-2-one; hexadecan-2-one; heptadecan-2-one	Green apple, green leaves	Cerejeira
Ketones	nonane; undecane; tridecane; pentadecane; heptadecane; octadecane; nonadecane; docosane	Bitter, lemon	Cerejeira
Alkanes			

Information derived from Alcarde et al. 2010; Bortoletto et al. 2021; Catão et al. 2011; Dias et al. 1998; Nóbrega 2003; Cardeal and Mariott 2009; Silva et al. 2009; Souza et al. 2009; Duarte et al. 2011; Duarte et al. 2013; Amorim et al. 2016; Gonçalves et al. 2016; Santiago et al. 2016; Zacaroni et al. 2017; Nascimento e Silva et al. 2020.

Esters are important aroma compounds and are formed during fermentation, distillation, and ageing (Bortoletto et al. 2016; Portugal et al. 2017; Oliveira et al. 2020). Ethyl carbamate (EC) is a carbamic acid ester formed during fermentation, distillation, and ageing (Nóbrega et al. 2009; Machado et al. 2013; Bortoletto and Alcarde 2016) and is a concern due to its carcinogenic properties (Machado et al. 2013; Gonçalves et al. 2016). Higher alcohols contribute to cachaça aroma and are generated by yeast via the Ehrlich pathway together with aldehydes and short-chain fatty acids (Hazelwood et al. 2008). Acetaldehyde contributes positively to the aroma of cachaça (Ribeiro-Filho et al. 2021). Other aldehydes such as furfural and hydroxymethylfural can be formed when sugar cane crop is burned before harvesting and generated during distillation due to a poor separation of yeast after fermentation (Bortoletto 2023). The alcohol, methanol is undesirable as it can cause health problems even at low concentrations (Alcarde et al. 2014).

Quality control

Cachaça is influenced by the geographical origin, field and industrial practice (Alcarde et al. 2012; Bortoletto 2023;) but the quality of the spirit must be controlled and standardised (Brazil 2022b). The geographical origin inevitably results in variation but promotes diverse flavour profiles, contributing to the complexity of the cachaça. Similarly, field practice encompasses the sugar cane supply chain from choosing a cultivar to harvesting. The industrial processes are key to minimising undesirable organic or inorganic contaminants such as copper, dimethyl sulphide, and ethyl carbamate.

Copper

The Brazilian legislation limits copper in cachaça to 5 mg/L (Brazil 2022b). Sugar cane contains copper and the juice contains ca. 0.06 mg copper/L) which is required by yeast during fermentation. The divalent cation acts as an essential cofactor for enzymes

(including cytochrome C oxidase, lactase, and Cu, Zn superoxide dismutase) and is important for yeast metabolism during iron homeostasis (De Freitas et al. 2003). However, copper present in the must/wort does not contribute to the copper content of the spirit as it remains in the 'tail' fraction post distillation. Distillation using stainless steel columns does not contribute copper, whereas cachaça distilled in copper still pots does. Here, oxidation of internal walls of the copper still generate a copper salt, which dissolves in cachaça (Rosa et al. 2009; Alcarde et al. 2014; Böck et al. 2022). Therefore, copper content can increase to levels that are of concern (>5 mg/L). Accordingly, to reduce copper exposure and copper content within legal limits, the internal walls of copper pot stills should be cleaned before distillation (Rosa et al. 2009; Oliveira et al. 2020; Böck et al. 2022).

Dimethyl sulphide

Dimethyl sulphide is not regulated by Brazilian legislation, but generates sulphur off-flavours in the spirit (Rosa et al. 2009). Dimethyl sulphide is generated by yeast during fermentation but is captured during distillation in copper still pots. However, cachaça distilled in stainless steel columns contains dimethyl sulphide and can result in product with sulphur off-flavours (Alcarde et al. 2014). These may be mitigated by the addition of up to 6 g/L of sucrose to reduce the perception of off-flavours.

Ethyl carbamate

Sugar cane is a cyanogenic crop characterised by containing cyanogenic glycosides. Although little is known about the metabolic pathway of ethyl carbamate (EC) formation during fermentation of sugar cane juice (Rosa et al. 2009; Lachenmeier et al. 2010). Ethyl carbamate is a concern, potentially carcinogenic and an undesirable compound in cachaça (Nóbrega et al. 2009; Alcarde et al. 2012; Machado et al. 2013; Mendonça et al. 2016; Santiago et al. 2017).

The formation of EC is influenced by the raw material, fermentation and distillation practice (Lachenmeier et al. 2010; Alcarde et al. 2012; Santiago et al. 2014; Mendonça et al. 2016). During fermentation, urea supplementation increases EC formation (Nóbrega et al. 2009; Ljungdahl and Daignan-Fornier 2012)

with the amino acid arginine a precursor of urea, carbamyl phosphate, and cyanide (Jiao et al. 2014). The nitrogenous precursor interacts with ethanol to form ethyl carbamate (Jiao et al. 2014; Santiago et al. 2014). Studies reveal that the use of commercial yeast strains reduce the formation of ethyl carbamate (Lima et al. 2012; Santiago et al. 2017) as did wild yeasts from sugar cane juice supplemented with corn flour or rice bran (Santiago et al. 2014; Mendonça et al. 2016; Santiago et al. 2017).

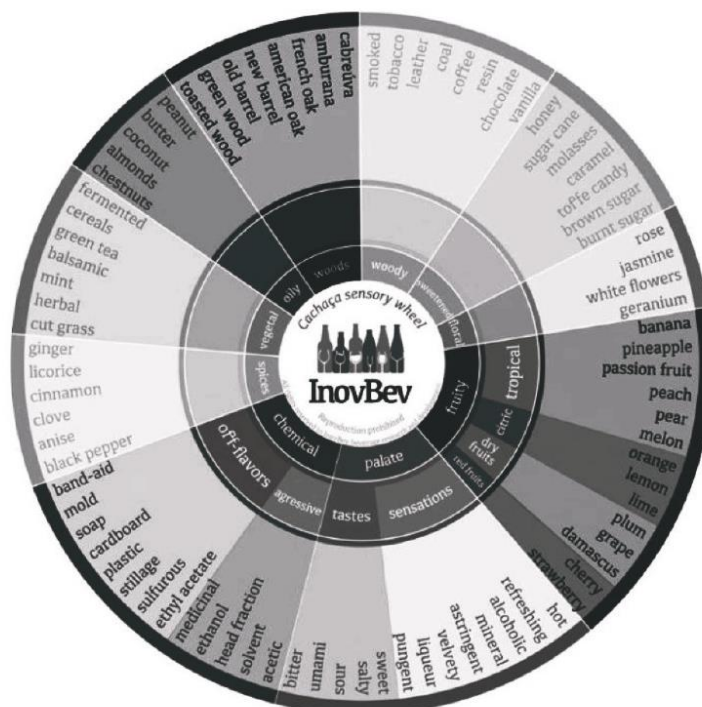
Ethyl carbamate is formed during fermentation, but its concentration increases during distillation (Nóbrega et al. 2009; Lachenmeier et al. 2010). Separation of the distilled spirit into three fractions (heads, heart, and tails) enables the reduction of ethyl carbamate as the heads fraction retains much of the ethyl carbamate (Alcarde et al. 2012; Nova et al. 2009; Rota et al. 2013; Santiago et al. 2017). Cachaça distilled in stainless steel columns is associated with the low levels of ethyl carbamate, but distillation in a copper pot results in a higher concentration of EC which can be reduced by longer distillation (Nóbrega et al. 2009; Lima et al. 2012;). Double, triple, or multiple distillation reduces the concentration of congeners (acetic acid, aldehyde, ethyl acetate, propyl alcohol and furfural) and, importantly, compounds of concern (methanol, butane-2-ol, copper and ethyl carbamate) (Alcarde et al. 2012; Bortoletto et al. 2016).

It was thought that ethyl carbamate increases during the ageing process. However, there is no evidence that ethyl carbamate increases to levels above the international limit of 150 µg/L during ageing (Santiago et al. 2014; Bortoletto et al. 2016; Mendonça et al. 2016). In passing, cachaça stored in colourless glass bottles for six months results in an increase in the EC content due the exposure to natural light (Zacaroni et al. 2015).

Sensory analysis

Cachaça is a spirit with a distinctive sensory character with flavours from fermentation, distillation, and the ageing process. The aroma and flavour of cachaça offer a unique and complex matrix, which requires greater knowledge of its sensory attributes (Bortoletto et al. 2021). Sensory analysis determines the main attributes of the beverage and can facilitate the improvement and standardisation

Sensory wheel for cachaca (Bortoletto 2023).



Future trends

Further studies are required to improve understanding of the adaptation of sugar cane cultivars, the characteristics of sugar cane juice (including microbiota), and the influence on fermentation, distillation, and ageing of cachaça. Further, given the ecological diversity and varieties of sugar cane, the understanding of yeast handling for cachaça production is poor. Selecting and characterising the indigenous yeasts (physiology, genetic identity and diversity, metabolome etc) from different sugar cane cultivars and producers can create special cachaças with a mix of yeasts to create novel sensory characteristics. Fermentation would benefit from studies targeted at 1) the dominance and persistence of yeasts, 2) fermentation kinetics, 3) nutrient transport, 4) yeast drying, 5) hydration of dried yeast, and 6) storage of dried yeast.

The distillation process for cachaça takes inspiration from production of 'bagaceira' (Portuguese brandy) and whiskey. There are opportunities for the improvement in cachaça distillation such as the volatilisation kinetics of compounds during distillation in copper and/or stainless steel stills. Similarly, the use of different tropical woods for barrel ageing of cachaça ageing opens the door to new opportunities. Underpinning any developments or innovations is the application of sensory science to fingerprint the spirit and assure quality. Finally, the application of new analytical techniques – such as fluorescence and infrared spectroscopy – can be applied for the quality control of cachaça. Such methods are worthy of development as they non-destructive and have application in on-line monitoring.

Conclusions

Cachaça production is defined process with supporting regulation. Field practice involves planting of sugar cane, harvesting and transportation whilst industrial practice involves juice extraction, fermentation, distillation, ageing, and standardisation. Cachaça's sensory characteristics are determined by the climate, soil, environment, sugar cane cultivar and associated microbiota. Fermentation practice contributes to the sensory characteristics of cachaça, determined by indigenous or starter commercial yeasts. Cachaça fermentation is performed in open vessels resulting in the loss through gas washing of some aroma volatiles. Producers prefer to use a copper pot still for cachaça distillation with the increasing use of different tropical wood for barrels ageing. The options for different toasting regimes of barrels influences the flavour and aroma of cachaça providing opportunities for differentiation of the spirit.

Author contributions

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Declaration of competing interest

The authors declare there are no conflicts of interest.

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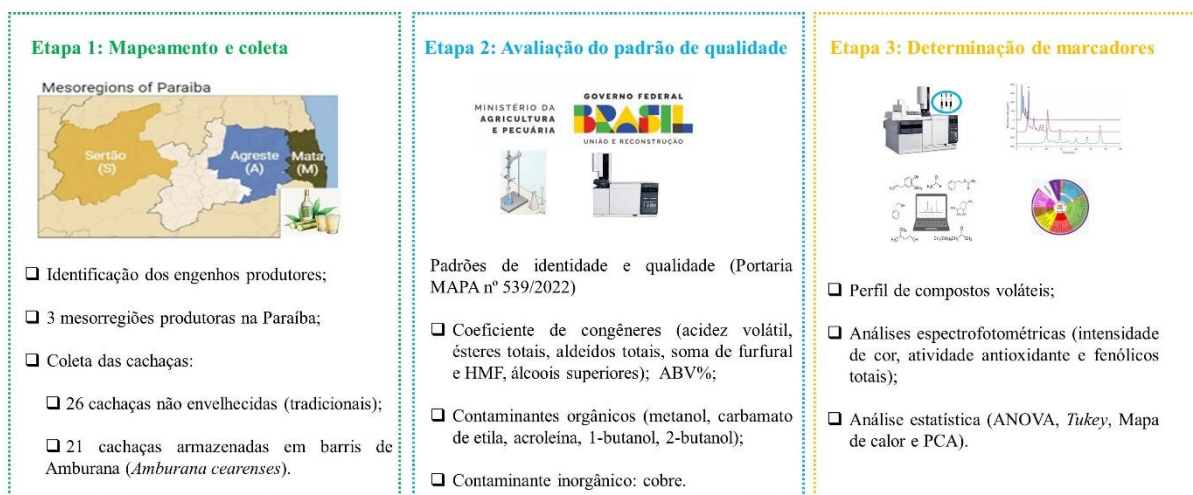
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4 DELINEAMENTO EXPERIMENTAL

A coleta das amostras para o estudo foi realizada em três mesorregiões do Estado da Paraíba (Mata Paraibana, Agreste e Sertão Paraibano), onde foram coletadas cachaças produzidas por diferentes produtores durante o ano de 2021. A coleta das amostras foi dividida em dois tipos de cachaça produzidas: cachaças não envelhecidas (tradicionais) e cachaças armazenadas em barris de Amburana (*Amburana cearenses*). O delineamento experimental apresentado na Figura 1, resume a metodologia para o desenvolvimento do projeto em três etapas.

Figura 1. Delineamento experimental



Fonte: Autora (2025)

4.1 ETAPA 1: MAPEAMENTO E COLETA

A primeira etapa do esquema representado na Figura 1 (Etapa 1) representa o mapeamento e coletas das cachaças nos engenhos produtores nas três mesorregiões do estado da Paraíba (Mata Paraibana, Agreste e Sertão Paraibano). No total foram coletadas 47 cachaças: 26 cachaças não envelhecidas (tradicionais) que foram produzidas por fermentação "natural", destiladas em alambiques de cobre e armazenadas em tanques de aço inoxidável ou barris de madeira freijó por no máximo 6 meses, e envasadas em garrafas de vidro; e 21 cachaças armazenadas em barris de Amburana (*Amburana cearenses*) produzidas por fermentação

"natural", destiladas em alambiques de cobre, armazenadas em barris de madeira Amburana por períodos entre 4 meses e 4 anos, e envasadas em garrafas de vidro. Todas as amostras foram protegidas da luz e mantidas a uma temperatura média de 20°C. As análises foram realizadas em triplicata.

4.2 ETAPA 2: AVALIAÇÃO DO PADRÃO DE QUALIDADE

A segunda etapa do esquema representado na Figura 1 (Etapa 2) apresenta a caracterização química das cachaças analisadas, de acordo com os padrões de identidade e qualidade: coeficiente de congêneres (acidez volátil, ésteres totais, aldeídos totais, soma de furfural e HMF, álcoois superiores), % ABV, contaminantes orgânicos (metanol, carbamato de etila, acroleína, 1-butanol, 2-butanol) e contaminante inorgânico (cobre); determinadas pela Portaria MAPA Nº 539/2022 (Brasil, 2022).

4.3 ETAPA 3: DETERMINAÇÃO DE MARCADORES

A terceira etapa do esquema representado na Figura 1 representa a etapa da determinação dos compostos marcadores das cachaças paraibanas, através da determinação do perfil de compostos voláteis e análises espectrofotométricas (intensidade de cor, atividade antioxidante e fenólicos totais), apenas para as cachaças armazenadas em barris de Amburana).

5 RESULTADOS

Os resultados obtidos nesta pesquisa são apresentados em formato de 2 artigos científicos, de acordo com a Norma Complementar nº 01/2024 do PPGCTA e submetidos a periódicos de alto fator de impacto.

O Artigo I, teve como objetivo avaliar a conformidade com os parâmetros legais e o perfil aromático de cachaças não envelhecidas (tradicionais) produzidas em diferentes origens geográficas da Paraíba (incluindo as mesorregiões da Mata Paraibana, do Agreste e do Sertão Paraibano). Vinte e seis amostras foram coletadas e submetidas a análises de qualidade e perfil volátil utilizando método cromatográfico (CG-MS). As cachaças analisadas apresentaram conformidade com os padrões legais em quase sua totalidade. Com relação ao perfil aromático, as cachaças produzidas na mesorregião do Agreste apresentaram semelhanças com as cachaças do Sertão Paraibano e diferenças com as cachaças da Mata Paraibana. O estudo destaca como a origem geográfica influenciada por fatores edafoclimáticos e de produção, exerce influencia na qualidade da cachaça em conformidade os padrões legais de identidade e qualidade; bem como influencia seu perfil aromático, contribuindo para a compreensão do *terroir* da cachaça.

O artigo II, teve como objetivo avaliar a qualidade e a composição volátil e não-volátil de cachaças armazenadas em barris de Amburana (*Amburana cearenses*) produzidas no estado da Paraíba. Foram coletadas 21 cachaças e submetidas as análises de qualidade, perfil volátil utilizando método cromatográfico (CG-MS) e perfil não-volátil mediante análises de intensidade de cor, atividade antioxidante e compostos fenólicos totais. Parte das cachaças analisadas apresentaram inconformidade com os padrões legais, influenciadas por fatores de produção como higiene inadequada, controle da fermentação, práticas de destilação, tempo de armazenamento e condições dos barris. O perfil aromático identificou 45 compostos voláteis, contribuindo com aromas doce, floral, frutado e amadeirado. Foram revelados diferentes perfis aromáticos entre as cachaças analisadas, influenciados pelo tempo de armazenamento (entre 4 meses e 4 anos) e condições dos barris. O estudo revelou grande variabilidade na qualidade das cachaças armazenadas em barris de Amburana, destacando a necessidade de protocolos de higiene, padronização das condições de envelhecimento, como reutilização de barris e tempo de armazenamento, garantindo segurança e qualidade para cachaça armazenada em barril de Amburana; no entanto, o processo de envelhecimento enriqueceu a bebida com aromas desejáveis que melhoram seu perfil sensorial.

5.1 ARTIGO I - CHEMICAL COMPOSITION AND SENSORY DIVERSITY OF COLOURLESS CACHAÇAS FROM PARAÍBA: INFLUENCE OF GEOGRAPHIC ORIGIN AND PRODUCTION PRACTICES.

O artigo foi aceito para publicação no periódico *Journal of the Institute of Brewing*, sob o título *Chemical Composition and Sensory Diversity of Colourless cachaças from Paraíba: Influence of Geographic Origin and Production Practices*, desde o dia 11 de junho de 2025 (ANEXO A).

Chemical Composition and Sensory Diversity of Colourless Cachaças from Paraíba: Influence of Geographic Origin and Production Practices

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Abstract

(i) Why was the work done: This study was conducted to investigate how geographic origin, including edaphoclimatic conditions, field practices, and industrial processing, influences the chemical composition and aroma profile of colourless cachaças from the Paraíba state in Brazil. While cachaça's technological development has advanced, the specific effects of regional factors on compliance with legal standards and sensory diversity remain underexplored. **(ii) How was the work done:** Twenty-six samples of crystal cachaça from the Mata, Agreste, and Sertão mesoregions were analysed. Physical-chemical parameters (e.g., alcohol content, volatile acidity, and copper concentration) were determined using flame atomic absorption spectroscopy (FAAS). Volatile compounds, including esters, alcohols, aldehydes, and ethyl carbamate, were identified and quantified using gas chromatography with flame ionization detection (GC-FID) and gas chromatography-mass spectrometry (GC-MS). **(iii) What are the main findings:** The results showed that five producers failed to meet legal copper limits, with deficiencies linked to inadequate cleaning of copper stills and poor distillation control. Eighty-five volatile compounds were identified, revealing significant regional differences in aroma profiles. **(iv) Why is the work important:** The findings demonstrate that the sensory and chemical diversity of cachaça is driven by a complex interaction of edaphoclimatic conditions, sugarcane management, and processing methods. These factors contribute to the distinctiveness of regional cachaças and reinforce their potential for market differentiation and consumer loyalty.

Keywords: Cachaças, geographic origin, Industrial practice, legal parameters, aroma profile

35 Introduction

36 Sugarcane is one of the most used crops in the world and its agricultural and industrial
 37 potential is well known. In general, sugarcane is a plant with great industrial and economic
 38 potential, due to the existence of more than 260 sugarcane derivatives, in Brazil,
 39 derivatives such as ethanol, sugar and cachaça have prominence and greater impact
 40 economic (Solomon 2011, Rosa *et al.* 2009). Cachaça is the traditional Brazilian spirit,
 41 which should contain between 38-48% of alcohol (Da-Silva *et al.* 2023). Cachaça is
 42 produced using fresh sugarcane juice that is submitted to fermentation (spontaneous
 43 fermentation or fermentation using yeast starters) and distillation (using stainless steel
 44 columns or copper still pots) (Bortoletto 2023, Borges *et al.* 2014). Currently, twenty-seven
 45 Brazilian states contain cachaça producers, but the largest state producers (including
 46 producers that use stainless steel columns and copper still pots) are São Paulo,
 47 Pernambuco, Ceará, Minas Gerais and Paraíba (Santiago *et al.* 2015). However, when
 48 consider only cachaças distilled using copper still pots, Paraíba state is the biggest
 49 cachaça producer when considered products distilled using copper still pots, containing
 50 more than 40 regulated producers. Moreover, cachaças produced in Paraíba state, have
 51 been well recognised due to their quality, which indicate that producers found a great
 52 balance between up-scaling and quality. The differences of cachaças can be associated
 53 with the characteristics of each region or mesoregions because each region/mesoregion is
 54 characterised by differing their weather, soil, environment, the adaptation of sugarcane
 55 cultivars, and consequently, the type of wild yeast native.

56 Brazil is a large country, lately, Paraíba state is on the spotlight due to its cachaças' quality
 57 and became the biggest cachaça producer when considered cachaças distilled using
 58 copper still pots. Paraíba state is divided into four mesoregions including Sertão Paraibano
 59 (2 producers), Borborema (zero producers), Agreste Paraibano mesoregion (20
 60 producers) and Mata Paraibana mesoregion (4 producers). The sertão paraibano
 61 mesoregion is defined by hot temperatures, and highly irregular and low rainfall (Medeiros-
 62 Silva *et al.* 2019). The Mata Paraibana mesoregion is defined by hot and humid tropical
 63 climate (Medeiros-Silva *et al.* 2019, Vilela *et al.* 2021). The Agreste mesoregion is defined
 64 by medium to low temperatures, irregular rainfall and produces most of the cachaça in
 65 Paraíba (Medeiros-Silva *et al.* 2019). These four mesoregions distinct characteristics in
 66 terms of landscape, agriculture, and cachaça production, which reflects in the different
 67 cachaça quality generated by a distinct cachaça *terroirs*, each influenced by variations of
 68 edaphoclimatic condition (including soil, climate, and topography), field practice, and
 69 industrial practice (Medeiros-Silva *et al.* 2019, Vilela *et al.* 2021, Capitello *et al.* 2021).
 70 Therefore, cachaças may have their characteristics influenced by the mesoregion and its
 71 edaphoclimatic condition/industrial practice, which reflects in different cachaça quality and
 72 its *terroir* (Vilela *et al.* 2021, Capitello *et al.* 2021).

73 Cachaça can be classified as crystal (colourless), short-aged and aged cachaça, which
 74 are related to the type of storage, which can be conducted in stainless steel barrels or
 75 wooden barrels (maturation). Although several producers store their freshly distilled
 76 product in stainless steel barrels, which not contribute to cachaça ageing; lately, cachaça
 77 producers started to use wood barrels built from tropical woods such as feijó and jequitibá,
 78 which do not change the cachaça colour but improve the chemical composition of cachaça
 79 (Alcarde *et al.* 2014, Bortoletto 2023). Therefore, cachaça producers can improve the
 80 flavour characteristics of a crystal cachaça without change the colour characteristics of the
 81 product, which became a part of industrial practice in some regions. The production and
 82 quality of cachaça is a well-defined supported by regulations; however, aroma profile
 83 refers to the specific combination and intensity of aromas present, which are essential to
 84 define the unique sensory qualities and perceived quality of cachaça.

Cachaça has been extensively studied, providing valuable insights that have driven the development of innovative technologies aimed at improving both field and industrial practices. Despite these advancements, producers continue to face significant challenges, particularly regarding how geographic origin influences compliance with legal standards and the complex flavour profile of the spirit. While previous studies have addressed aspects of legal compliance and aroma profiles of cachaça, there remains a distinct gap in understanding the extent to which geographic origin - encompassing edaphoclimatic conditions - and production practices collectively impact these factors. The chemical composition of cachaça is shaped not only by environmental variables such as soil type, climate, and regional cultivation techniques but also by the specific practices employed during fermentation, distillation, and maturation. Therefore, this study aims to evaluate the compliance of colourless cachaças from the Mata, Agreste, and Sertão mesoregions of Paraíba with Brazilian legal standards and to analyse their volatile aroma profiles. Furthermore, it seeks to elucidate how the interplay between geographic origin, reflecting diverse edaphoclimatic conditions, and field and industrial practices contributes to the chemical and sensory diversity observed in these cachaças.

101

102 **Methodology**

103 **Sample collection**

104 Twenty-six colourless cachaça were collected in triplicate from twenty-six producers, which
105 are located in three mesoregions of the state of Paraíba including Sertão Paraibano
106 mesoregion (2 samples), Mata Paraibana mesoregion (4 samples) and Agreste Paraibano
107 mesoregion (20 samples)(Fig. 1). The cachaça samples were produced by the
108 fermentation of fresh sugarcane juice, distilled in copper stills and stored in stainless steel
109 tanks or freijó wood barrels for a maximum of 6 months. Afterwards, they were bottled in
110 glass bottles for commercialisation. All the samples were protected from light and kept at
111 20°C.

112

113 **Analytical methods**

114 **Determination of Alcohol by volume (ABV%)**

115 Alcohol by volume (ABV) was determined using alcoholmeter (Gay Lussac
116 Alcoholometer). Sample (200 mL) was transferred to a volumetric cylinder and
117 alcoholmeter was submerged in the sample for data collection. Results were accurate at
118 20 °C using a correction table (AOAC 2000). Results were expressed in ABV (% v:v).
119

120 **Total Acidity (TA)**

121 This procedure is based on acid neutralization titration (AOAC 2000). Pipette 50 mL of the
122 sample into the an Erlenmeyer flask and add four drops of 1% phenolphthalein (indicator).
123 Then, samples were titrated with 0.1 M sodium hydroxide solution. Results were calculated
124 using Equation 1:

$$TA = \frac{n \times M \times f \times MM}{10 \times V} \quad (\text{Equation 1})$$

125

126 Where, n = volume spent on titrating the sodium hydroxide solution, in mL, M = molarity of
127 the sodium hydroxide solution, f = correction factor of the sodium hydroxide solution, MM =
128 Molar Mass of acetic acid (60 g /mol), V = volume taken from the sample, in mL
129

Dry acidity (DA)

Dry acidity is determined by evaporating the sample and titrating the residual acids with alkali (AOAC 2000). First, Pipette 50 mL of the sample into the porcelain capsule and evaporate in a water bath. Carefully add water to the walls of the capsule, washing off the residue and continue evaporation until almost completely dry. Transfer this residue with 100 mL of water to an Erlenmeyer flask and add four drops of 1% phenolphthalein (indicator). Then, samples were titrated with 0.1 M sodium hydroxide solution (AOAC 2000). Results were calculated using Equation 2:

$$DA = \frac{n \times M \times f \times MM}{10 \times V} \quad (\text{Equation 2})$$

Where, n = volume spent on titrating the sodium hydroxide solution, in mL, M = molarity of the sodium hydroxide solution, f = correction factor of the sodium hydroxide solution, MM = Molar Mass of acetic acid (60 g/mol), V = volume taken from the sample, in mL

Volatile acidity (VA)

Volatile acidity was obtained by the difference between the total acidity and the dry acidity (AOAC 2000). Results were calculated using Equation 3 and were expressed in mg of acetic acid per 100 mL.

$$VA = TA - DA \quad (\text{Equation 3})$$

Where, VA= volatile acidity, TA = total acidity, DA = dry acidity

Higher alcohols and aldehydes

Isobutyl alcohol, 1-propanol, isoamyl alcohol, methanol, 1-butanol, 2-butanol, ethyl acetate, acetaldehyde, acrolein and the sum of furfural and hydroxymethylfurfural was determined from direct sample injection in a Gas Chromatography a equipment (Agilent, HP 6890), using a Flame Ionization Detector (FID) and column capillary Varian CP-Wax 52 CB (60m x 0.25mm x 0.25µm). The following conditions from the methodology of Bortoletto *et al.* (2016). The oven temperature program was as follows: started at 40 °C for 4 min., followed by an increase to 120 °C at 20 °C/min, an isothermal period of 1 min, an increase to 180 °C at 30 °C/min and a maintenance period of 4 min. The injector and detector (FID) temperatures were set at 245 °C and 250 °C respectively. The analyses were carried out with a 1:25 split ratio, using helium as the carrier gas (flow rate 1.5 mL/min). The solutions for the calibration curves and analytical standards were prepared with 40% (v/v) ethanol. The analyses were carried out by directly injecting 1.0 µL aliquots of the samples with 3-pentanol (internal standard).

Ethyl carbamate

Ethyl carbamate (EC) content was determined from direct sample injection in a Gas Chromatograph (GC) Agilent Technologies 7890B attached to a mass-selective detector Agilent Technologies 5977B (MSD), equipped with column capillary Agilent VF-WAXms (60m x 0,25mm x 0,25µm), coupled to a Mass Spectrometer. The following conditions from the methodology of Nóbrega *et al.* (2009). The oven temperature program was as follows: 90 °C (2 min); 10 °C/min to 150 °C (0 min); and 40 °C/min to 230 °C (10 min). The injector temperature was 250 °C, and the GC/MS interface was maintained at 230 °C. The MS was operated in the electron impact mode with an ionisation energy (70 eV) and helium at 1.5 ml/min was used as carrier gas. The mass spectrometer detector operated in the SIM mode (m/z 62). Preparation of calibration curves and EC analysis were based on of

methodology adapted from Serafim *et al.* (2016). The solutions of ethyl carbamate and butyl carbamate from prepared with ethanol 40% (v/v). The butyl carbamate (150 µg/L) was used as an internal standard. The injected volume was 1.0 µL in splitless mode. Ethyl carbamate was quantified by internal standard addition. The limits of detection and quantitation were 10 and 40 µg/L of EC, respectively.

181

182 Inorganic compound (Copper, Cu)

The copper analysis method was recommended by Miranda *et al.* (2010). Deionized water (18 MΩ cm resistivity) was used for all solution preparations, obtained from a Milli-Q® Plus Total Water System (Millipore Corp., Bedford, MA, USA). To prevent contamination, all glassware and polypropylene flasks were pre-cleaned with detergent, soaked in 10% (v/v) nitric acid (HNO₃) for 24 h, rinsed with deionized water, and air-dried. Reference solutions were freshly prepared daily by diluting 1000 mg/L Ag, Bi, Co, Cu, and Ni stock standards (Tec-Lab, Hexis, São Paulo, Brazil). Commercial cachaça samples were obtained from local markets in São Carlos, São Paulo State, Brazil.

Cachaça samples underwent total digestion using a Multiwave microwave system (Anton-Paar, Graz, Austria) with perfluoroalkoxy (PFA) vessels and a temperature sensor. Each sample (1 mL) was digested with 1 mL of 14 mol/L HNO₃ and 6 mL of deionized water in a closed vessel. The digestion process included two heating steps: (1) 2 minutes at 300 W and (2) 5 minutes at 600 W. Digested samples were transferred to volumetric flasks and diluted to 10.0 mL with deionized water. All samples were processed in triplicate, and Cu concentrations were determined using inductively coupled plasma optical emission spectrometry (ICP-OES, Vista AX, Varian, Mulgrave, Australia). The calibration curves from prepared with ethanol 40% (v/v).

For direct Cu analysis, 5 mL of cachaça was mixed with 400 µL of an internal standard solution containing Ag, Bi, Co (50 mg/L), and Ni (250 mg/L) in a 15 mL polypropylene flask. The volume was adjusted to 10 mL with 1.0 mol/L HNO₃. After dilution, the final concentrations of internal standards were 10 mg/L Bi and 2 mg/L Ag, Co, and Ni. Calibration was performed using reference solutions with Cu concentrations ranging from 0.10 to 6.0 mg/L, with internal standards added to all blanks, reference solutions, and samples. Copper was quantified at 324.8 nm using Flame atomic absorption spectrometry (FS-FAAS). A Varian AA240FS spectrometer (Mulgrave, Australia), equipped with a deuterium lamp for background correction, was employed. Hollow cathode lamps for silver (Ag), bismuth (Bi), cobalt (Co), copper (Cu), and nickel (Ni) were used as primary radiation sources with respective electric currents of 4, 10, 7, 4, and 4 mA. The analyses were conducted using an oxidizing air/acetylene flame (13.5 L min⁻¹ / 2 L min⁻¹), with Cu measured at 324.8 nm using a spectral bandwidth of 0.5 nm.

213

214 Volatile profile

The volatile compounds (higher alcohols, aldehydes, ketones, terpenes and esters) were determined using a chromatograph (GC) Agilent Technologies 7890B attached to a mass-selective detector Agilent Technologies 5977B (MSD), equipped with column capillary Agilent HP-5MS UI (30m x 0.25 mm x 0.25 µm), coupled to a Mass Spectrometer. The following conditions from the methodology of Zacaroni *et al.* (2017) were used: oven temperature of 35 °C to 240 °C with a heating rate of 4 °C/min. The injector temperature was set at 270 °C. Helium 5.0 was used as carrier gas at a flow rate of 1.78 mL/min in 1:4 split mode injection system. The temperature of the detector interface and the ion source remained at 240 °C and 200 °C, respectively. The mass spectrometer was operated in

electronic impact mode (70 eV) and the mass scan range was from 50 to 400 mz^{-1} at 4.44 scan. s^{-1} . The extraction of volatiles was performed using the headspace solid-phase microextraction technique (HS- SPME) with SPME device (Supelco, Bellefonte, USA). The sample was diluted to 10% ethanol using ultra pure water. An aliquot of 4 mL added to internal standard 3-pentanol, was transferred to a 20 mL glass vial and immediately sealed with a Teflon-coated septum cap. The volatiles were extracted at 45 °C no equilibrium time, using the fiber Divinylbenzene/Carboxene/Polydimethylsiloxane (DVB/CAR/PDMS) 50/30 μm fiber was exposed to the headspace for 50 minutes of adsorption. Subsequently exposed for 5 minutes for desorption in the gas chromatograph. The fiber used was conditioned in 270 °C for 60 minutes accordance with the manufacturer's specifications before the extraction procedure. Compounds were identified using the NIST library database (2014) combined with a mass spectrum and linear retention index. The linear retention index (LRI) of each compound was calculated using the retention times of a homologous series of C6–C20 n-alkanes. Results were expressed as total chromatographic peak area.

Statistical Analysis

All analyses were carried out in triplicate and the results and data presented as the mean $\pm$ standard deviation of three repetitions, and the data were submitted to Analysis of Variance (ANOVA) and Tukey's test (to compare the results of the cachaças analyzed in relation to the parameters required by the legislation), where means with $p < 0.05$ were considered statistically different. Principal Component Analysis and Heat Mapping were also carried out (for the results of the volatile compounds). The data was analyzed using XLSTAT software (version 2014.5.03, Addinsoft, New York, USA).

Results and Discussion

Twenty-six cachaça samples from Paraíba, Brazil, using various analytical methods were evaluated including alcohols, esters, aldehydes, and copper. Samples were collected from different regions and produced via sugarcane fermentation, distilled in copper stills, and stored before bottling. Ethanol concentration and volatile acidity were determined by densimetry and acidity difference. Higher alcohols, esters, aldehydes, and ethyl carbamate were analysed using gas chromatography. Volatile compounds were assessed using headspace solid-phase microextraction. Copper content was measured via flame atomic absorption spectroscopy. Statistical analyses included ANOVA, Tukey's test, and Principal Component Analysis.

Compliance with Legal Parameters

The cachaça legislation requires a group of analysis (including ABV (%), volatile acidity, total esters, total aldehydes, sum of furfural and HMF, sum of isobutyl, isoamyl and n-propyl alcohols, methanol, 1-butanol, 2-butanol, ethyl carbamate, and copper). Each cited analysis contain a range of acceptable limit (Table 1). All cachaças were within the limits required by current legislation, except samples CA-05, CA-07, CA-10, CA-12, CA-19, and CM-03, which indicates that some producers needs to improve their industrial practices. On the other hand, the majority of producers apply a good production practice, which offer an authentic, and quality products, which helps build trust with consumers and enhances cachaça's reputation.

The alcohol content of cachaça, which is generated during fermentation and concentrated during distillation process, is defined by the volumetric percentage of alcohol present in the spirit (Rosa *et al.* 2009). Results reveal that all cachaças evaluated contain ABV from 38 to 44 % (v:v) as expected (38 - 48 %, v:v)(Table 1). Alcohol content was generated during fermentation when yeasts convert fermentable carbohydrates into ethanol, CO₂ and energy. Upon the completion of fermentation, spirit producers drive the fermented wort to a distillation equipment, which separates compounds by boiling point (Alcarde *et al.* 2012). Cachaça distillation can be conducted using stainless steel columns or a copper still pot (alembic)(Rosa *et al.* 2009; Bortoletto 2023). Copper still pot is the most common distillation equipment used for cachaça production because it is captured dimethyl sulphide during distillation, which generates sulphur off-flavours in the spirit (Rosa *et al.* 2009). All cachaças evaluated were distilled using copper still pots because all samples contain a small content of copper (Table 1).

Copper is the metal commonly used in the manufacture of still pots, as it has good thermal conduction and resistance to corrosion. In the past, cachaças from Paraíba tended to have high copper levels (>5 mg/L) (Serafim *et al.* 2016). Results reveal that among the 26 crystal cachaças evaluated, all had copper levels < 5 mg/L, except for samples CA-05, CA-07, CA-10, CA-12 and CA-19, which contained copper concentrations > 5 mg/L (Table 1). Although five cachaças contained copper concentrations > 5 mg/L, in the last 9 years, cachaças from Paraíba improved the copper control. Nowadays, the majority of producers apply a good practice for copper control (Table 1). The presence of copper in cachaça is associated with the use of copper stills and their hygiene. Copper salts are generated through the oxidation of the internal walls of the stills, and these are dissolved in the cachaça (Alcarde *et al.* 2014, Böck *et al.* 2022). To reduce the presence of copper, the internal walls of the stills must be cleaned before distillation (Oliveira *et al.* 2020, Böck *et al.* 2022). Moreover, during distillation, copper acts as a catalyser, and catalyses the reaction between alcohols and acetic acid generating acetate esters, and the reaction between short-and medium chain fatty acids and acetic acid generating fatty acid ethyl esters.

Volatile acidity (VA) is expressed in mg of acetic acid/100 mL because acetic acid is the main volatile acid present in cachaça (Da-Silva *et al.* 2023). Results indicate that sample CA-05 (146.1 mg/100 mL AA) contains the highest concentration of AV ($p<0.05$) (Table 1). In contrast, sample CA-06 (1.5 mg/100 mL) contains the lowest concentration of AV ($p<0.05$) (Table 1). Samples CA-04 (75.9 mg/100 mL) and CA-08 (77.5 mg/100 mL AA) presented similar values (Table 1). Samples CA-14 (37.4 mg/100 mL AA) and CA-17 (35.4 mg/100 mL AA) did not show significant differences between them (Table 1). Volatile acidity indicates the amount of acetic acid present in the cachaça, this acetic acid is generated during fermentation due to an accumulation of acetaldehyde (Ribeiro-Filho *et al.* 2021). Acetic acid (acetate) is a precursor of cytosolic acetyl-CoA and is important for maintaining the metabolic activity of yeasts and the generation of their secondary compounds (including esters) (Chen *et al.* 2013, Takahashi *et al.* 2006, Galdieri *et al.* 2014, Pietrocola, *et al.* 2015). Acetic acid is a volatile acid that is concentrated during the distillation of cachaça. During distillation acetic acid content can be observed into all three fractions including head, heart and tail; But, heads contains the lowest acetic acid content. The heart-tail separation limit directly influences the final concentration of acetic acid (volatile acidity). Volatile acidity is a important quality parameter because in high concentration, it can generate an unpleasant vinegar flavour. However, when present in low concentration, acetic acid can stimulate the senses and is a important precursors of esters during fermentation (when donates carbon to CoA generation Acetyl-CoA, which interacts to alcohols or short-and medium chain fatty acids) or distillation (when interacts to alcohols or short-and medium chain fatty acids) (Ribeiro-Filho *et al.* 2021).

321 Esters are important compounds, related to the sensory aroma characteristics of
 322 cachaças. The increase in levels of these compounds may occur as a result of storing
 323 cachaças in wooden barrels (Bortoletto and Alcarde, 2015; Bortoletto *et al.* 2018). Esters
 324 are formed mainly by esterification reactions of fatty acids with ethanol. Total esters (TE)
 325 are expressed as mg ethyl acetate/100 mL AA because ethyl ester is the most
 326 predominant ester in cachaças (Bortoletto and Alcarde, 2015, Bortoletto *et al.* 2016,
 327 Bortoletto *et al.* 2018). Results indicate that sample CA-05 (156.7 mg/100 mL AA) contains
 328 the highest concentration of TE ($p < 0.05$) (Table 1). In contrast, sample CS-01 (2.5 mg/100
 329 mL AA) contains the lowest TE concentration ($p < 0.05$) (Table 1). TE concentration drives
 330 to a sweet, fruity and flowery aromas, which generates a pleasant odour. Esters are
 331 generated during fermentation; however, distillation in copper stills contributes to increase
 332 the concentration of esters in the drink (Portugal *et al.* 2017).

333 Aldehydes are formed by oxidation reactions of alcohols, amino acids or fatty acids.
 334 Acetaldehyde contributes positively to the aroma of cachaças (Bortoletto and Alcarde
 335 2015, Ribeiro-Filho *et al.* 2021). Results indicate that sample CA-16 (18.1 mg/100 mL AA)
 336 contains a higher concentration of acetaldehyde ($p < 0.05$) (Table 1). In contrast, sample
 337 CA-13 (3.4 mg/100 mL AA) contains a lower concentration ($p < 0.05$) (Table 1). Samples
 338 CA-08 (3.6 mg/100 mL), CA-09 (3.7 mg/100 mL AA) and CA-13 (3.4 mg/100 mL AA)
 339 presented similar values (Table 1). Samples CA-10 (11.7 mg/100 mL AA) and CA-12 (11.4
 340 mg/100 mL AA) did not show significant differences between them (Table 1).
 341 Acetaldehyde, when present in low concentrations, leads to sweet, fruity aromas, but in
 342 high concentrations it leads to a pungent odour. Adequate cuts in the distillate fractions
 343 reduce the formation of this compound in high quantities.

344 The compounds furfural and hydroxymethylfurfural are produced by thermal degradation of
 345 sugars, dehydrating pentoses and hexoses (Bortoletto and Alcarde, 2015). Results
 346 indicate that sample CM-01 (1.2 mg/100 mL AA) contains the highest concentration
 347 ($p < 0.05$) (Table 1). In contrast, sample CS-02 (0.04 mg/100 mL) contains the lowest
 348 concentration ($p < 0.05$) (Table 1). The concentration of these compounds may be related
 349 to the burning of sugarcane, which, despite being prohibited, still occurs clandestinely in
 350 some regions to facilitate the burning of straw during manual harvesting. These
 351 compounds also come from the caramelization and Maillard reactions that occur during the
 352 toasting of wooden barrels (Bortoletto *et al.* 2016).

353 Higher alcohols are generated via the Ehrlich pathway, which occurs due to the
 354 consumption of amino acids (Ribeiro-Filho *et al.* 2021). In cachaça legislation, the limit of
 355 the concentration of higher alcohols is calculated by the sum of isobutyl (2-methyl-1-
 356 propanol,) isoamyl (2-methyl-1-butanol and 3-methyl-1-butanol) and n-propyl (1-propanol)
 357 alcohols (Da-Silva *et al.* 2023). Results indicate that sample CA-11 (346.7 mg/100 mL AA)
 358 contains the highest concentration ($p < 0.05$) (Table 1). In contrast, sample CA-19 (91.0
 359 mg/100 mL AA) contains a lower concentration ($p < 0.05$) (Table 1). All higher alcohol
 360 content identified was generated during fermentation and concentrated or separated
 361 during distillation. During fermentation, yeasts consumes amino acids as a nitrogen
 362 source, as a results, it generates ketones, aldehydes and higher alcohols (oxygen
 363 absence)(Ribeiro-Filho *et al.* 2022). Then, higher alcohols are esterified with acetyl-CoA to
 364 synthesize their respective esters or secreted into the wort (Hazelwood *et al.* 2008, Vidal
 365 *et al.* 2014). Fermented sugarcane must is an azeotropic mixture of ethanol and water (as
 366 the main compounds), which influences the volatility of other compounds (including higher
 367 alcohols). Higher alcohols such as propanol, isobutanol, amyl alcohols are miscible with
 368 water and highly soluble in ethanol, which indicates that higher alcohols tend to remain in
 369 the ethanol-rich phase affecting their volatility during distillation. A distilled spirit can be
 370 separated into three fractions (heads, heart, and tails). In the heads fraction typically
 371 contains more volatile compounds, including aldehydes (such as acetaldehyde) and some

higher alcohols (such as 1-propanol and 2-methyl-1-propanol), may be present in the heads fraction due to their volatility. The hearts fraction is the desired portion of the distillate that contains the majority of the flavour compounds, which contribute to the spirit flavour (Bortoletto and Alcarde 2015). The main higher alcohols (including 2-methylbutanol, 3-methylbutanol, and 2-phenylethanol), which generate fruity, malty, and floral notes, respectively, are often identified in the hearts fraction (proper Cachaça)(Bortoletto and Alcarde 2015, Bortoletto *et al.* 2016, Portugal *et al.* 2016). Although fermentation is the heart of distilled spirit production, distillers can ensure that the hearts fraction contains the desired higher alcohols, which can contribute to the spirit flavour profile by separating heads and tails, consequently, reducing the presence of unpleasant flavour.

The organic contaminants methanol, 1-butanol and 2-butanol present in cachaças can be formed during the fermentation and distillation stages (Bortoletto *et al.* 2016). During the fermentation, the pectin content present in the must can be metabolised by yeast, generating galacturonic acid, generating methanol (Bortoletto and Alcarde 2015). Results indicate that sample CA-19 (8.8 mg/100 mL AA) contains the highest concentration of methanol ($p < 0.05$) (Table 1). In contrast, sample CM-02 (0.26 mg/100 mL AA) contains a lowest methanol concentration ($p < 0.05$) (Table 1). The presence of methanol in the heart fraction of standardised commercial cachaças indicates that the sugarcane varieties contain a concentration of pectins that were transferred to the must during the extraction process and, as a consequence, generated the formation of methanol. Additionally, methanol is separated into the heads fraction during distillation, therefore, the methanol values found in the samples were low due to the separation of the head fraction.

The compounds such as n-butyl alcohols (1-butanol) and sec-butyl alcohols (2-butanol) are generated during resting, which is the period between fermentation and distillation. The importance of resting period is related to yeast decantation and fermented must stability. As a result, The generation of desirable flavour development, reducing harsh notes, removing undesirable compounds, increasing complexity, and consistency are improved in the final distilled spirit. In long resting period, n-butyl alcohols (1-butanol) and sec-butyl alcohols (2-butanol) are formed by the action of bacteria (Bortoletto *et al.* 2018). Results indicate that sample CA-13 (1.7 mg/100 mL AA) contains a higher concentration of 1-butanol ($p < 0.05$) (Table 1). In contrast, sample CA-19 (0.15 mg/100 mL AA) contains the lowest 1-butanol concentration ($p < 0.05$) (Table 1). The sample CA-06 (3.4 mg/100 mL AA) contains the highest concentration of 2-butanol ($p < 0.05$) (Table 1). In contrast, samples CA-02 (0.41 mg/100 mL AA), CA-17 (0.41 mg/100 mL AA) and CA-05 (0.42 mg/100 mL AA) contain the lowest concentrations of ($p < 0.05$) 2-butanol ($p < 0.05$) (Table 1). Therefore, some of producers need to evaluate their industrial practice considering the resting period because it is when n-butyl (1-butanol) and sec-butyl (2-butanol) are formed. But, when generated, they are no longer possible to be eliminated by the separation of the head, heart and tail fractions because they contain boiling points, respectively, 117.7 and 99.5°C, which keep them into the hearts fraction.

Acrolein (2-propenal) is a carcinogenic compound, considered an extremely mutagenic substance for humans and animals (Zhang and Schwab 2022). Its production occurs during fermentation due to bacterial contamination, or during fermentation under certain wort acids concentrations, which may activates some enzymes that may convert glycerol to acrolein or during distillation by dehydration of glycerol in the presence of acids on hot metallic surfaces. Acrolein is more volatile than ethanol and does not act like fusel alcohols, which can be difficult to separate; however, it is normally removed in the heads fraction separation (Alvarenga *et al.* 2023, Masson *et al.* 2012). All samples analysed did not present acrolein content (Table 1), which indicates that all cachaças evaluated were produced under a good fermentation and distillation practice. Acrolein is an undesirable

off-flavors and its presence generates unpleasant aroma such as peppery and horseradish-like (Masson *et al.* 2012, Rosa *et al.* 2009).

Ethyl carbamate is a carcinogenic compound, undesirable in distilled beverages. Occurring during fermentation, distillation and aging (Nóbrega *et al.* 2009, Alcarde *et al.* 2012; Machado *et al.* 2013, Bortoletto and Alcarde 2015, Mendonça *et al.* 2016, Santiago *et al.* 2017). Among the 26 crystal cachaças evaluated, all had ethyl carbamate values < 210 µg/L (Brazil 2022), except for sample CM-03 (419.4 µg/L), which presented the highest concentration ($p < 0.05$) (Table 1). Samples CA-14 and CA-16 did not show the presence of ethyl carbamate (Table 1). Ethyl carbamate is formed by the enzymatic degradation of cyanogenic glycosides present in sugar cane, involving the cyanide ion, which is oxidized to cyanate, which reacts with ethanol in the presence of copper ion, forming ethyl carbamate (Lachenmeier *et al.* 2010; Bortoletto and Alcarde, 2015). Adequate separation of the heads, hearts and tails fractions significantly reduces the levels of ethyl carbamate, as its highest concentration is found in the head fraction. It is also known that double distillation also reduces ethyl carbamate levels in cachaça (Nova *et al.* 2009, Alcarde *et al.* 2012; Rota *et al.* 2013, Bortoletto *et al.* 2016, Santiago *et al.* 2017).

439

440 **Aroma Profile by Compound Groups**

The aroma profile of cachaça is a key factor in sensory appeal, branding and marketing, and aroma quality. Furthermore, producers can create compelling marketing stories that improve the consumer experience by the differentiation of their products, and as a result, increase sales, and strengthen brand loyalty. Our data collection identified 85 volatile compounds (15 alcohols, 21 esters, 31 terpenes, 7 ketones and 11 aldehydes) (Fig. 2). Firstly, all compounds were grouped for facilitating the understanding of the aroma content of cachaças produced in different geographic origin (including mata mesoregion, agrest mesoregion and sertão mesoregion). Results revealed that cachaças produced in the mata mesoregion, agrest mesoregion and sertão mesoregion differ their aroma composition because mesoregions differ their edaphoclimatic conditions (soil and climate), field practice and industrial practice. Sample CA-02 contain the highest concentration of terpenes, alcohols and esters (Fig. 2). Samples CA-13, CA-20 and CM-01 contain high concentration of ketones, esters, alcohols and aldehydes (Fig. 2). On the other hand, samples such as CA-03, CA-04, CA-05, CA-09, CA-11, CA-12, CA-14, CA-15, and CA-19, contain the lowest concentrations of ketones, esters, alcohols, terpenes, and aldehydes (Fig. 2). Geographic origin inevitably results in variations that drive different flavour profiles and contribute to the complexity of Cachaça, which is influenced by a range of field and industrial variables.

A notable variations among samples produced within different mesoregion were observed. Results reveals that the aroma profile of cachaça (including terpenes, alcohols, esters, ketones, and aldehydes) changed when produced within different mesoregion (Fig. 2). In contrast, when produced within same mesoregion, the aroma profile of cachaça revealed similarities (Fig. 2). Our data disclosed that the main compounds groups, which changed the aroma profile of cachaça when produced within different mesoregion were alcohols, esters, ketones, and aldehydes because its generation are attributed to edaphoclimatic factors (including sugarcane genotypes adaptation, and fermentation vessel open) and industrial practice (Fig. 2). These compounds are mostly generated during fermentation via Ehrlich pathway thus ester synthesis (Ribeiro-Filho *et al.* 2021).

Furthermore, esters can be also synthesised during distillation and/or aging (Bortoletto and Alcarde 2013, Bortoletto *et al.* 2016). Moreover, terpenes composition come from the raw materials (sugarcane) that can be influenced by sugarcane genotype and edaphoclimatic

conditions. However, terpenes are significantly influenced by the storage methods used by producers prior to bottling because some producers conduct short-aged process using stainless steel tanks (inert) or wooden barrels (freijó or jequitibá)(Alcarde *et al.* 2014, Bortoletto 2023). When wooden barrels such as freijó or jequitibá are used for short-aged cachaças, all cachaça composition including acetic acid, glycerol, esters, higher alcohols, ketones, aldehydes, and terpenes are improved by providing a spirit with better sensorial characteristics without change the cachaça's colour.

Summary of All Cachaça Samples

Alcohols

The analysis of the main components for the alcohol profile of the cachaças explained 65% of the data variance, for the PC1 and PC2 axes they presented 38.5% and 26.5% of the explained variance, respectively (Fig. 3A). PC1 revealed that the alcohol profile of cachaças from Sertão mesoregion such as CS-01 and CS-02 differ from the alcohol profile of cachaças produced in the Mata mesoregion including CM-01 and CM-03. In contrast, CS-01 and CS-02 showed similarities with Mata mesoregion including CM-02 and CM-04 (Fig. 3A). PC2 revealed that cachaças from Mata and Sertão mesoregions differ from the alcohol profile of cachaça produced in Agreste mesoregion, except cachaça such as CA-14, CA-19, CA-17, CA-16, CA-02, CA-20 and CA-13 (Fig. 3A). Among the alcohols with important sensory characteristics, phenethyl alcohol and isoamyl alcohol (2-methyl-1-butanol + 3-methyl-1-butanol) were identified. Phenethyl alcohol directs characteristic alcoholic, flowery, honey-like, rose and sweet aromas. Isoamyl alcohol is a precursor to isoamyl acetate, which gives characteristics of fruity (banana), sweet, alcoholic, winey and pungent aromas (Portugal *et al.* 2016). Higher alcohols such as isobutyl (2-methyl-1-propanol) were identified in cachaças; however, its concentration was lower than 80 mg/L (Threshold), which does not generate problems. If it was identified above the mentioned concentration, it can have a negative sensory impact with an excessive alcohol sensation in addition to generating undesirable odours (including odours similar to solvents) (Portugal *et al.* 2016, Ribeiro-Filho *et al.* 2021). Cachaças produced in the agreste mesoregion contain low concentrations of isobutyl alcohol. Other higher alcohols such as n-propanol were not identified in cachaças from Paraíba due to the separation of the head fraction.

Esters

The analysis of the main components for the ester profile of cachaça (Fig. 3B) explained 60.2% of the data variance, for the PC1 and PC2 axes they presented 44.6% and 15.7% of the explained variance, respectively. PC1 revealed that the ester profile of twelve cachaças from the Agreste mesoregion such as CA-03, CA-05, CA-14, CA-09, CA-06, CA-01, CA-19, CA-01, CA-11, CA-12, CA-7, CA-04 plus two cachaças from the Sertão mesoregion such as CS-01 and CS-02 differ from other group of cachaças from the Agreste mesoregion such as CA-04, CA-10, CA-17, CA-08, CA-18, CA-20, CA-02, CA-13 plus cachaças from Mata mesoregion (including CM-01, CM-02, CM-03 and CM-04). PC2 revealed similarities between the cachaças from Mata mesoregions, except CM-02; and the Agreste mesoregion, except samples such as CA-04, CA-07, CA-11, CA-12, CA-15 and CA-16, which showed greater expression for the aromatic compound such as phenethyl acetate. This compound leads to fruity, floral, honey and sweet aromas (Fig. 3B). Cachaças from the Mata mesoregion and some from the Agreste mesoregions, contain fatty acid esters, which contribute to the aroma, flavour and viscosity of the drinks. A small part of these compounds are generated during fermentation, but their largest production is

connected to the distillation and aging stages (Bortoletto *et al.* 2018). As most of the cachaças analysed were rested in stainless steel tanks, it can be said that the majority of these compounds were formed during distillation. In contrast, some producers rest the crystal cachaças in freijó barrels. Resting/storing in wooden barrels for short periods of time does not influence the colour of the drink, but does influence the formation of these aromatic compounds (Bortoletto and Alcarde 2015).

Terpenes

The analysis of the main components for the terpene profile of the cachaças explained 66.2% of the data variance, for the PC1 and PC2 axes they presented 49.8% and 16.4% of the explained variance, respectively (Fig. 3C). PC1 revealed that difference among the terpene profile of cachaças from Sertão mesoregion and from Mata mesoregion, except CM-03, which showed similarities with the cachaças from Sertão mesoregion (Fig. 3C). A lower concentrations of terpenes in cachaças produced in the Sertão and Agreste mesoregions, except for the CA-02 sample, Brejo microregion, which presented high concentrations of terpenes that lead to woody and herbal aromas. PC2 revealed differences among the terpene profile of cachaças produced in the Sertão mesoregion and Mata mesoregion, except CM-02 (Fig. 3C). The terpene profile of sample CA-02 differ from all samples evaluated because contains the highest concentration of terpenes such as muurolene, epicubenol, and cadalene and gleenol (Fig. 3C), which presented greater expressiveness for aromatic compounds that direct floral, citrus fruity, floral and sweet and herbal aromas.

Before bottling, a short storage using freijó (*Cordia goeldiana*) or jequitibá wooden barrels are common practice for the cachaça producers because short aging these cited barrels are classified as neutral woods; therefore, aging in them improve the spirit sensorial characteristics without change its colour. The storage time of the booze, as well as the size and time of use of the barrels can influence the compounds generated in the drink, with greater or lesser extraction of some of these terpenes that are related to storage in wood. Terpenes such as α -muurolene and δ -elemene are commonly generated during resting or aging in wooden barrels such as Amburana cearenses and Cedrella fissilis (Guedes *et al.* 2022). The terpene nerolidol can be generated during resting or aging in chestnut (*Bertholletia excelsa*) and cabreúva (*Mycrocarpus frondosus*) barrels according to the type of toast to which the barrel was subjected. A wide variety of cachaças contain terpenes such as menthol, nerolidol, farnesol, 2,3 dihydrofarnesol, limonene, (E)- β -Farnesene and eugenol, which refer to floral, citrus fruity and spicy aromas (Oliveira *et al.* 2020, Santiago *et al.* 2016). Therefore, colourless cachaças can have their terpenes profile influenced by the geographic origin (soil and climate), field practice (sugarcane genotypes and its adaptation) and industrial practice (maturation).

Ketones

The analysis of the main components for the ketone profile of the cachaças (Fig. 3D) explained 64.5% of the data variance, for the PC1 and PC2 axes they presented 37.6% and 26.9% of the explained variance, respectively. PC1 revealed a lower concentration of ketones in cachaças produced in the Sertão mesoregion than cachaças produced in the Mata mesoregion (Fig. 3D). Dividing the cachaças from the Agreste mesoregion according to the concentrations of this compound. PC2 revealed difference between the cachaças from Sertão mesoregion and Mata mesoregion (Fig. 3D). Cachaça produced in the Agreste mesoregion showed differences among themselves (Fig. 3D). Ketones such as 2-heptone, 2-nonanone, and 2-undecanone were identified in all cachaças evaluated. These

compounds contribute to aroma-actives including grass, fruity, and floral (Zhang *et al.* 2020). Ketones are generated by the degradation of amino acids through microbial transamination reactions (Ehrlich pathway) (Zhang *et al.* 2023). Amino acids such as isoleucine, leucine, and valine undergo metabolism involving amino transferase, producing α -ketoic acids, which are then degraded into various compounds, including ketones (Zhang *et al.*, 2023, Zhang *et al.* 2020). This process generates specific flavour compounds, contributing to fruity and malty flavors in products.

577

578 **Aldehydes**

579 The analysis of the main components for the aldehyde profile of the cachaças (Fig. 3E) explained 70.8% of the data variance, for the PC1 and PC2 axes they presented 51.4% and 19.4% of the explained variance, respectively. PC1 revealed the sample CM-01, produced in the Mata mesoregion, contains a higher concentrations of aldehydes than cachaças produced in the mesoregion of Agreste and Sertão. PC2 revealed differences among cachaças from Sertão mesoregion and some cachaças from Mata and Agreste mesoregions. Aldehydes can be formed through oxidation of alcohols, Strecker oxidative degradation of amino acids and autoxidation of unsaturated fatty acids during fermentation and aging of the beverage (Escudero *et al.*, 2025, Bueno-Aventin *et al.*, 2021). Aldehydes with up to eight carbon atoms have an unpleasant aroma and those containing more than ten carbon atoms impart an unpleasant taste and aroma to beverages (Escudero *et al.*, 2025). Good manufacturing practices, fermentation control and adequate separation of head fractions are necessary to avoid the generation of high aldehyde content. Acetaldehyde is the main aldehyde present in cachaça, which generates a fruity and a herbaceous aroma; however, high levels produce a pungent and irritating aroma (Alcarde *et al.* 2014, Portugal, *et al.* 2017, Bortoletto *et al.* 2018).

595

596 **Overall**

597 The PCA score plot illustrates variations in the overall aroma profile of cachaças, highlighting key compounds such as higher alcohols, esters, ketones, aldehydes, and terpenes (Fig. 3F). The plot distinguishes four main groups based on similarities: Group 1 (CA-02), Group 2 (CA-13, CA-20, and CM-01), Group 3 (CA-18, CA-08, CM-03, CM-04, CA-17, CM-02, and CS-01), and Group 4 (CA-16, CA-10, CA-11, CA-19, CA-15, CA-07, CA-03, CA-12, CA-05, CA-06, CA-04, CS-02, CA-09, CA-14, and CA-01)(Fig. 3F). This grouping suggests that while certain samples share compositional characteristics, notable differences exist even among cachaças from the same mesoregion, reinforcing the complexity of cachaça terroir. Cachaças from different mesoregions (Agreste, Mata, and Sertão) exhibit distinct concentrations of volatile compounds. However, variations also arise within a single mesoregion due to microregional edaphoclimatic conditions and their local industrial practice. The Agreste mesoregion consists of nine microregions, with the Brejo Paraibano being the biggest producer of cachaça in Paraíba. It is important to mention that into Brejo Paraibano microregion, a municipality called Areia is also entitled "Capital Paraibana da cachaça", which has been recognised by the amount in volume and quality of cachaças produced. Our data showed that all cachaças from CPC showed high similarities, except sample CA-13 (Fig. 3F). Therefore, cachaças produced in municipality of Areia are differ from most of cachaças from Agreste (same mesoregion), Mata and Sertão mesoregions, which reinforces the fame obtained. Despite belonging to the same mesoregion, cachaças from different microregions display unique characteristics, emphasising the influence of geographic origin and local industrial practice. The PCA plot indicates that samples from the Agreste, Mata, and Sertão mesoregions do not form

completely separate clusters, suggesting overlapping compositional traits. This overlap may result from shared agricultural practices, similar industrial processes, or the fact that some producers use to buy raw material (sugarcane) from other mesoregions, or sugarcane genotype adaptations to comparable edaphoclimatic conditions. However, differences in soil composition, rainfall, and temperature across these mesoregions contribute to distinct metabolic pathways during sugarcane must fermentation, leading to variations in chemical composition of cachaça.

Edaphoclimatic factors, such as soil composition, nutrient availability, and climate conditions (temperature and rainfall), significantly affect sugarcane genotype selection, field management strategies, and industrial processing techniques (Medeiros-Silva et al. 2019, Carvalho and Furtado 2018). The presence of essential nutrients in the soil dictates fertilisation requirements, while insufficient rainfall necessitates irrigation, directly influencing sugarcane growth and biochemical composition. These environmental conditions shape not only the raw material but also the subsequent biochemical pathways active during fermentation. For instance, ketones, aldehydes, and higher alcohols are produced via the Ehrlich pathway, ultimately leading to ester formation (Ribeiro-Filho et al. 2021). The amino acid profile of sugarcane, strongly determined by soil and climate conditions, directly influences the types and concentrations of fermentation-derived aroma compounds (Hazelwood et al. 2008, Parish 1965). Field practices, shaped by these environmental constraints, in turn guide industrial decisions. In the Brejo Paraibano microregion, producers traditionally employ spontaneous fermentation in open vessels, fostering diverse microbial communities that contribute to unique and complex flavour profiles. In contrast, producers in the Mata and Sertão mesoregions often store colourless cachaça in neutral wooden barrels (jequitibá or freijó) for up to a year. This short-term maturation allows for the extraction and evolution of ketones, aldehydes, higher alcohols, terpenes, and esters, enhancing aromatic complexity and minimising differences that would otherwise be more pronounced relative to cachaças produced in the Agreste mesoregion.

The variations in chemical composition and aroma profiles are the result of a dynamic interplay between edaphoclimatic factors, field practices, and industrial practices (Capitello et al. 2021). This study contributes to a deeper understanding of how these interconnected elements—including environmental conditions, sugarcane genotype adaptation, fermentation processes, and ageing techniques—collectively shape the sensory and compositional attributes of cachaça. The observed similarities and differences across mesoregions, microregions, and even among samples from the same microregion highlight the complex interaction between natural and human-driven influences. Ultimately, this research reinforces the concept that cachaça's terroir is a multifactorial construct, shaped by the synergy between edaphoclimatic variability, agricultural decision-making, and industrial technique. These combined factors exert a decisive influence on the final chemical composition and sensory identity of the spirit. From an industrial standpoint, the regulatory framework governing cachaça production in Brazil is essential for maintaining product quality, safety, and market credibility. Adhering to these rigorous standards demonstrates producers' dedication to a genuine, high-quality cachaça. Compliance with established regulations minimises inconsistencies in quality, enhances consumer trust, and helps preserve the spirit's reputation nationally and internationally. Cachaça's aroma profile, which consists of 85 volatile compounds—including alcohols, esters, terpenes, ketones, and aldehydes—is influenced by a complex interaction between its geographical origin, field and industrial practice. Differences in soil composition, temperature, and precipitation across the Mata, Agreste, and Sertão mesoregions directly impact sugarcane genotype adaptation and cultivation techniques. These environmental factors, in conjunction with specific field practices and industrial processing methods such as

670 fermentation control, distillation cuts, and storage strategies, play a critical role in
 671 determining the chemical composition and shaping the final aroma and overall quality of
 672 cachaça.

673 Distinct production practices further contribute to the differentiation of cachaças among
 674 these mesoregions. Producers utilise maturation techniques such as resting cachaça in
 675 stainless steel tanks or ageing it in wooden barrels made of freijó or jequitibá to enhance
 676 flavour complexity and aroma development while maintaining its clear appearance
 677 (Alcarde et al. 2014, Santiago et al. 2017). These storage strategies reflect how
 678 edaphoclimatic conditions, in tandem with agricultural and industrial decisions, influence
 679 the final product. Consequently, cachaças from different Paraíba mesoregions exhibit
 680 unique production practices that significantly impact their sensory attributes and market
 681 appeal. Our findings contribute to a more comprehensive understanding of the
 682 characteristics and flavour profile of cachaça, offering valuable insights into consumer
 683 preferences and decision-making processes. Sensory attributes and intrinsic quality
 684 markers play a crucial role in brand perception, which, in turn, strongly influences
 685 perceived quality and purchase decisions. This study underscores the importance of
 686 maintaining and improving product quality standards while also demonstrating that
 687 geographic origin, together with cultivation and production strategies, influences perceived
 688 quality. Refining field and industrial practices in alignment with local environmental
 689 conditions can enhance product differentiation and foster greater consumer loyalty.

690 Furthermore, our findings provide deeper insights into the concept of cachaça geographic
 691 origin, which encompasses the interplay of environmental factors, soil composition,
 692 sugarcane genotype adaptation, fermentation, distillation, maturation, aging, and local
 693 traditions (Da-Silva et al. 2023). These elements, collectively influenced by the region's
 694 edaphoclimatic context and human intervention throughout the production chain, define
 695 the unique characteristics of cachaça and contribute to its geographical indication. In
 696 essence, the terroir of cachaça encapsulates the natural and human factors that shape its
 697 distinct identity and sense of place. Given its complexity, cachaça production requires a
 698 multidisciplinary approach. Future research should focus on: (1) developing aging
 699 protocols for different types of wood barrels and wood chips, (2) selecting yeasts from
 700 various geographic origins, (3) differentiating cachaças from similar geographic regions,
 701 (4) evaluating the contribution of regionally selected yeast strains to aroma and flavor
 702 development, (5) applying mid-infrared (MIR) and near-infrared (NIR) spectroscopy to
 703 optimize production processes such as distillation and aging, and (6) correlating MIR and
 704 NIR data with sensory analysis. These studies, combined with our findings, will provide
 705 more robust data that contribute to a refined understanding of cachaça terroir. This
 706 knowledge will support both consumers and producers in recognising and valuing the
 707 diversity and complexity of this iconic Brazilian spirit. Ultimately, these factors advocate for
 708 the unique sensory and quality attributes of cachaça, reinforcing the argument that
 709 cachaça possesses a distinct terroir. This concept is crucial in distinguishing its regional
 710 specialities and promoting its cultural and economic significance on a broader scale.

711

712 **Conclusion**

713 The compliance with legal standards for cachaças produced in the state of Paraíba was
 714 evaluated. Five producers must improve their industrial practices to ensure better copper
 715 control. Specifically, producer CM-03 should optimise the separation of the head fraction
 716 during distillation, while producers CA-05, CA-07, CA-10, CA-12, and CA-19 need to
 717 enhance the cleaning protocols of copper stills. Variations in the aroma profile are not only
 718 influenced by geographic origin but are also deeply shaped by the region's unique

edaphoclimatic conditions, field practices such as sugarcane management, and industrial decisions made during fermentation, distillation, and storage. Together, the origin and production know-how play a decisive role in shaping the sensory appeal and market differentiation of cachaça. In this study, 85 volatile compounds were identified in colourless cachaças from different mesoregions of Paraíba, revealing significant regional differences. Most cachaças from the Agreste mesoregion exhibit distinct aroma profiles compared to those from the Mata region. Meanwhile, cachaças from the Sertão share more similarities with those from Agreste, likely due to their industrial practice. These compositional differences are a result of the complex interplay between soil type, climate, sugarcane genotype adaptation, field management, and industrial techniques. Collectively, they contribute to the rich complexity and distinctive flavour profiles that characterise regional cachaças, influencing consumer preference and fostering brand loyalty.

CRedit authorship contribution statement

Vanessa Da-Silva: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – original draft. **Eike Torres:** Formal analysis, Methodology. **Leila Carvalho:** Formal analysis, Methodology. **Mércia Galvão:** Formal analysis, Methodology. **Marta Madruga:** Formal analysis. **Normando Ribeiro-Filho:** Conceptualization, Data curation, Formal analysis, Supervision, Investigation, Methodology, Software, Validation, Visualization, Writing – original draft, Writing - review & editing. **Taliana Bezerra:** Conceptualization, Supervision, Funding acquisition, Project administration, Writing - review & editing.

Declaration of Competing Interest

The authors declare there are no conflicts of interest.

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List of tables

Table 1. Legislation parameters for cachaças.

Sample	ABV (%)	Volatile acidity (acetic acid)*	Total esters (ethyl acetate)*	Total aldehydes (acetaldehyde)*	Sum of furfural and HMF*	Parameters by the legislation						Ethyl carbamate (µg/L)	Copper (mg/L)
						Sum of isobutyl, isoamyl and n-propyl alcohols*	Methanol*	1-butanol*	2-butanol*				
	38-48	150	200	30	5	360	20	3	10	210	5		
CA-01	43.0 ± 0.1 ^b	49.2 ± 0.8 ^k	30.50 ± 0.04 ^h	7.5 ± 0.02 ^{ij}	0.14 ± 0.03 ^{ijkl}	263.6 ± 0.3 ^{gh}	4.4 ± 0.03 ^{gh}	0.93 ± 0.01 ^{bcd}	0.66 ± 0.01 ^f	35.4 ± 4.8 ^{ijklm}	0.65 ± 0.01 ^{no}		
CA-02	44.0 ± 0.1 ^a	23.8 ± 0.8 ^r	14.8 ± 0.8 ^l	6.4 ± 0.2 ^{kl}	0.09 ± 0.01 ^{lmno}	195.8 ± 3.4 ^l	1.0 ± 0.2 ^l	0.46 ± 0.02 ^{efghi}	0.41 ± 0.01 ⁿ	72.0 ± 7.0 ^{fghi}	0.47 ± 0.02 ^{pq}		
CA-03	43.0 ± 0.2 ^b	108.4 ± 0.8 ^d	46.4 ± 0.03 ^f	4.8 ± 0.01 ^m	0.15 ± 0.01 ^{ijk}	241.7 ± 0.3 ^{ij}	3.5 ± 0.02 ^{ij}	0.59 ± 0.01 ^{defgh}	0.44 ± 0.01 ^{lm}	10.8 ± 6.2 ^{lm}	0.79 ± 0.02 ⁿ		
CA-04	38.0 ± 0.1 ^f	75.9 ± 2.4 ^{fg}	46.9 ± 0.2 ^f	4.3 ± 0.01 ^{nm}	0.21 ± 0.01 ^{gh}	236.5 ± 1.4 ^{ij}	5.6 ± 0.04 ^e	1.02 ± 0.03 ^b	0.55 ± 0.01 ^h	43.8 ± 5.8 ^{hijkl}	0.47 ± 0.01 ^{pq}		
CA-05	43.0 ± 0.2 ^b	146.1 ± 1.4 ^a	156.7 ± 0.5 ^a	7.4 ± 0.02 ^{ij}	0.19 ± 0.01 ^{ghi}	200.5 ± 0.2 ^l	3.3 ± 0.02 ^j	0.31 ± 0.01 ^{hi}	0.42 ± 0.01 ⁿ	29.5 ± 6.7 ^{klm}	8.25 ± 0.10 ^a		
CA-06	40.0 ± 0.1 ^d	1.5 ± 0.01 ⁿ	14.7 ± 0.1 ^l	8.7 ± 0.09 ^{gh}	0.12 ± 0.01 ^{ijklm}	289.3 ± 0.1 ^{ed}	5.9 ± 0.01 ^d	0.90 ± 0.02 ^{bcd}	3.40 ± 0.01 ^a	173.9 ± 5.6 ^{bc}	0.55 ± 0.03 ^{op}		
CA-07	40.0 ± 0.2 ^d	28.2 ± 0.01 ^{pq}	8.5 ± 0.1 ⁿ	9.9 ± 0.01 ^{ef}	0.15 ± 0.01 ^{ijk}	231.8 ± 0.4 ^{jk}	6.8 ± 0.01 ^b	0.98 ± 0.01 ^{bc}	0.44 ± 0.01 ^m	145.6 ± 28 ^{cd}	5.89 ± 0.11 ^e		
CA-08	39.0 ± 0.1 ^e	77.5 ± 2.6 ^{ef}	53.3 ± 0.2 ^c	3.6 ± 0.01 ^{no}	0.35 ± 0.01 ^e	221.7 ± 0.2 ^k	4.5 ± 0.03 ^{fg}	0.63 ± 0.01 ^{cdefgh}	0.46 ± 0.01 ^{ij}	78.3 ± 6.7 ^{fgh}	2.03 ± 0.06 ^l		
CA-09	40.0 ± 0.2 ^d	73.6 ± 0.9 ^g	27.1 ± 0.1 ⁱ	3.7 ± 0.01 ^{no}	0.17 ± 0.01 ^{hij}	242.2 ± 0.2 ^j	3.6 ± 0.03 ⁱ	0.72 ± 0.01 ^{bcd}	0.46 ± 0.01 ^{ijkl}	100.3 ± 3.3 ^{ef}	0.06 ± 0.02 ^r		
CA-10	40.0 ± 0.2 ^d	41.5 ± 0.01 ^l	19.8 ± 0.2 ^k	11.7 ± 0.02 ^d	0.70 ± 0.04 ^b	270.9 ± 0.2 ^{fg}	6.4 ± 0.07 ^c	0.67 ± 0.01 ^{bcd}	0.46 ± 0.01 ^{ijk}	194.3 ± 12.4 ^b	6.28 ± 0.07 ^d		
CA-11	39.0 ± 0.1 ^e	31.9 ± 0.01 ^{no}	21.5 ± 0.1 ^j	6.9 ± 0.02 ^{jk}	0.54 ± 0.01 ^c	346.1 ± 0.4 ^a	6.4 ± 0.03 ^c	0.48 ± 0.02 ^{efghi}	0.47 ± 0.01 ⁱ	67.0 ± 8.7 ^{fghij}	3.49 ± 0.03 ^h		
CA-12	38.0 ± 0.1 ^f	54.6 ± 0.01 ^j	41.4 ± 0.3 ^g	11.4 ± 0.06 ^d	0.05 ± 0.01 ^o	172.2 ± 0.3 ^m	2.7 ± 0.02 ^k	0.73 ± 0.03 ^{bcd}	0.46 ± 0.00 ^{ijkl}	61.4 ± 10 ^{ghij}	6.48 ± 0.06 ^c		
CA-13	40.0 ± 0.1 ^d	81.0 ± 0.9 ^e	30.50 ± 0.01 ^h	3.4 ± 0.01 ^o	0.12 ± 0.01 ^{ijklm}	262.4 ± 0.3 ^{gh}	4.2 ± 0.03 ^h	1.7 ± 0.55 ^a	1.12 ± 0.01 ^c	127.4 ± 5.7 ^{de}	2.39 ± 0.02 ^{ik}		
CA-14	38.0 ± 0.1 ^f	37.4 ± 0.01 ^m	11.60 ± 0.04 ^m	9.6 ± 0.01 ^{ef}	0.08 ± 0.01 ^{nmo}	233.5 ± 0.09 ^{ij}	3.6 ± 0.03 ⁱ	1.5 ± 0.15 ^a	0.45 ± 0.01 ^{klm}	n.d.	0.03 ± 0.01 ^r		
CA-15	38.0 ± 0.1 ^f	124.8 ± 1.6 ^b	57.8 ± 0.5 ^d	6.2 ± 0.02 ^{kl}	0.17 ± 0.01 ^{hij}	176.5 ± 1.1 ^m	4.7 ± 0.03 ^f	0.66 ± 0.04 ^{bcd}	0.75 ± 0.02 ^e	19.9 ± 6.5 ^{klm}	4.70 ± 0.02 ^f		
CA-16	40.0 ± 0.1 ^d	112.6 ± 1.5 ^c	62 ± 2 ^c	18.1 ± 0.6 ^a	0.10 ± 0.01 ^{klmno}	194.3 ± 0.6 ^l	0.7 ± 0.05 ^{lm}	0.34 ± 0.01 ^{ghij}	0.47 ± 0.01 ^{ij}	n.d.	4.26 ± 0.02 ^g		
CA-17	43.0 ± 0.2 ^b	35.4 ± 2.1 ^{nm}	6.70 ± 0.02 ^o	16.9 ± 0.04 ^b	0.13 ± 0.01 ^{ijklm}	301.1 ± 0.4 ^b	7.0 ± 0.4 ^b	0.59 ± 0.01 ^{defgh}	0.41 ± 0.01 ⁿ	51.8 ± 2.3 ^{ghijk}	2.62 ± 0.05 ⁱ		
CA-18	43.0 ± 0.2 ^b	67.5 ± 1.4 ^h	19.1 ± 0.1 ^k	12.8 ± 0.03 ^c	0.07 ± 0.02 ^{no}	243.7 ± 0.3 ⁱ	2.8 ± 0.03 ^k	0.65 ± 0.01 ^{cdefgh}	1.69 ± 0.01 ^b	85.3 ± 1.3 ^{fg}	1.67 ± 0.01 ^m		
CA-19	41.0 ± 0.1 ^c	112.3 ± 0.8 ^c	94.6 ± 0.5 ^b	4.8 ± 0.3 ^m	0.42 ± 0.01 ^d	91.0 ± 0.2 ⁿ	8.8 ± 0.05 ^a	0.15 ± 0.01 ⁱ	0.44 ± 0.01 ^{klm}	6.9 ± 6.4 ^{lm}	7.82 ± 0.01 ^b		
CA-20	39.0 ± 0.1 ^e	63.8 ± 1.5 ⁱ	42.6 ± 0.2 ^g	9.1 ± 0.2 ^{fg}	0.53 ± 0.01 ^c	277.1 ± 6.8 ^{ef}	0.8 ± 0.1 ^{lm}	0.68 ± 0.04 ^{bcd}	0.86 ± 0.01 ^d	143.7 ± 2.6 ^{cd}	2.51 ± 0.11 ^{ij}		
CM-01	44.0 ± 0.1 ^a	15.3 ± 0.8 ^t	3.80 ± 0.03 ^p	10.0 ± 0.07 ^e	1.20 ± 0.02 ^a	234.1 ± 1.1 ^{ij}	0.5 ± 0.01 ^{nm}	0.47 ± 0.01 ^{efghi}	0.46 ± 0.01 ^{ijk}	87.8 ± 11.5 ^{fg}	2.03 ± 0.01 ^l		
CM-02	43.0 ± 0.2 ^b	15.2 ± 0.01 ^t	2.9 ± 0.1 ^p	9.7 ± 0.9 ^{ef}	0.10 ± 0.01 ^{klmno}	255.5 ± 16.3 ^h	0.3 ± 0.04 ⁿ	0.68 ± 0.04 ^{bcd}	0.45 ± 0.01 ^{klm}	31.8 ± 9.8 ^{ijklm}	2.31 ± 0.01 ^k		
CM-03	39.0 ± 0.1 ^e	44.1 ± 0.01 ^l	15.6 ± 0.04 ^l	5.9 ± 0.03 ^l	0.29 ± 0.03 ^f	286.0 ± 0.06 ^{de}	0.7 ± 0.06 ^m	0.37 ± 0.03 ^{efghi}	0.64 ± 0.01 ^g	419.4 ± 45 ^a	2.04 ± 0.02 ^l		
CM-04	40.0 ± 0.1 ^d	31.6 ± 0.9 ^{pp}	3.50 ± 0.08 ^p	13.6 ± 0.2 ^c	0.24 ± 0.01 ^{fg}	284.1 ± 2.1 ^{de}	0.7 ± 0.07 ^m	0.37 ± 0.03 ^{fghi}	0.46 ± 0.01 ^{ijkl}	4.3 ± 1.7 ^m	0.62 ± 0.03 ^o		
CS-01	41.0 ± 0.1 ^c	19.3 ± 0.9 ^s	2.50 ± 0.07 ^p	7.9 ± 0.6 ^{hi}	0.08 ± 0.07 ^{nmo}	256.6 ± 4.1 ^h	0.3 ± 0.1 ⁿ	0.46 ± 0.03 ^{efghi}	0.45 ± 0.01 ^{klm}	9.3 ± 0.2 ^{lm}	0.55 ± 0.02 ^{op}		
CS-02	41.0 ± 0.1 ^c	27.5 ± 0.01 ^q	7.50 ± 0.02 ^{no}	9.6 ± 0.08 ^{ef}	0.04 ± 0.01 ^o	298.1 ± 0.3 ^{bc}	4.3 ± 0.02 ^{gh}	0.64 ± 0.03 ^{cdefgh}	0.44 ± 0.01 ^{klm}	89.7 ± 9.9 ^{efg}	0.33 ± 0.01 ^q		

*mg/100mL anhydrous alcohol; CA - Cachaças of Agreste; CM - Cachaças of Mata; CS - Cachaças of Sertão. different letters in the same column differ by tukey test (0.05). Acroelin data was not provided because in all samples evaluated, this compound was not identified.

List of Figures

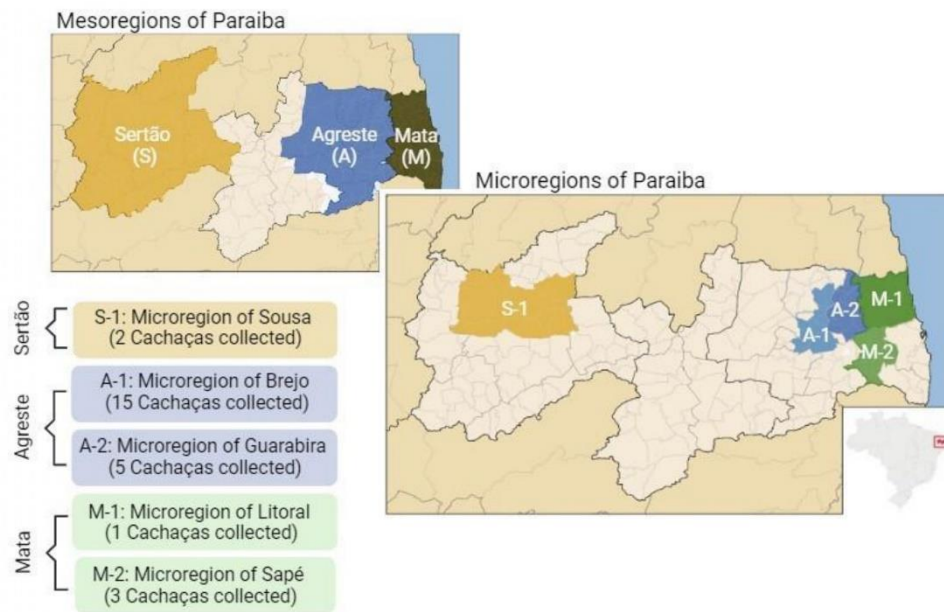


Figure 1: Mesoregions and microregions of the Paraíba state.

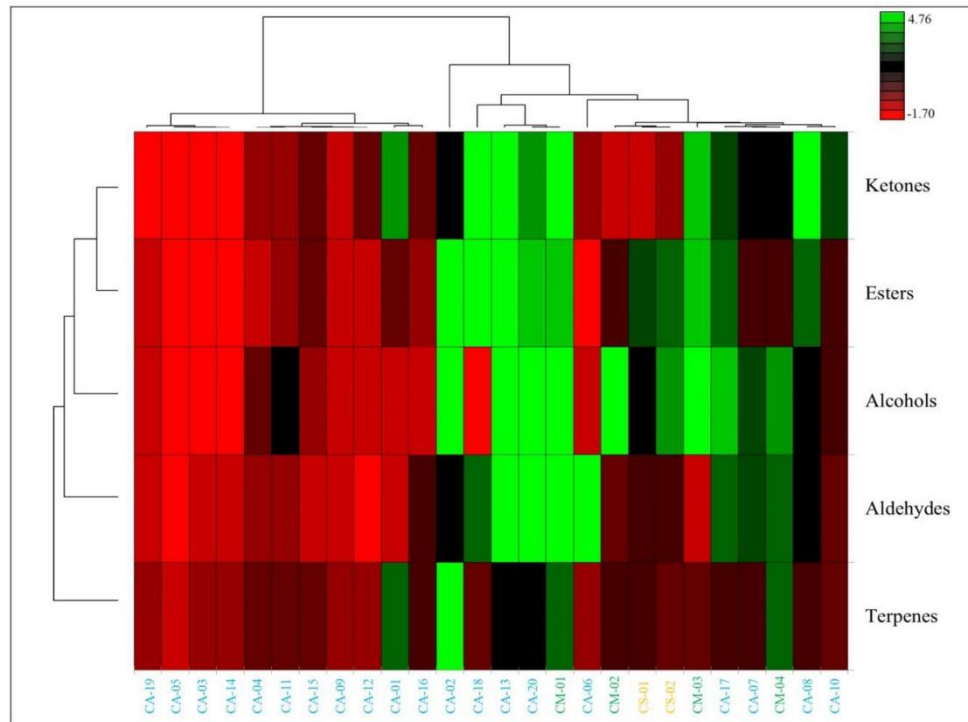


Figure 2: Heat map of groups of volatile compounds (including ketones, esters, alcohols, aldehydes and terpenes) measured in 26 samples of crystal cachaça produced in 3 different mesoregions of the state of Paraíba. CA – Cachaças do Agreste, CM – Cachaças da Mata; CS – Cachaças do Sertão.

Figure 3: Principal component analysis (Bi-Plot) of the profiles of volatile compounds present in cachaça including A) Alcohols, B) Esters, C) Terpenes, D) Ketones, E) Aldehydes, and F) PCA score plot illustrates variations in the overall aroma profile of cachaças. Twenty-six crystal cachaças produced in three different geographic regions were evaluated. CA – Cachaças do Agreste, CM – Cachaças da Mata; CS – Cachaças do Sertão. The compounds are represented by vectors and the cachaça samples are represented in black dots.

5.2 ARTIGO II – INFLUENCE OF AMBURANA CEARENSIS BARRELS ON THE SENSORY PROFILE AND COMPLEXITY OF CACHAÇA DURING MATURATION.

O artigo encontra-se submetido ao periódico *Wood Science and Technology*, sob o título *Influence of Amburana cearensis Barrels on the Sensory Profile and Complexity of Cachaça During Maturation*, desde o dia 20 de maio de 2025 (ANEXO B).

Influence of *Amburana cearensis* Barrels on the Sensory Profile and Complexity of Cachaça During Maturation.

Abstract

Cachaça is a traditional Brazilian distilled beverage produced from the fermentation and distillation of sugarcane juice. Its aging process, particularly in wooden barrels such as Amburana (*Amburana cearensis*), significantly alters its physicochemical and sensory characteristics. This study aimed to evaluate the quality and volatile composition of 21 samples of cachaça stored in Amburana barrels for periods ranging from four months to four years, collected from different mesoregions of the state of Paraíba, Brazil. The physicochemical parameters analyzed included alcohol content, volatile acidity, higher alcohols, aldehydes, esters, furfural, methanol, ethyl carbamate, and copper content. Additionally, the volatile aromatic profile was assessed via chromatographic analysis, and spectrophotometric methods were used to quantify total phenolic compounds, color intensity, and antioxidant activity. Statistical analysis involved ANOVA, Tukey's test and heat maps. Results demonstrated that all samples complied with the legal alcohol content range, though two showed volatile acidity above the regulatory limit, possibly due to inadequate hygiene during production. Significant variations were observed in the levels of ethyl acetate, acetaldehyde, higher alcohols, methanol, and furfural, influenced by factors such as fermentation control, distillation practices, storage time, and barrel conditions. Ethyl carbamate, a carcinogenic compound, exceeded the legal limit in 10 samples, particularly those aged for longer periods, emphasizing the importance of proper distillation cuts and hygiene. Copper content exceeded the legal threshold in three samples, indicating potential failures in equipment maintenance. Aromatic profiling identified 45 volatile compounds across the samples, including alcohols, esters, ketones, aldehydes, and terpenes. The most abundant compounds, such as 2-methyl-1-butanol, 3-methyl-1-butanol, phenylethyl

alcohol, and various ethyl esters, contributed to desirable sweet, floral, fruity, and woody aromas. The aromatic wheel and heat maps revealed diverse sensory profiles influenced by storage conditions and barrel reuse. Non-volatile analyses showed a wide range of total phenolic content (0.73–61.88 mg GAE/L), with a positive correlation between phenolics, color intensity, and antioxidant activity. In conclusion, the study revealed high variability in the chemical quality of cachaças aged in Amburana barrels, driven by differences in production and storage practices. These findings highlight the need for standardization of aging conditions, such as barrel reuse, storage time, and hygiene protocols, to ensure consistent quality and safety of stored cachaça. Furthermore, the aging process in Amburana wood contributes positively to the complexity of the drink, enriching it with desirable aromas that enhance its sensory profile.

Keywords: Volatile compounds, Aromatic profiling, Aging process, Distillation practices, Fermentation control.

Introduction

Cachaça is a Brazilian drink, with an alcohol content of between 38 and 48% by volume at 20°C, obtained from the distillation of the fermented must of sugar cane juice, and can also be aged in wooden barrels (Brasil, 2022). Aging aggregates value to the drink, as well as providing aromatic sensory characteristics from the compounds extracted from the wood used (Santiago et al., 2017; Souza et al., 2025).

Different woods are used for the ageing/storage of commercial cachaças, such as oak (*Quercus rubra*), amburana (*Amburana cearensis*), chestnut (*Bertholletia excelsa*) and balsam (*Sedum dendroideum*), which provide distinct chemical and sensory characteristics to the cachaça (Bortoletto et al., 2013).

Amburana (*Amburana cearensis*) is a native Brazilian wood that has been widely used in the aging of cachaças (Bortoletto et al., 2016), as it incorporates fatty acid esters and some phenolic compounds in its volatile profile, with a predominance of vanillic acid, coumarin and 4-methylumbelliferone (Santiago et al., 2016), which are important for the flavor and aroma of the drink, giving aroma markers of vanilla, cinnamon and other spices, as well as floral and fruity notes (Souza et al., 2025).

It is worth noting that aging/storage can also cause changes in the quality parameters of the drink. Studies have found changes in the congener coefficient, represented by the sum of total aldehydes (in acetaldehyde), total esters (in ethyl acetate), higher alcohols (1-propanol, iso-butanol and isoamyl), furfural and hydroxymethylfurfural and volatile acidity (in acetic acid) and in the carbamate content (Alcarde et al., 2014; Bortoletto et al., 2015; Souza et al., 2025).

The aforementioned changes are influenced by changes in the parameters of cachaça storage, such as the wood used, storage time, volume of the drink and barrel capacity. However, the Brazilian ordinance that regulates cachaça (Brasil, 2022) does not specify a minimum or

maximum storage time limit for “stored” cachaça, which means that cachaça processors use different storage times and barrel capacities, leading to a different quality of the final product to be sold.

The objective of this study was to verify and evaluate the quality and volatile chemical composition of cachaças stored in Amburana (*Amburana cearensis*) barrels, produced in different regions of the state of Paraíba, stored under different conditions, in order to obtain information that contributes to the standardization of stored cachaça, defining production requirements for a quality drink.

Material and Methods

Reagents and standards

The following standards were used: 1-butanol, 1-propanol, 2-butanol, 2-methyl-1-butanol, 2-methyl-1-propanol, 3-methyl-1-butanol, 3-pentanol, 5-HMF, acetaldehyde, acrolein, butyl carbamate, ethanol, ethyl acetate, ethyl carbamate, furfural and methanol, all with a purity of > 99%, for gas chromatography (GC) (Sigma-Aldrich, St. Louis, USA). A copper standard for AAS (Supelco) and Milli-Q ultrapure water (Millipore, Molsheim, France) were also used.

Sampling

A total of twenty-one samples of cachaça were stored in Amburana wood barrels (*Amburana cearensis*), four months to four years of storage (Supplementary table 1), collected from producers across three mesoregions of the state of Paraíba: 15 samples from Agreste Paraibano, 4 samples from Mata Paraibana, and 2 samples from Sertão Paraibano. Were produced through the fermentation of sugarcane juice, distilled in copper stills, and stored in Amburana woody barrels. The samples collected were filled into glass bottles and kept away from light and stored at 20°C. The analyses were carried out in triplicate.

Analytical methods

Determination of Alcohol by volume (ABV%)

The ethanol concentration in cachaça was determined using densimetry. A 250 mL sample was placed in a cylinder, and the alcohol content was directly measured using the Gay Lussac scale. The readings were corrected to 20°C, with measurements taken in triplicate (AOAC, 2000).

Volatile acidity

Volatile acidity was determined following the AOAC (2000) methodology. It was calculated by subtracting the fixed acidity from the total acidity of the sample. The result was expressed as grams of acetic acid per 100 mL of sample.

Higher alcohols, esters and aldehydes

The compounds: 1-butanol, 2-butanol, 1-propanol, acetaldehyde, acrolein, ethyl acetate, isoamyl alcohol, isobutyl alcohol, methanol and the sum of furfural and hydroxymethylfurfural were determined by direct sample injection into a Gas Chromatograph (Agilent, HP 6890), equipped with a Flame Ionization Detector (FID) and a Varian CP-Wax 52 CB capillary column (60m x 0.25mm x 0.25µm). The analytical conditions were based on the methodology outlined by Bortoletto et al. (2016).

The oven temperature program was as follows: an initial temperature of 40°C maintained for 4 minutes, followed by a ramp to 120°C at a rate of 20°C/min, an isothermal period of 1 minute, a ramp to 180°C at 30°C/min and a final maintenance period of 4 minutes. The injector and detector (FID) temperatures were set at 245°C and 250°C respectively.

The analyses were performed with a 1:25 split ratio, using helium as the carrier gas at a flow rate of 1.5 mL/min. Calibration curves and analytical standards were prepared using 40%

(v/v) ethanol. A 1.0 μL aliquot of each sample, along with 3-pentanol (internal standard), was directly injected for analysis.

Ethyl carbamate

Ethyl carbamate content was determined by direct sample injection into a Gas Chromatograph (GC) Agilent Technologies 7890B, coupled with a mass-selective detector (MSD) Agilent Technologies 5977B. The system was equipped with a capillary column, Agilent VF-WAXms (60m x 0.25mm x 0.25 μm), and connected to a Mass Spectrometer. The analysis followed the methodology outlined by Nóbrega et al. (2009).

The oven temperature program was as follows: an initial temperature of 90°C for 2 minutes, followed by a ramp of 10°C/min to 150°C (0 minutes), and then a ramp of 40°C/min to 230°C (10 minutes). The injector temperature was set to 250°C, and the GC/MS interface was maintained at 230°C. The mass spectrometer was operated in electron impact mode with an ionization energy of 70 eV. Helium was used as the carrier gas at a flow rate of 1.5 mL/min. The mass spectrometer detector operated in SIM mode (m/z 62).

The preparation of calibration curves and ethyl carbamate (EC) analysis was based on a methodology adapted from Serafim et al. (2016). Solutions of ethyl carbamate and butyl carbamate were prepared using 40% (v/v) ethanol. Butyl carbamate (150 $\mu\text{g/L}$) was used as the internal standard. A volume of 1.0 μL was injected in splitless mode. Ethyl carbamate was quantified using the internal standard addition method. The limits of detection and quantification for EC were 10 $\mu\text{g/L}$ and 40 $\mu\text{g/L}$, respectively.

Inorganic compound (Cooper, Cu)

The concentration of copper was determined by Flame Atomic Absorption Spectrometry (FAAS) using a Flame Atomic Absorption Spectrophotometer (AA-6300,

Shimadzu). A copper (Cu) hollow cathode lamp was used as the primary radiation source, with an electric current of 4 mA. An air/acetylene oxidizing flame (13.5 L/min for air and 2.0 L/min for acetylene) was employed. Copper was measured at 324.8 nm with a spectral bandwidth of 0.5 nm. The conditions followed the methodology outlined by Miranda et al. (2010). Calibration curves were prepared using 40% (v/v) ethanol.

Volatile profile

The concentration of volatile compounds was determined using a Gas Chromatograph (GC) Agilent Technologies 7890B, coupled with a mass-selective detector (MSD) Agilent Technologies 5977B. The system was equipped with an Agilent HP-5MS UI capillary column (30m x 0.25mm x 0.25 μ m) and connected to a Mass Spectrometer.

The following conditions, based on the methodology of Zacaroni et al. (2017), were used: oven temperature ramped from 35°C to 240°C at a rate of 4°C/min. The injector temperature was set to 270°C. Helium 5.0 was used as the carrier gas at a flow rate of 1.78 mL/min, with a 1:4 split injection mode. The temperature of the detector interface and the ion source remained at 240 °C and 200 °C, respectively. The mass spectrometer operated in electron impact mode (70 eV) and the mass scan range was from 50 to 400 mz^{-1} at 4.44 scan. s^{-1} . The extraction of volatiles was performed using the headspace solid-phase microextraction (HS-SPME) technique with an SPME device (Supelco, Bellefonte, USA). The sample was diluted to 10% ethanol using ultra-pure water. An aliquot of 4 mL, to which the internal standard 3-pentanol was added, was transferred to a 20 mL glass vial and immediately sealed with a Teflon-coated septum cap. The volatiles were extracted at 45°C with no equilibrium time, using a Divinylbenzene/Carboxene/Polydimethylsiloxane (DVB/CAR/PDMS) 50/30 μ m fiber, which was exposed to the headspace for 50 minutes to allow adsorption. Afterward, the fiber was exposed for 5 minutes for desorption in the gas chromatograph. The fiber was conditioned at

270°C for 60 minutes prior to the extraction procedure, in accordance with the manufacturer's specifications.

Compounds were identified using the NIST library database (2014), in combination with the mass spectrum and linear retention index (LRI). The LRI for each compound was calculated using the retention times of a homologous series of C6–C20 n-alkanes. Analyses were performed in triplicate, and the results were expressed as the total chromatographic peak area.

Spectrophotometric analysis

The color intensity of the samples was determined by directly measuring the absorbance at 420 nm, according to the methodology described by de Souza et al. (2025). The total content of phenolic compounds was quantified using spectrophotometry at 765 nm, after reaction with the Folin-Ciocalteu reagent, following the protocol of Amerine & Ough (1980). The analytical curve was constructed using gallic acid as a standard in a 40% (V/V) hydroalcoholic solution, and the results were expressed as mg of gallic acid equivalent per 100 mL of sample.

Antioxidant activity was assessed by the ability of the distillates to eliminate the DPPH radical, using UV-VIS spectrophotometry at 515 nm, according to the methodology of Brand-Williams et al. (1995). The calibration curve was drawn up with Trolox at concentrations ranging from 20 to 1200 μ M, and the results were present in terms of percentage inhibition of radical oxidation.

Statistical analysis

The analyses were carried out in triplicate and the results are presented as the mean and standard deviation of the three replicates. The data was submitted to Analysis of Variance (ANOVA) and Tukey's test (to compare the results of the cachaças analyzed in relation to the

parameters required by the legislation), with the averages showing $p < 0.05$ being considered statistically significant. Principal Component Analysis and Heat Mapping were also carried out for the results of the volatile compounds. The data was analyzed using XLSTAT software (version 2014.5.03, Addinsoft, New York, USA).

Results and Discussion

Physicochemical evaluation of cachaças stored in Amburana wood barrels (*Amburana cearensis*)

During aging in wooden barrels, there is a reduction in the alcohol content of cachaças, as well as a reduction in their initial volume (Silva et al., 2023), which is caused by the permeability and porosity of the wood, permitting the preferential evaporation of ethanol in relation to water (Alcarde et al., 2014). To obtain the final product with the required alcohol content, the producers start the storage process with a higher alcohol content, as well as adding unaged cachaça to the stored cachaça. Evaluating the cachaças in the present study, all the samples showed an alcohol content (ABV%) within the limit determined by the legislation.

Evaluating the volatile acidity (VA) of stored cachaças, it is observed that samples UA-12 (251.1 mg/100 mL) and UA-06 (173.1 mg/100 mL) contain the highest concentration of VA ($p < 0.05$) (Table 1). In contrast, sample UA-01 (10.4 mg/100 mL) contains the lowest concentration of VA ($p < 0.05$). Samples UA-08 (97.8 mg/100 mL), UA-09 (94.3 mg/100 mL), UA-10 (98.3 mg/100 mL) and CA-08 (93.2 mg/100 mL) showed similar values. Samples UA-15 (26.0 mg/100 mL), UM-01 (27.0 mg/100 mL) and UM-03 (25.7 mg/100 mL) did not differ significantly from each other (Table 1).

Volatile acidity (VA) is expressed in mg of acetic acid/100 mL because acetic acid is the main volatile acid present in cachaça (Brasil, 2022). In aged cachaças, the increase in acidity

is promoted by the oxidation reaction of ethanol to acetaldehyde and the consequent formation of acetic acid, as well as by the extraction of organic acids and phenolic compounds from the wood (Alcarde et al., 2014). Santiago et al. (2016) observed a gradual increase in the concentration of acidity in cachaças aged for 12 months.

On the other hand, higher volatile acidity values can also be representative of high levels of microbial contamination in the process, using inappropriate hygiene practices (Caetano et al., 2022). Of the 21 cachaças analyzed, two (UA-06 and UA-12) presented values higher than those permitted by Brazilian legislation. These samples had been stored for a maximum of 6 months (Supplementary Table 1), indicating that the increase in VA is due to the probable inadequate use of hygiene practices in the process.

Esters are produced by yeasts during fermentation through esterification reactions of acids and alcohols (Bortoletto et al., 2018), also occurring during storage of cachaças in wooden barrels (Bortoletto & Alcarde, 2015), with an increase in concentration as a result of ageing (Santiago et al., 2016). The Table 1 results indicate that sample UA-06 (68.5 mg/100 mL) contains a higher concentration of ethyl acetate ($p < 0.05$). In contrast, sample UM-03 (6.4 mg/100 mL) contains the lowest concentration ($p < 0.05$). Ethyl acetate is the ester with the highest concentration in cachaças (Bortoletto et al., 2016), giving a sweet and fruity aroma (Bortoletto & Alcarde, 2013), but at high concentrations (150-200 mg/100 mL) it produces disagreeable odors (Portugal et al., 2017).

Aldehydes are produced in cachaças during fermentation, formed by the oxidation of alcohols, amino acids or fatty acids (Ribeiro-Filho et al., 2021). The aging of the drink also favors the formation of these compounds (Bortoletto & Alcarde, 2013). Acetaldehyde is the main aldehyde associated with alcoholic fermentation (Moreira et al., 2012), which influences the aroma of cachaças, giving sweet and fruity aromas when in low concentrations; however, at higher levels it produces a strong and unpleasant aroma (Portugal et al., 2017). Careful

distillation cuts can help reduce the excessive presence of this compound (Bortoletto & Alcarde, 2015; Silva et al., 2020), which has higher levels in aged cachaças, due to the oxidation of ethanol during ageing (Serafim et al., 2013). The results indicate that sample UA-11 (16.4 mg/100 mL) contains a higher concentration of acetaldehyde ($p < 0.05$). In contrast, sample UA-09 (2.9 mg/100 mL) contains a lower concentration ($p < 0.05$) (Table 1).

Furfural and hydroxymethylfurfural are formed through the thermal degradation of sugars, involving the dehydration of pentoses and hexoses (Bortoletto & Alcarde, 2015). Results indicate that sample UA-06 (2,34 mg/100 mL) contains the highest concentration ($p < 0.05$) (Table 1). In contrast, sample UA-15 (0.08 mg/100 mL) contains a lower concentration ($p < 0.05$) (Table 1). The concentrations of furfural and hydroxymethylfurfural are within the maximum limit established by Brazilian legislation (Brasil, 2022). The concentration of these compounds may be related to the burning of the sugar cane, carried out to facilitate the burning of the straw (Bortoletto et al., 2016). These compounds can also be formed by pyrogenation of the organic matter during distillation. To prevent formation, it is necessary to control the final sugar content in fermentation (0° Brix) before starting distillation. When these compounds are formed in cachaça, most of them are present in the tail fraction and can be removed by properly separating the fractions (Bortoletto et al., 2018). The aging of cachaça can also result in an increase in these compounds from the caramelization and Maillard reactions that occur during the toasting of the wooden barrels (Alcarde et al., 2014).

The content of higher alcohols in alcoholic beverages is generated during fermentation and later concentrated or separated during distillation. Higher alcohols are formed through the Ehrlich pathway, which occurs as a result of amino acid consumption. (Ribeiro-Filho et al., 2021). The formation of these alcohols depends on several factors such as: the yeast strain used in fermentation and its activity; the level of bacterial contamination; control of the conditions during fermentation: pH of the must (≥ 4.0), temperature (28 °C to 32 °C), nitrogenous

compound content, the final alcohol content of the must; control of the time between fermentation and distillation; and the type of distillation used (Bortoletto & Alcarde, 2015, Bortoletto, et al., 2018).

The concentration of higher alcohols is calculated by the sum of isobutyl (2-methyl-1-propanol), isoamyl (2-methyl-1-butanol and 3-methyl-1-butanol) and n-propyl (1-propanol) alcohols (Brasil, 2022). These alcohols are important for the aromatic profile of cachaças, but when in excess they are negative for the drink (Bortoletto, et al., 2018). Results the Table 1, indicate that sample US-02 (331,6 mg/100 mL) contains the highest concentration ($p < 0.05$). In contrast, sample UA-12 (138,6 mg/100 mL) contains a lower concentration ($p < 0.05$). The low concentrations of these higher alcohols are related to the care taken in cutting the cane and the time needed for milling and fermentation (Moreira et al., 2012). During the storage of cachaças in Amburana barrels for a period of 12 months, Santiago et al. (2016) observed a gradual increase in the concentration of higher alcohols. Alcarde et al., (2014) also observed this increase in cachaças aged in oak barrels for 180 days.

Methanol, 1-butanol, 2-butanol and acrolein are compounds formed in cachaça, mainly during fermentation and are recovered by distillation (Silva et al., 2023), and are not influenced during the maturation of the drink (Souza et al., 2025). Methanol, 1-butanol, and 2-butanol, which are organic contaminants in cachaças, produced during the fermentation and distillation processes (Bortoletto et al., 2016).

The formation of methanol is due to sugar cane varieties containing concentrations of pectin that during the extraction process were transferred to the must, and during fermentation these pectic enzymes present are metabolized by the yeast, generating galacturonic acid and resulting in the production of methanol (Bortoletto & Alcarde, 2015). The highest concentration of methanol is contained in the head fraction, which can be removed by adequately separating the distillate fractions or double distillation (Bortoletto et al., 2018).

Ageing does not increase the concentration of this compound (Alcarde et al., 2010). Results indicate that sample UM-04 (6,26 mg/100 mL) contains the highest concentration of methanol ($p < 0.05$). In contrast, sample UA-06 (2,67 mg/100 mL) contains a lower concentration ($p < 0.05$) (Table 1) are within the maximum limit established by Brazilian legislation (Brasil, 2022).

N-butyl (1-butanol) and sec-butyl (2-butanol) alcohols are produced by the action of bacteria during a prolonged resting period between fermentation and distillation; it is not possible to remove them by separating the distillate fractions (Bortoletto et al., 2018). Low concentrations of these compounds are important for the quality of the drink (Portugal et al., 2017). The results in Table 1 indicate that sample US-02 (1.27 mg/100 mL) contains a higher concentration of 1-butanol ($p < 0.05$). In contrast, sample UA-10 (0.27 mg/100 mL) contains a lower concentration ($p < 0.05$).

For the compound 2-butanol, the results indicate that sample UA-01 (2.51 mg/100 mL) contains a higher concentration ($p < 0.05$). In contrast, sample UA-09 (0.41 mg/100 mL) contains a lower concentration ($p < 0.05$) (Table 1). Samples UA-15 (0.46 mg/100 mL) and (0.43 mg/100 mL), UA-08 (0.63 mg/100 mL) and (0.58 mg/100 mL), UA-13 (0.66 mg/100 mL) and (0.61 mg/100 mL), UA-14 (0.73 mg/100 mL) and (0.78 mg/100 mL), showed similar levels for both compounds 1-butanol and 2-butanol respectively. The compounds 1-butanol and 2-butanol were within the maximum limit established by Brazilian legislation for all the samples analyzed (Brasil, 2022).

Acrolein (2-propenal) is a carcinogenic compound, the production of which can occur during fermentation due to bacterial contamination, or under certain concentrations of must acids, which can activate some enzymes that can convert glycerol into acrolein; and also during distillation by dehydration of glycerol in the presence of acids on hot metal surfaces. It is an undesirable compound in beverages, producing unpleasant aromas, and can be removed during

the separation of the head fraction (Bortoletto et al., 2018). All the samples analyzed had no acrolein content.

Ethyl carbamate is the principal contaminant in cachaças because of its carcinogenic potential (Bortoletto et al., 2015). Is formed by the enzymatic degradation of cyanogenic glycosides present in sugar cane, involving the cyanide ion, which is oxidized to cyanate, which reacts with ethanol in the presence of copper ion, forming ethyl carbamate (Nóbrega, et al., 2009; Lachenmeier et al., 2010; Alcarde et al., 2014; Bortoletto & Alcarde, 2015).

Of the 21 samples stored in Amburana barrels, 11 had ethyl carbamate values < 210 µg/L (Brazil 2022), and 10 presented values above the legal limit. Samples UA-13 (637.0 µg/L), UM-04 (604.8 µg/L) and UA-06 (547.7 µg/L) showed the highest values for this compound (Table 1). It is an undesirable compound in distilled beverages, occurring during fermentation, distillation and ageing (Alcarde et al., 2012; Machado et al., 2013; Mendonça et al., 2016; Santiago et al., 2017).

During storage, a significant increase in ethyl carbamate is observed in cachaças aged, but these higher concentrations also depend on the initial level of this compound formed in the drink, since proper separation during distillation, with correct removal of the head fraction; as well as double distillation reduce the initial levels of this compound in cachaça (Nova et al., 2009; Alcarde et al., 2012; Rota et al., 2013; Alcarde et al., 2014; Bortoletto et al., 2016; Santiago et al., 2017). These factors may have contributed to the high concentration of this compound in sample UA-13, which was stored in Amburana barrels for a period of more than 4 years (Supplementary table 1). Alcarde et al., (2014) observed a gradual increase in the concentration of ethyl carbamate in cachaças stored for 180 days in oak barrels.

Copper is the metal commonly used in the manufacture of still pots, as it has good thermal conduction and resistance to corrosion. The use of copper stills confers a higher sensory quality to cachaça, but the presence of high levels of copper is dangerous for human health

(Böck et al., 2022). All the cachaças evaluated were distilled in copper stills. Results in the Table 1 reveal that among the 21 cachaças stored in Amburana barrels evaluated, all had copper levels < 5 mg/L (Brasil, 2022), except for samples UA-06 (5,68 mg/L), UA-07 (6,94 mg/L) and UM-02 (5,42 mg/L).

The presence of copper in cachaça is associated with the use of copper stills for distilling cachaça. Failure to sanitize these stills before distillation results in oxidation of the internal walls, generating copper salts that are dissolved in the cachaça. During ageing in wooden barrels, the concentration of copper remains controlled due to the absorption of the metal by the wood (Alcarde et al., 2014, Bortoletto et al., 2018).

Profile of volatile compounds of cachaça stored in Amburana barrels

Were identified 45 volatile compounds identified in the 21 samples of cachaças stored in Amburana barrels. The main classes of compounds identified include alcohols (7), esters (14), ketones (4), aldehydes (4) and terpenes (16). Each sample had a different profile in terms of the number of compounds identified and their concentrations (Supplementary table 2).

Aromatic wheel of cachaças stored in Amburana barrels

The sensory characteristics associated with volatile compounds present in cachaças stored in wooden barrels are of major interest because they influence how the drink will be perceived sensorially by the consumer, providing aroma and flavor characteristics, as well as quality.

The volatile compounds described in Supplementary Table 2 and their related aroma descriptors were considered for the construction of the wheel of aromatic volatile compounds and their aroma descriptors produced by Paraíba cachaças stored in Amburana barrels (Figure 1), grouping the main volatile compounds found in cachaça stored in an Amburana barrel based

on their sensory contributions, connecting them to aroma descriptors. Each compound identified (by chromatography) is responsible for unique olfactory stimuli perceived as characteristic aromas.

By evaluating the aromatic wheel that characterizes the substances found in in cachaça stored in an amburana barrel, it is possible to observe that the wheel is divided into a broad category of aromas represented by sensory groups. It stands out for its sweet, floral, fruity and woody aromas, with hints of coconut, honey, rose and a fatty aroma.

The compounds 2-methyl-1-butanol and 3-methyl-1-butanol were the most abundant in all the samples, contributing sweet, fruity and alcoholic aroma descriptors, followed by phenylethyl alcohol described with sweet, honey, flowery and rose aromas, and 1-octanol which is described as oily and coconutty.

Fatty acid esters from ageing are formed during the oxidation phase of the compounds extracted from the wood. These esters influence the aroma of the drink. Various esters are described with fruity aromas, such as ethyl acetate, ethyl butanoate, ethyl hexanoate, ethyl octanoate, ethyl nonanoate, ethyl decanoate, isoamyl acetate and isoamyl octanoate. Another important ester is phenethyl acetate, described as a flowery, rose, sweet and honey aroma. Ethyl laurate, ethyl palmitate and ethyl linoleate with oil and fatty descriptors, contribute to the body and viscosity of the drink.

Terpenes also influenced the composition of the aromas, including farnesol, nerolidol, citronellol and α -curcumene, bringing flowery, herbaceous and woody aromatic descriptors.

According to the profile of aromatic volatile compounds produced by Paraíba cachaças stored in Amburana barrels, they can be generally characterized as a drink with sweet, flowery and fruity aromas with woody and herbaceous touches, which has body and viscosity resulting from the presence of oily and fatty aromas.

However, the final aroma of the drink can be influenced by various edaphoclimatic and production factors, as well as storage and ageing conditions.

Aromatic profile of a cachaças stored in Amburana barrels

A heatmap correlating the cachaças collected and the aromatic compounds distributed by class can be seen in Figure 2 (A-D). The compounds in higher concentrations were grouped in dark red, and those in dark blue showed lower concentrations.

The heat map for the volatile alcohol profile (Figure 2A) reveals 7 aromatic compounds identified. Among the alcohols with the highest concentration in the samples, the following stand out: 3-Methyl-1-butanol and 2-Methyl-1-butanol, these isomeric amyl alcohols form isoamyl alcohol, which is a precursor to isoamyl acetate (Portugal et al., 2016). These compounds give the drink alcoholic, sweet and fruity aromas (Figure 1).

Among the alcohols with important sensory characteristics that have a positive impact on beverages, phenethyl alcohol stands out, producing characteristic aromas of flowers, honey, roses and sweets (Figure 1). In evaluating the alternative maturation of cachaças with Amburana, Souza et al. (2025) observed the presence of this compound as a potential marker. Lower concentrations of phenethyl alcohol in cachaças stored in Amburana barrels can be considered one of the factors indicating the higher number of uses of this barrel, with a consequent reduction in the extraction of this compound. Samples UA-01, UA-03, UA-06, UA-07, UA-08, UA-12, UM-03, UM-04 and US-02 (Figure 2A) had the lowest concentrations of this compound. Other alcohols include 2-Heptanol, 1-Octanol, 2-Nonanol and 2-Undecanol (Figure 2A), which are present in some of the samples, but also in low concentrations. These compounds are associated with citrus, coconut, fruit and herbaceous aromas.

Esters are compounds formed during fermentation, distillation, and ageing in cachaças (Silva et al., 2023). In the cachaças stored in Amburana barrels analyzed, 14 compounds were identified (Figure 2B), including esters and fatty acid esters.

Fatty acid esters contribute positively to the viscosity of the drink, giving it body, but high concentrations contribute negatively, as they are reminiscent of fat (Souza et al., 2025). Ethyl hexanoate, ethyl octanoate and ethyl heptanoate showed high concentrations in all the samples analyzed, making a positive contribution by imparting sweet and fruity aromas to the drinks. Another ester that showed greater expression was phenethyl acetate, whose precursor is phenylethyl alcohol (Ribeiro-Filho et al., 2021), which leads to fruity, floral, rose, honey and sweet aromas (Figure 1). Cachaças stored for up to 12 months in Amburana barrels showed a gradual increase in the concentration of esters, which are responsible for imparting fruity aromas to the drink (Santiago et al., 2016).

Figure 2C illustrates the profile of aldehyde and ketone volatile compounds. We identified 4 aldehydes and 4 ketones formed in the cachaças stored in Amburana barrels. Aldehydes with up to eight carbon atoms have an unpleasant aroma and those containing more than ten carbon atoms impart an unpleasant taste and aroma to beverages. Good manufacturing practices, fermentation control and adequate separation of head fractions are necessary to avoid the production of high concentrations of these compounds that interfere with the aromatic profile of the drink (Bortoletto et al., 2018; Alcarde et al., 2014). The aldehydes hexanal, nonanal, decanal and undecanal were identified in the cachaças stored in Amburana (Figure 2C). These compounds had low concentrations or were not present at all in the samples evaluated. They are compounds whose aromatic profile is reminiscent of sweet, floral, herbaceous, fruity and citrus. Among the ketones identified (Figure 2C), the β -Damascenone compound stands out, as it was present in all the samples analyzed, and in concentrations responsible for producing sweet and honey aromas (Figure 1).

A total of 16 terpenes were identified (Figure 4D) in the cachaças stored in Amburana barrels. The storage time of the drink, as well as the size and time of use of the barrels, can influence the compounds generated in the drink, with greater or lesser extraction of some of these terpenes that are related to storage in wood.

Storage in Amburana barrels gives the drink aromatic compounds specific to this wood: β -Curcumene is associated with woody aromas, while Nerolidol contributes floral and fruity aromas (Souza et al., 2025). Terpenes such as α -muurolene and δ -elemene are commonly generated during resting or aging in wooden barrels such as Amburana (Guedes et al., 2022; Bortoletto et al., 2021), contributing herbaceous and woody aromas (Figure 1).

Souza et al. (2025), during the maturation of cachaças using Amburana chips, observed that the reuse of the chips altered the structure of the compounds formed, as is the case with the β -Curcumene and trans-Calamenene compounds present in the first use and which were converted to their α -Curcumene and Calamenene isomers during reuse. This can also be explained for the cachaças stored in Amburana barrels analyzed, whose concentration of compounds produced or not produced may be related to the amount of reuse of these barrels, even generating different compounds depending on the amount of reuse.

Non-volatile characterization of cachaças

Figure 3 (A-C) and Supplementary Table 3 show the results of the non-volatile characterization of the 21 samples of cachaças stored in Amburana cearensis barrels, taking into account the content of total phenolic compounds (Figure 3A), color intensity (Figure 3B) and antioxidant activity (Figure 3C). The behavior of the cachaças was similar for all the analyses carried out. In general, a similar pattern was observed between the variables analyzed, with samples that had a higher concentration of phenolic compounds tending to exhibit greater color

intensity and antioxidant activity. Each region sampled had a standout drink: in the Sertão, sample US-02; in the Agreste, sample UA-09; and in the Mata region, sample UM-03.

The total phenolic compounds (TPC) of the cachaças ranged from 0.73 to 61.88 mg GAE/L, showing a wide variability, even between drinks stored in barrels of the same plant species and from the same geographical region. This suggests that other factors have a greater influence on the extraction and formation of these compounds, such as the length of time the cachaça has been stored, the degree of toasting of the barrels, the number of times the barrel has been reused, the ratio between the volume of distillate and the volume of the container, the formation of blends and the alcohol content of the stored drink (Silva et al., 2023; Ratkovich et al., 2023).

The results obtained for TPC are within the range reported by Bortoletto & Alcarde (2013), who evaluated brandies stored in barrels made from ten different types of wood and found concentrations of phenolic compounds ranging from 7 to 54.9 mg GAE/L. *Amburana cearensis* wood stood out for its higher phenolic content (54.9 mg GAE/L). In the present study, only sample US-02 had a higher phenolic content than that reported by Bortoletto & Alcarde (2013), possibly due to the specific production conditions, considering that commercial drinks can be stored in barrels with different degrees of toasting, times of use and number of reuses.

On the other hand, it is understood that the higher the concentration of phenolic compounds, the greater the formation of visible color, measured by the spectral region of the 420 to 495 nm range, characterized by the formation of chromophores that will confer, in addition to color intensity, aspects of yellow color and high saturation, as pointed out by Souza et al. (2025).

However, this behavior was not strictly linear. For example, sample UA-09 had the highest content of phenolic compounds among the Agreste cachaças, but sample UA-12 exhibited greater color intensity (Figure 3 B). This phenomenon can be explained by the

presence of different classes of phenolic compounds, as well as the contribution of other compounds, especially furfurals, which play a significant role in intensifying color. In addition, from the point of view of industrial production, Brazilian legislation (Brasil, 2022) allows the addition of colorants to aged cachaças, making it possible for some commercial drinks to have more intense coloring without necessarily containing higher levels of phenolic compounds.

With regard to antioxidant activity (Figure 3 C), samples US-02, UA-09 and UM-03 showed the highest percentages of DPPH radical inhibition, with values of 35.66%, 23.77% and 16.86%, respectively, while the other samples showed low antioxidant potential. The Sertão sample (US-02) was the only one to show antioxidant activity of over 30%, which can be attributed to its high content of total phenolic compounds.

In comparison, Souza et al. (2025) reported antioxidant activity of approximately 20% for brandy stored with *Amburana cearensis* chips, a value lower than that found for the UA-09 and US-02 samples in this study, despite the fact that they had lower concentrations of total phenolic compounds (~70 mg GAE/L). This behavior reinforces the idea that antioxidant activity is not only related to the total content of phenolic compounds, but also to the specific chemical structure of these compounds and their ability to donate electrons or hydrogen.

Higher antioxidant activity values can contribute to greater oxidative stability of the drink, helping to preserve its physicochemical and sensory characteristics over time (Yan et al., 2023). Thus, in addition to the sensory influence, aging distillates in wooden barrels also provides bioactive compounds that can slow down oxidative processes and, consequently, increase the stability of the final product.

Conclusion

A wide variability in the levels of compounds such as esters, aldehydes, higher alcohols, furfural and methanol was observed in the samples analyzed, reflecting differences in production practices, storage time and the conditions of the barrels used.

The cachaças stored for longer generally had higher concentrations of ethyl carbamate, exceeding the limits allowed by Brazilian legislation in almost half of the samples, highlighting the importance of the quality of the sugar cane, strict control in distillation and separation of the fractions, as well as storage time.

Analysis of the volatile profile revealed the presence of 45 compounds, especially higher alcohols, esters and terpenes responsible for the sweet, fruity, floral and woody aromas characteristic of Amburana wood. The diversity and intensity of the aromatic compounds also showed variations related to the number of barrels reused and the ageing time, with a direct impact on the sensory perception of the final product, indicating that undetermined barrel reuse can compromise the desired aromatic profile. Cachaças with a higher content of phenolic compounds generally showed greater color intensity and antioxidant activity.

The data obtained reinforces the need for more detailed regulations regarding the minimum time and storage conditions for cachaças classified as “stored”. Regulating these criteria could help guarantee the safety, quality and sensory identity of the final product, as well as enhancing the value of the drink.

Declaration of competing interest

The authors declare there are no conflicts of interest.

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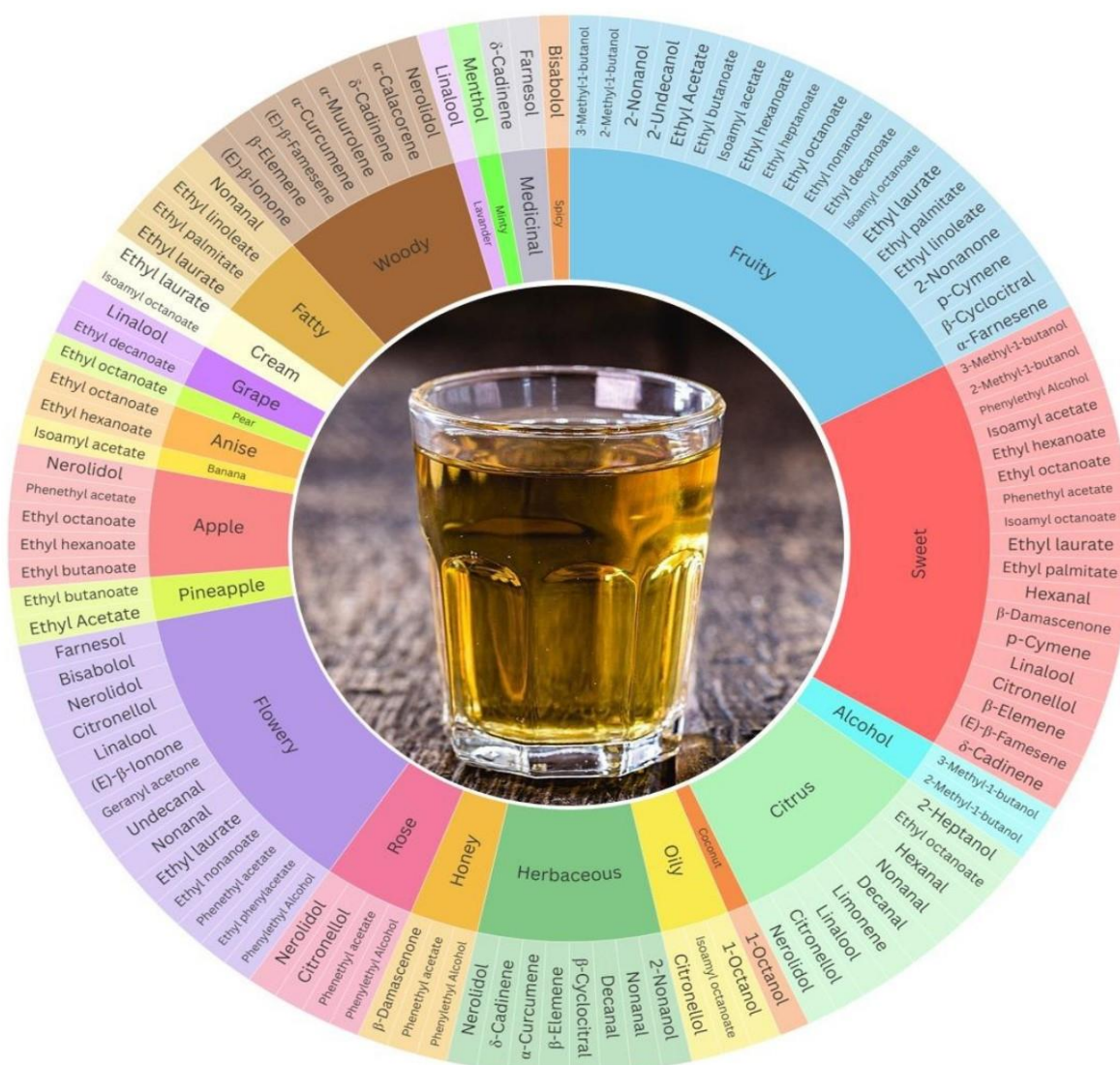
Table 1. Quality control parameters of cachaças stored barrel woody *Amburana (Amburana cearensis)*.

Sample	Legal parameters											
	ABV (%) v/v	Volatile acidity (acetic acid)*	Total esters (ethyl acetate)*	Total aldehydes (acetaldehyde)*	Sum of furfural and HMF*	Sum of isobutyl, isoamyl and n- propyl alcohols*	Coeficiente de congêneres*	Methanol*	1-butanol*	2-butanol*	Ethyl carbamate (µg/L)	Copper (mg/L)
	38-48	150	200	30	5	360	200-650	20	3	10	210	5
UA-01	40.0 ± 0.1 ^d	10.4 ± 0.01 ⁿ	24.4 ± 0.1 ^k	6.9 ± 0.06 ⁱ	0.11 ± 0.01 ^{ij}	263.3 ± 0.1 ^e	305.1 ± 0.2 ⁿ	5.17 ± 0.03 ^{de}	0.78 ± 0.02 ^{de}	2.51 ± 0.01 ^a	152.8 ± 10.2 ^{fg}	0.46 ± 0.02 ^{gh}
UA-02	40.0 ± 0.2 ^d	56.3 ± 0.01 ^k	15.3 ± 0.02 ^l	4.7 ± 0.02 ^l	0.11 ± 0.01 ^{ij}	240.9 ± 0.6 ^{hi}	317.3 ± 0.6 ^m	4.74 ± 0.04 ^g	0.77 ± 0.01 ^{def}	0.49 ± 0.01 ⁱ	216.2 ± 6.7 ^e	1.00 ± 0.09 ^{fgh}
UA-03	40.0 ± 0.1 ^d	74.1 ± 0.01 ^h	28.9 ± 0.05 ^j	3.6 ± 0.02 ^p	0.29 ± 0.01 ^e	229.5 ± 0.8 ^j	336.4 ± 0.7 ^l	4.48 ± 0.04 ^h	0.91 ± 0.01 ^{bc}	1.19 ± 0.01 ^b	220.4 ± 7.3 ^e	0.55 ± 0.1 ^{gh}
UA-04	39.0 ± 0.1 ^e	86.1 ± 0.9 ^f	23.8 ± 0.3 ^k	3.2 ± 0.03 ^q	0.16 ± 0.01 ^{ghi}	208.2 ± 1.6 ^l	321.4 ± 1.8 ^m	3.83 ± 0.02 ^j	0.66 ± 0.01 ^{gh}	0.47 ± 0.01 ^{jk}	155.0 ± 4.6 ^{fg}	0.02 ± 0.01 ^h
UA-05	39.0 ± 0.2 ^e	67.4 ± 0.9 ⁱ	36.5 ± 0.5 ^e	7.8 ± 0.02 ^h	0.91 ± 0.01 ^c	248.2 ± 0.1 ^g	360.8 ± 1.1 ^{hi}	6.00 ± 0.1 ^b	0.78 ± 0.01 ^{de}	0.47 ± 0.01 ^{jk}	224.9 ± 12.0 ^e	0.06 ± 0.02 ^h
UA-06	38.0 ± 0.2 ^f	173.1 ± 1.6 ^b	68.5 ± 0.2 ^a	3.9 ± 0.01 ^o	2.34 ± 0.07 ^a	159.7 ± 0.03 ⁿ	407.4 ± 1.6 ^c	5.55 ± 0.04 ^c	1.00 ± 0.03 ^b	0.49 ± 0.01 ⁱ	547.7 ± 18.4 ^c	5.68 ± 0.3 ^b
UA-07	40.0 ± 0.2 ^d	59.3 ± 0.01 ^{jk}	35.0 ± 0.3 ^f	9.2 ± 0.01 ^e	0.15 ± 0.01 ^{hi}	178.1 ± 0.4 ^m	281.6 ± 0.1 ^o	2.67 ± 0.06 ^l	0.64 ± 0.01 ^{gh}	0.47 ± 0.01 ^j	68.0 ± 5.3 ^h	6.94 ± 0.4 ^a
UA-08	39.0 ± 0.3 ^e	97.8 ± 0.9 ^d	35.2 ± 0.06 ^f	4.2 ± 0.01 ⁿ	0.13 ± 0.01 ^{ij}	244.1 ± 0.2 ^{gh}	381.4 ± 1.0 ^{fg}	4.06 ± 0.02 ⁱ	0.63 ± 0.01 ^{gh}	0.58 ± 0.01 ^g	292.7 ± 4.2 ^d	2.99 ± 0.2 ^{cd}
UA-09	40.0 ± 0.2 ^d	94.3 ± 2.3 ^e	31.1 ± 0.2 ⁱ	2.9 ± 0.02 ^r	0.13 ± 0.01 ^{ij}	249.3 ± 1.0 ^g	377.7 ± 2.9 ^g	3.77 ± 0.04 ^j	0.59 ± 0.03 ^h	1.02 ± 0.01 ^c	170.2 ± 14.3 ^f	1.41 ± 0.07 ^{efg}
UA-10	43.0 ± 0.1 ^a	98.3 ± 0.8 ^d	36.6 ± 0.2 ^e	5.4 ± 0.01 ^j	0.14 ± 0.01 ^{ij}	179.5 ± 0.09 ^m	320.0 ± 0.7 ^m	3.27 ± 0.01 ^k	0.27 ± 0.02 ^j	0.41 ± 0.01 ⁿ	74.3 ± 1.5 ^h	3.04 ± 0.05 ^{cd}
UA-11	42.0 ± 0.2 ^b	60.7 ± 1.4 ^j	33.4 ± 0.04 ^g	16.4 ± 0.06 ^a	0.24 ± 0.01 ^{ef}	289.6 ± 2.5 ^d	400.4 ± 2.0 ^d	4.83 ± 0.08 ^{fg}	0.62 ± 0.03 ^{gh}	0.45 ± 0.01 ^l	57.1 ± 2.5 ^{hi}	2.22 ± 0.04 ^{de}
UA-12	38.0 ± 0.1 ^f	251.1 ± 1.6 ^a	52.1 ± 0.3 ^c	4.4 ± 0.02 ^m	1.28 ± 0.01 ^b	138.6 ± 0.7 ^o	447.4 ± 1.8 ^a	5.97 ± 0.05 ^b	0.30 ± 0.01 ^j	0.56 ± 0.01 ^h	313.8 ± 1.2 ^d	2.89 ± 0.2 ^{cd}
UA-13	41.0 ± 0.2 ^c	60.7 ± 0.01 ^j	32.4 ± 0.4 ^d	8.1 ± 0.2 ^g	0.41 ± 0.01 ^d	288.5 ± 7.5 ^d	390.1 ± 8.1 ^e	5.31 ± 0.2 ^d	0.66 ± 0.01 ^{fgh}	0.61 ± 0.01 ^f	637.0 ± 18.2 ^a	2.07 ± 0.02 ^{def}
UA-14	39.0 ± 0.2 ^e	124.6 ± 0.01 ^c	50.6 ± 0.07 ^d	5.0 ± 0.03 ^k	0.12 ± 0.01 ^{ij}	181.9 ± 0.2 ^m	362.2 ± 0.2 ^h	4.06 ± 0.05 ^j	0.73 ± 0.03 ^{efg}	0.78 ± 0.01 ^d	30.0 ± 3.9 ^j	3.57 ± 1.1 ^c
UA-15	41.0 ± 0.1 ^c	26.0 ± 1.4 ^m	9.3 ± 0.02 ^o	15.1 ± 0.03 ^b	0.08 ± 0.01 ^j	235.9 ± 0.1 ⁱ	286.4 ± 1.6 ^o	4.71 ± 0.03 ^g	0.46 ± 0.01 ⁱ	0.43 ± 0.01 ^m	220.2 ± 9.4 ^e	1.04 ± 0.06 ^{fgh}
UM-01	41.0 ± 0.3 ^c	27.0 ± 0.8 ^m	13.7 ± 0.1 ^m	10.5 ± 0.03 ^c	0.15 ± 0.01 ^{hi}	256.7 ± 0.06 ^f	307.9 ± 0.9 ⁿ	4.99 ± 0.01 ^{ef}	0.34 ± 0.01 ^j	0.44 ± 0.01 ^m	32.7 ± 10.4 ⁱ	0.45 ± 0.01 ^{gh}
UM-02	39.0 ± 0.1 ^e	93.2 ± 0.9 ^e	36.6 ± 0.04 ^e	5.5 ± 0.01 ^j	0.21 ± 0.01 ^{fg}	220.0 ± 0.5 ^k	355.5 ± 0.9 ^{ij}	4.65 ± 0.02 ^{gh}	0.55 ± 0.01 ^{hi}	0.46 ± 0.01 ^{kl}	125.2 ± 10.6 ^g	5.42 ± 1.2 ^b
UM-03	43.0 ± 0.2 ^a	25.7 ± 0.8 ^m	6.4 ± 0.03 ^p	8.2 ± 0.01 ^{fg}	0.11 ± 0.01 ^{ij}	304.6 ± 0.09 ^b	345.1 ± 0.9 ^k	3.77 ± 0.03 ^j	0.80 ± 0.02 ^{de}	0.44 ± 0.01 ^m	228.3 ± 3.3 ^e	1.15 ± 0.02 ^{efgh}

UM-04	40.0 ± 0.1 ^d	38.0 ± 0.9 ⁱ	6.9 ± 0.03 ^p	7.9 ± 0.05 ^h	0.20 ± 0.01 ^{fgh}	298.3 ± 0.4 ^c	351.4 ± 0.5 ^{jk}	6.26 ± 0.01 ^a	0.86 ± 0.05 ^{cd}	0.64 ± 0.01 ^e	604.8 ± 18.3 ^b	1.44 ± 0.01 ^{efg}
US-01	40.0 ± 0.1 ^d	79.0 ± 0.9 ^g	53.1 ± 0.07 ^b	8.3 ± 0.02 ^f	0.15 ± 0.01 ^{ghi}	246.3 ± 0.3 ^{gh}	386.9 ± 0.8 ^{ef}	4.99 ± 0.01 ^{ef}	0.65 ± 0.01 ^{gh}	0.45 ± 0.01 ⁱ	n.d.	0.91 ± 0.1 ^{gh}
US-02	40.0 ± 0.2 ^d	60.2 ± 0.9 ^j	11.7 ± 0.1 ⁿ	10.2 ± 0.06 ^d	0.95 ± 0.03 ^c	331.6 ± 1.3 ^a	414.7 ± 1.2 ^b	6.03 ± 0.04 ^b	1.27 ± 0.1 ^a	0.50 ± 0.01 ⁱ	151.8 ± 6.7 ^{fg}	0.17 ± 0.02 ^h

*mg/100mL anhydrous alcohol; UA - Cachaças of Agreste; UM - Cachaças of Mata; US - Cachaças of Sertão. Different letters in the same column differ by tukey test (0.05). n.d. - not determined. Acrolein not determined.

Figure 1. Wheel of aromatic volatile compounds produced in Paraiba cachaças stored in Amburana barrels and their aroma descriptors.



Basead in (Supplementary table 2). References: Welke et al. (2014); Costa et al. (2008); Zhang et al. (2020); Wang et al. (2023); Zhao et al. (2024); Amorim et al. (2016); Bortoletto et al. (2021); Gonzalez-Robles & Cook (2016), Gabetti, et al. (2021).

Figure 2. Heat map for the volatile profile in Paraiba cachaças stored in Amburana barrels

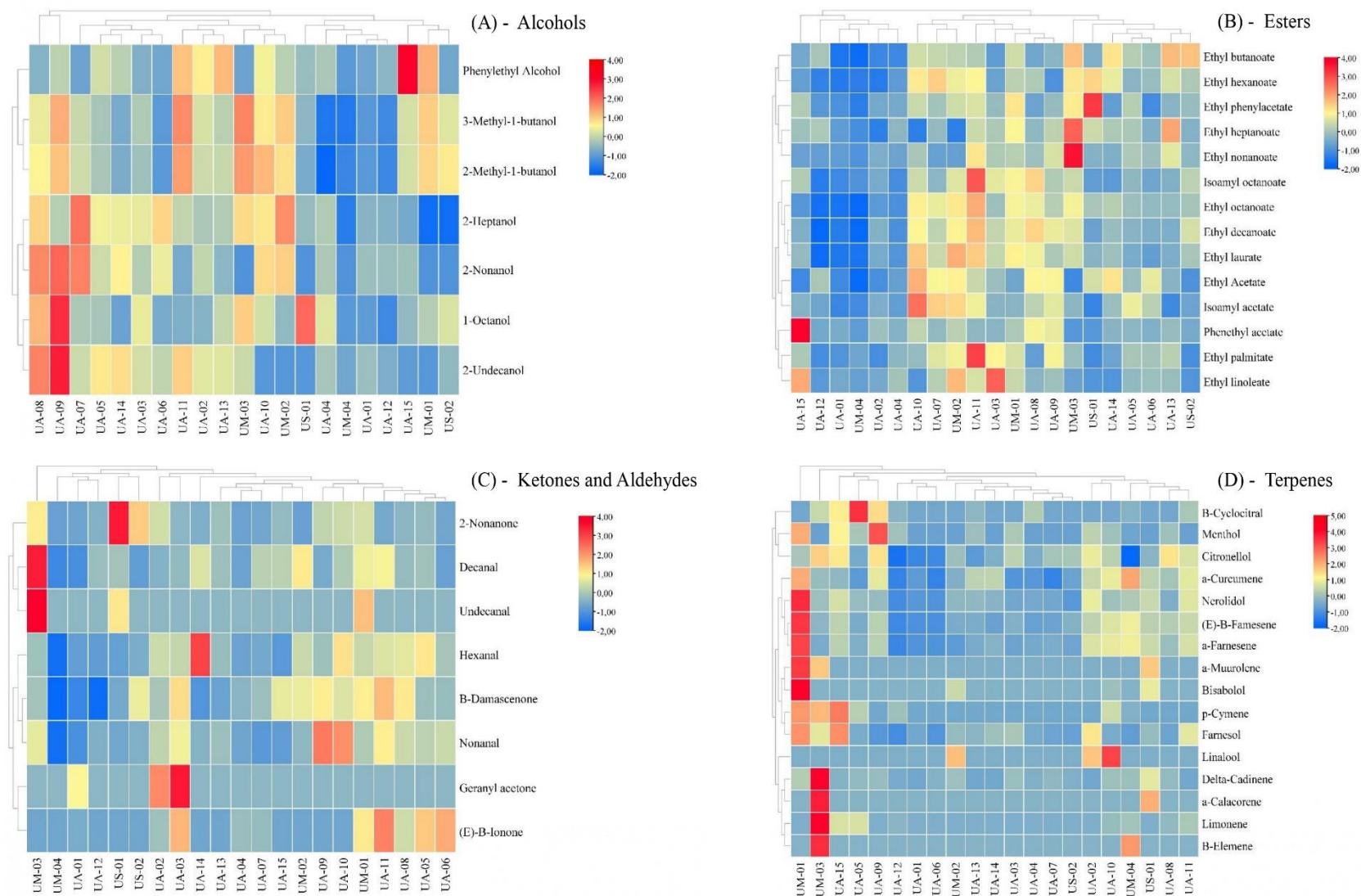
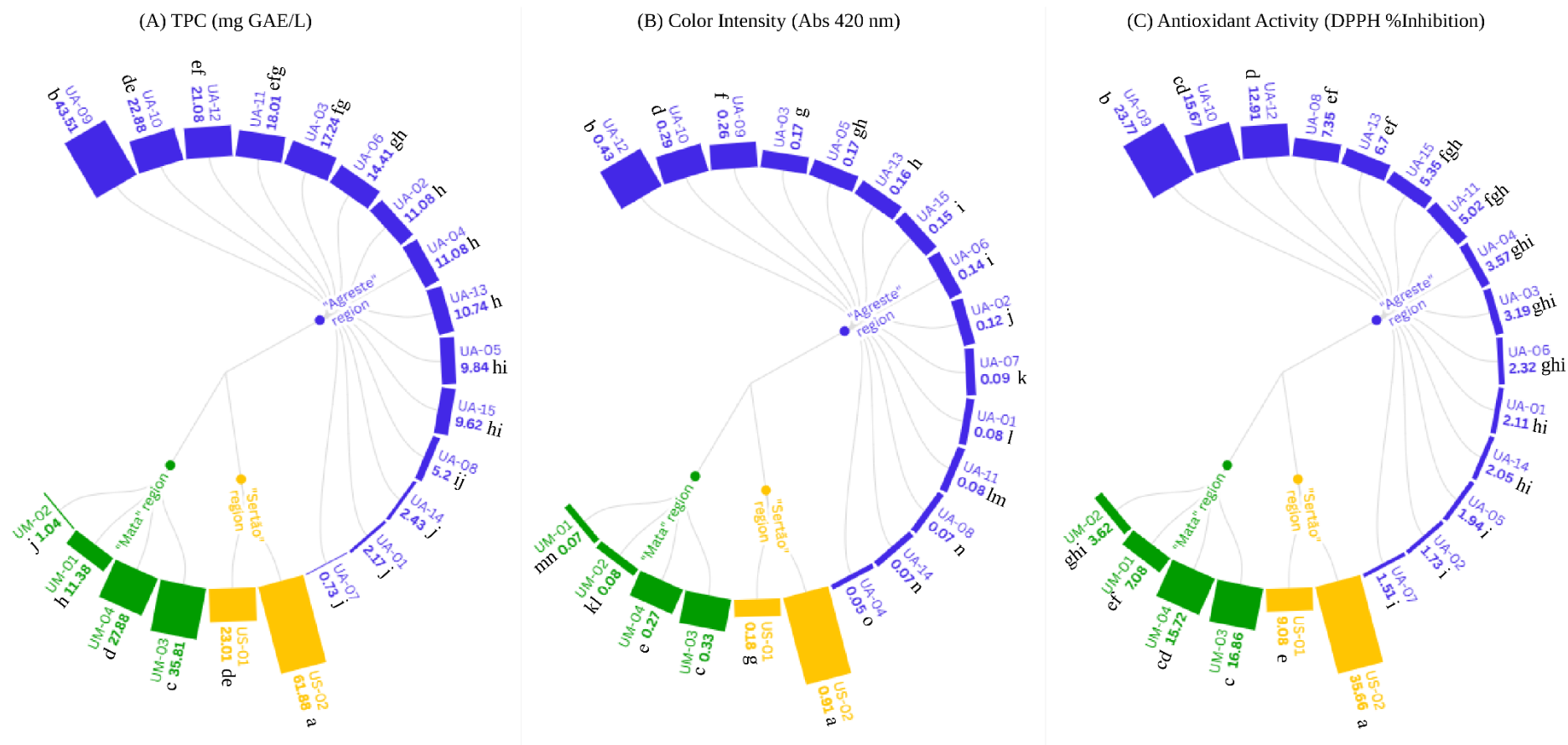


Figure 3. Determination of total phenolic compound (TPC) content, colour intensity and antioxidant activity by DPPH radical inhibition for commercial cachaças stored in *Amburana cearensis* barrels.



Note(s): Different lowercase letters indicate significant differences between the samples (Tukey's HSD, post-hoc test, $P < 0.05$).

Supplementary table 1. Storage of cachaças from Paraíba in Amburana (*Amburana cearenses*) barrels.

Sample	Stored		
	Time	Use	Container
UA-01	6 months	reuse	barrel > 700 L
UA-02	12 months	reuse	barrel > 700 L
UA-03	12 months	reuse	barrel > 700 L
UA-04	12 months	reuse	barrel > 700 L
UA-05	12 months	reuse	barrel > 700 L
UA-06	4 months	reuse	barrel > 700 L
UA-07	4 months	reuse	barrel > 700 L
UA-08	4 months	reuse	barrel > 700 L
UA-09	6 months	1 ^o use	barrel > 700 L
UA-10	6 months	1 ^o use	barrel > 700 L
UA-11	6 months	reuse	barrel > 700 L
UA-12	6 months	reuse	barrel > 700 L
UA-13	> 4 years	reuse	barrel > 700 L
UA-14	12 months	reuse	barrel > 700 L
UA-15	12 months	1 ^o use	barrel > 700 L
UM-01	12 months	1 ^o use	barrel > 700 L
UM-02	6 months	reuse	barrel > 700 L
UM-03	12 months	reuse	barrel < 700 L
UM-04	12 months	reuse	barrel > 700 L
US-01	6 months	reuse	barrel > 700 L
US-02	12 months	reuse	barrel > 700 L

Supplementary Table 2. Volatile profile of cachaças from Paraíba stored in Amburana (*Amburana cearenses*) barrels.

COV	CAS	Odor described	Samples (mg/L)						
			UA-01	UA-02	UA-03	UA-04	UA-05	UA-06	UA-07
Alcohols									
3-Methyl-1-butanol	123-51-3	Fruity ^{3,8} , alcohol ^{5,8} , sweet ⁸ .	179.67 ± 12.95 ^{kl}	372.14 ± 35.79 ^{efg}	313.84 ± 17.64 ^{fgh}	92.64 ± 6.88 ^m	299.25 ± 9.90 ^{ghi}	183.85 ± 25.16 ^{ijkl}	348.66 ± 4.95 ^{efg}
2-Methyl-1-butanol	137-32-6	Alcohol ^{5,8} , fruity ⁸ , sweet ⁸ .	58.66 ± 3.75 ^{jk}	134.73 ± 17.00 ^{fg}	104.62 ± 6.79 ^{ghi}	0.51 ± 0.06 ^l	111.47 ± 2.32 ^{fgh}	58.64 ± 7.23 ^{jk}	135.58 ± 2.61 ^{fg}
2-Heptanol	543-49-7	Lemon ¹ , orange ¹ .	3.93 ± 0.21 ^{efg}	5.05 ± 0.62 ^{efg}	7.09 ± 0.84 ^{bcd}	5.28 ± 0.54 ^{defg}	7.44 ± 0.50 ^{bc}	8.92 ± 1.36 ^b	12.14 ± 0.67 ^a
1-Octanol	111-87-5	Coconut ⁶ , walnut ⁶ , oily ⁶ .	2.52 ± 0.41 ^{lm}	4.43 ± 0.66 ^{hijkl}	9.36 ± 0.80 ^d	8.86 ± 0.14 ^{de}	6.02 ± 0.50 ^{ghij}	3.72 ± 0.51 ^{ikl}	6.29 ± 0.64 ^{fghi}
2-Nonanol	628-99-9	Fruity ¹ , green ¹ .	2.92 ± 0.35 ^{hi}	4.39 ± 0.44 ^{fg}	3.88 ± 0.47 ^{fgh}	3.57 ± 0.37 ^{gh}	4.80 ± 0.60 ^f	6.06 ± 0.40 ^e	11.23 ± 0.57 ^b
Phenylethyl Alcohol	60-12-8	Honey ¹ , rose ^{1,7} , flowery ^{5,7} , sweet ⁷ .	4.23 ± 0.54 ^{hij}	29.45 ± 2.41 ^c	7.12 ± 0.89 ^{ghij}	13.85 ± 1.88 ^{efg}	20.63 ± 3.20 ^{de}	4.46 ± 0.52 ^{hij}	3.46 ± 0.41 ^{ij}
2-Undecanol	1653-30-1	Fruity ¹ .	2.58 ± 0.39 ^{ghij}	5.20 ± 0.77 ^{de}	4.62 ± 0.22 ^{ef}	3.02 ± 0.41 ^{fghi}	6.76 ± 0.92 ^{cd}	3.88 ± 0.30 ^{efgh}	4.36 ± 0.09 ^{efg}
Esters									
Ethyl Acetate	141-78-6	Fruity ^{1,3} , pineapple ^{1,7} .	33.31 ± 2.23 ^j	38.02 ± 2.85 ^{ij}	79.45 ± 5.85 ^{fg}	53.24 ± 6.09 ^{hij}	79.24 ± 1.28 ^{fg}	118.03 ± 15.16 ^{bcd}	128.95 ± 0.68 ^{bc}
Ethyl butanoate	105-54-4	Strawberry ¹ , apple ¹ , pineapple ^{3,7} , fruity ⁵ .	0.53 ± 0.01 ^{hi}	0.96 ± 0.05 ^{gh}	1.44 ± 0.11 ^{fg}	0.97 ± 0.15 ^{gh}	2.60 ± 0.12 ^{bcd}	2.32 ± 0.27 ^{cde}	2.82 ± 0.03 ^{bc}
Isoamyl acetate	123-92-2	Sweet ¹ , fruity ¹ , banana ^{1,3,5,7} .	3.50 ± 0.32 ⁱ	10.95 ± 1.02 ^{efg}	12.12 ± 0.93 ^{efg}	8.00 ± 1.10 ^{gh}	23.17 ± 1.43 ^c	17.54 ± 1.24 ^d	29.27 ± 0.23 ^b
Ethyl hexanoate	123-66-0	Sweet ^{1,7} , green apple ^{1,6} , fruity ^{5,6,7} , anise ⁷ .	7.57 ± 0.05 ^k	8.03 ± 1.15 ^k	29.09 ± 3.36 ^{hi}	25.09 ± 4.11 ⁱ	38.43 ± 2.43 ^{gh}	43.14 ± 3.66 ^{fg}	86.71 ± 5.98 ^a
Ethyl heptanoate	106-30-9	Fruity ¹ .	2.50 ± 0.32 ^{hi}	n.d.	6.48 ± 0.45 ^{def}	4.86 ± 0.82 ^{fg}	6.37 ± 0.22 ^{def}	4.16 ± 0.26 ^{gh}	5.21 ± 0.11 ^{fg}
Ethyl octanoate	106-32-1	Sweet ^{1,6} , fruity ^{1,6,7} , pear ¹ , apple ⁶ , lemon ⁷ , anise ⁷ .	373.0 ± 10.40 ^e	735.6 ± 90.71 ^d	1098.9 ± 65.38 ^c	640.9 ± 45.25 ^d	1091.1 ± 27.39 ^c	1035.9 ± 104.46 ^c	1515.6 ± 26.91 ^b
Ethyl phenylacetate	101-97-3	Flowery ⁵ .	0.58 ± 0.05 ^{ijkl}	1.32 ± 0.19 ^{ghij}	1.88 ± 0.32 ^{fgh}	1.33 ± 0.12 ^{ghij}	2.46 ± 0.10 ^{def}	0.42 ± 0.08 ^{kl}	2.11 ± 0.05 ^{efg}
Phenethyl acetate	103-45-7	Flowery ^{1,5} , roses ⁶ , honey ¹ , Sweet ⁶ , apple ⁶ .	7.51 ± 0.77 ^{fghi}	15.29 ± 1.83 ^{cdef}	10.80 ± 1.46 ^{efg}	9.71 ± 1.44 ^{fgh}	11.62 ± 0.43 ^{efg}	8.96 ± 1.21 ^{fgh}	12.77 ± 1.56 ^{efg}
Ethyl nonanoate	123-29-5	Fruity ¹ , flowery ¹ .	23.74 ± 0.78 ^{kl}	42.61 ± 5.78 ^{hij}	75.36 ± 2.43 ^{de}	30.87 ± 2.02 ^{ijk}	74.54 ± 4.78 ^{de}	62.44 ± 7.77 ^{efg}	28.21 ± 0.74 ^{ijkl}
Ethyl decanoate	110-38-3	Fruity ¹ , grape ^{1,7} .	1222.9 ± 21.65 ^{jk}	2520.4 ± 344.29 ^{gh}	3426.8 ± 253.10 ^e	1707.0 ± 94.90 ^{ij}	2583.8 ± 75.50 ^{gh}	2427.3 ± 137.05 ^{gh}	3342.7 ± 60.21 ^{ef}
Isoamyl octanoate	2035-99-6	Sweet ¹ , fruity ¹ , cream ¹ , oily ¹ .	11.05 ± 0.40 ^{mn}	35.60 ± 5.22 ^{ijk}	80.44 ± 7.37 ^{cd}	13.74 ± 1.74 ^{mn}	44.67 ± 1.77 ^{ghi}	31.30 ± 1.94 ^{ijkl}	43.65 ± 1.15 ^{hi}
Ethyl laurate	106-33-2	Sweet ^{1,5,7} , flowery ^{1,7} , fruity ¹ , cream ¹ , soap ⁷ .	464.8 ± 7.72 ^k	1125.7 ± 184.41 ^{hi}	1711.1 ± 158.80 ^{cde}	856.2 ± 67.14 ^j	1146.3 ± 32.97 ^{hi}	976.2 ± 52.15 ^{ij}	1744.7 ± 37.90 ^{cd}

Ethyl palmitate	628-97-7	Sweet ¹ , fruity ¹ , fatty ^{1,7} , rancid ^{1,7} .	33.67 ± 2.09 ^{hi}	18.79 ± 3.26 ⁱ	274.34 ± 41.57 ^b	113.62 ± 19.15 ^{fg}	154.11 ± 5.23 ^{ef}	147.91 ± 21.43 ^{ef}	229.60 ± 12.57 ^{bc}
Ethyl linoleate	544-35-4	Fruity ⁵ , fatty ⁷ .	1.96 ± 0.55 ^{efgh}	2.03 ± 0.25 ^{efgh}	18.44 ± 5.63 ^a	4.61 ± 0.76 ^{cdef}	3.57 ± 0.34 ^{cdefgh}	4.15 ± 0.33 ^{cdefg}	5.70 ± 0.33 ^{cde}
Aldehydes									
Hexanal	66-25-1	Sweet ¹ , orange ¹ .	0.78 ± 0.08 ^j	1.48 ± 0.10 ^{de}	1.42 ± 0.09 ^{def}	0.84 ± 0.11 ^{hij}	1.93 ± 0.11 ^{bc}	1.23 ± 0.21 ^{efg}	0.90 ± 0.04 ^{ghij}
Nonanal	124-19-6	Flowery ¹ , citrus ^{1,2} , green ² , fatty ² .	9.06 ± 0.41 ^l	21.44 ± 2.43 ^{def}	26.69 ± 1.36 ^{bc}	13.04 ± 2.12 ^{ijkl}	21.14 ± 0.94 ^{def}	22.49 ± 1.74 ^{cde}	10.69 ± 1.20 ^{kl}
Decanal	112-31-2	Grassy ¹ , orange ¹ .	1.90 ± 0.14 ^{jk}	5.94 ± 1.14 ^{efghi}	6.88 ± 0.91 ^{efghi}	4.29 ± 0.44 ^{ghijk}	7.86 ± 0.51 ^{defgh}	4.12 ± 0.50 ^{hijk}	9.53 ± 0.49 ^{cde}
Undecanal	112-44-7	Flowery ¹ .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ketones									
2-Nonanone	821-55-6	Fruity ¹ .	0.62 ± 0.08 ^f	2.58 ± 0.30 ^c	1.23 ± 0.18 ^{ef}	0.57 ± 0.05 ^f	1.38 ± 0.16 ^{def}	0.68 ± 0.08 ^f	0.71 ± 0.05 ^{ef}
β-Damascenone	23726-93-4	Sweet ¹ , honey ¹ .	13.16 ± 0.68 ^{gh}	30.03 ± 3.23 ^d	47.60 ± 3.26 ^{ab}	25.65 ± 2.92 ^{de}	28.55 ± 1.00 ^{de}	27.77 ± 3.43 ^{de}	28.52 ± 1.37 ^{de}
Geranyl acetone	3796-70-1	Flowery ¹	1.15 ± 0.03 ^c	2.30 ± 0.36 ^b	3.49 ± 0.62 ^a	n.d.	n.d.	n.d.	n.d.
(E)-β-Ionone	79-77-6	Woody ⁴ , flowery ⁴ .	0.10 ± 0.02 ^{ef}	0.53 ± 0.06 ^e	2.32 ± 0.40 ^b	0.46 ± 0.05 ^{ef}	2.09 ± 0.34 ^{bc}	2.44 ± 0.37 ^{ab}	0.41 ± 0.06 ^{ef}
Terpenes									
p-Cymene	99-87-6	Fruity ¹ , sweet ¹ , citrus ² .	n.d.	n.d.	n.d.	n.d.	1.46 ± 0.15 ^d	n.d.	n.d.
Limonene	138-86-3	Citrus ² , fresh ² .	n.d.	n.d.	n.d.	n.d.	5.41 ± 0.76 ^b	n.d.	n.d.
Linalool	78-70-6	Sweet ¹ , citrus ^{1,2} , grape ¹ , flowery ^{1,2,3,7,8,9} , lavender ^{2,8,9} .	n.d.	5.65 ± 0.62 ^b	n.d.	n.d.	n.d.	n.d.	n.d.
Menthol	1490-04-6	Minty ⁹ .	n.d.	3.75 ± 0.50 ^d	3.01 ± 0.31 ^d	n.d.	2.83 ± 0.38 ^d	n.d.	n.d.
β-Cyclocitral	432-25-7	Fruity ⁴ , herbaceous ⁴ .	n.d.	n.d.	n.d.	3.71 ± 0.45 ^c	19.86 ± 2.45 ^a	n.d.	n.d.
Citronellol	106-22-9	Citrus ¹ , sweet ^{1,8} , oily ⁴ , rose ^{4,8} , flowery ⁸ .	2.47 ± 0.04 ^{ij}	7.77 ± 0.74 ^{bc}	6.37 ± 0.85 ^{de}	4.45 ± 0.33 ^{figh}	4.32 ± 0.26 ^{gh}	2.62 ± 0.42 ^{ij}	5.69 ± 0.25 ^{ef}
β-Elemene	515-13-9	Herbaceous ² , woody ² , green ² , sweet ³ .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
(E)-β-Farnesene	18794-84-8	Woody ^{3,4} , sweet ⁴ .	5.79 ± 0.76 ^{ghi}	30.28 ± 1.35 ^d	17.41 ± 1.11 ^{ef}	11.59 ± 1.07 ^{figh}	15.86 ± 1.07 ^{ef}	5.32 ± 0.59 ^{hi}	12.91 ± 0.72 ^f
α-Curcumene	644-30-4	Herbaceous ² , woody ² .	0.70 ± 0.07 ^{gh}	2.45 ± 0.02 ^{bc}	0.52 ± 0.08 ^{hi}	0.58 ± 0.08 ^h	0.67 ± 0.03 ^h	n.d.	n.d.
α-Murolene	10208-80-7	Woody ^{2,4} .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
α-Farnesene	502-61-4	Fruity ³ .	3.98 ± 0.04 ^{gh}	22.73 ± 3.64 ^{bc}	7.89 ± 1.38 ^{fg}	9.17 ± 0.86 ^{efg}	10.50 ± 1.35 ^{ef}	4.00 ± 0.31 ^{gh}	9.76 ± 0.28 ^{efg}

δ-Cadinene	483-76-1	Woody ^{2,3} , sweet ² , medicinal ² , thyme ⁴ , herbaceous ⁴ .	n.d.	n.d.	3.96 ± 0.37 ^{de}	1.38 ± 0.10 ^{ghi}	3.10 ± 0.39 ^{def}	1.72 ± 0.20 ^{fgh}	2.75 ± 0.17 ^{efg}
α-Calacorene	21391-99-1	Woody ² .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Nerolidol	7212-44-4	Apple ¹ , citrus ¹ , rose ^{1,7} , green ¹ , flowery ⁴ , woody ⁷ .	45.10 ± 5.82 ^h	240.24 ± 27.47 ^b	81.97 ± 11.47 ^g	85.38 ± 4.11 ^g	129.35 ± 3.64 ^{de}	36.15 ± 0.70 ^h	80.74 ± 1.22 ^g
Bisabolol	515-69-5	Spicy ² , flowery ² , peppery ⁷ .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Farnesol	4602-84-0	Flowery ¹ , medicinal ⁷ .	4.14 ± 0.42 ^{defgh}	18.28 ± 2.64 ^b	9.02 ± 2.87 ^c	4.55 ± 0.29 ^{defg}	4.71 ± 0.69 ^{cdefg}	1.27 ± 0.15 ^{gh}	3.54 ± 0.43 ^{efgh}
COV	CAS	Odor described	Samples (mg/L)						
			UA-08	UA-09	UA-10	UA-11	UA-12	UA-13	UA-14
Alcohols									
3-Methyl-1-butanol	123-51-3	Fruity ^{3,8} , alcohol ^{5,8} , sweet ⁸ .	403.74 ± 40.04 ^{de}	547.29 ± 14.30 ^{ab}	450.98 ± 1.91 ^{cd}	598.92 ± 64.55 ^a	140.32 ± 19.50 ^{lm}	327.93 ± 32.09 ^{efgh}	228.01 ± 6.08 ^{ijk}
2-Methyl-1-butanol	137-32-6	Alcohol ^{5,8} , fruity ⁸ , sweet ⁸ .	170.87 ± 0.59 ^{de}	203.40 ± 9.59 ^{abc}	211.11 ± 1.51 ^{ab}	228.12 ± 33.66 ^a	38.14 ± 0.09 ^k	131.66 ± 4.75 ^{fg}	80.38 ± 2.09 ^{hij}
2-Heptanol	543-49-7	Lemon ¹ , orange ¹ .	8.99 ± 0.35 ^b	5.49 ± 0.91 ^{def}	8.31 ± 0.81 ^b	5.84 ± 0.11 ^{cde}	3.75 ± 0.26 ^{fg}	5.35 ± 0.78 ^{def}	7.23 ± 0.37 ^{bcd}
1-Octanol	111-87-5	Coconut ⁶ , walnut ⁶ , oily ⁶ .	13.53 ± 2.50 ^c	20.39 ± 1.03 ^a	8.17 ± 1.11 ^{defg}	4.07 ± 0.66 ^{ijkl}	0.93 ± 0.10 ^m	7.53 ± 0.33 ^{defg}	3.02 ± 0.13 ^{klm}
2-Nonanol	628-99-9	Fruity ¹ , green ¹ .	10.99 ± 0.15 ^b	12.35 ± 0.56 ^a	7.77 ± 0.31 ^{cd}	1.51 ± 0.01 ^j	2.88 ± 0.08 ^{hi}	2.30 ± 0.37 ^{ij}	6.89 ± 0.27 ^{de}
Phenylethyl Alcohol	60-12-8	Honey ¹ , rose ^{1,7} , flowery ^{5,7} , sweet ⁷ .	8.02 ± 1.40 ^{ghi}	16.98 ± 2.27 ^{ef}	26.41 ± 4.66 ^{cd}	36.63 ± 2.38 ^b	8.77 ± 0.83 ^{ghi}	38.16 ± 0.10 ^b	16.56 ± 0.61 ^{ef}
2-Undecanol	1653-30-1	Fruity ¹ .	10.59 ± 0.39 ^b	14.42 ± 2.05 ^a	n.d.	7.69 ± 1.12 ^c	1.46 ± 0.13 ^{ijkl}	5.07 ± 0.32 ^{de}	7.17 ± 0.05 ^c
Esters									
Ethyl Acetate	141-78-6	Fruity ^{1,3} , pineapple ^{1,7} .	130.36 ± 17.52 ^b	127.84 ± 6.40 ^{bc}	165.10 ± 2.22 ^a	101.97 ± 11.22 ^{de}	87.47 ± 10.02 ^{ef}	69.92 ± 7.80 ^{fgh}	139.37 ± 4.26 ^b
Ethyl butanoate	105-54-4	Strawberry ¹ , apple ¹ , pineapple ^{3,7} , fruity ⁵ .	1.67 ± 0.01 ^f	2.02 ± 0.20 ^{def}	2.95 ± 0.04 ^b	2.54 ± 0.35 ^{bcd}	2.40 ± 0.31 ^{bcde}	4.40 ± 0.25 ^a	3.84 ± 0.15 ^a
Isoamyl acetate	123-92-2	Sweet ¹ , fruity ¹ , banana ^{1,3,5,7} .	17.70 ± 2.88 ^d	22.07 ± 1.59 ^c	39.48 ± 1.22 ^a	23.40 ± 3.60 ^c	9.38 ± 1.32 ^{fg}	4.12 ± 0.65 ^{hi}	14.16 ± 0.74 ^{de}
Ethyl hexanoate	123-66-0	Sweet ^{1,7} , green apple ^{1,6} , fruity ^{5,6,7} , anise ⁷ .	50.21 ± 1.53 ^f	20.09 ± 1.18 ^{ij}	78.67 ± 2.83 ^{abc}	74.70 ± 5.15 ^{bc}	10.89 ± 0.29 ^{jk}	62.23 ± 1.71 ^{de}	68.47 ± 5.39 ^{cd}
Ethyl heptanoate	106-30-9	Fruity ¹ .	4.96 ± 0.08 ^{fg}	7.31 ± 0.59 ^{de}	n.d.	8.01 ± 0.82 ^d	6.45 ± 0.16 ^{def}	14.78 ± 0.01 ^b	6.53 ± 0.33 ^{def}
Ethyl octanoate	106-32-1	Sweet ^{1,6} , fruity ^{1,6,7} , pear ¹ , apple ⁶ , lemon ⁷ , anise ⁷ .	1541.2 ± 125.62 ^b	1230.9 ± 16.55 ^c	1665.2 ± 1.17 ^b	2044.3 ± 196.74 ^a	347.6 ± 6.02 ^e	1050.9 ± 3.42 ^c	1232.7 ± 37.17 ^c
Ethyl phenylacetate	101-97-3	Flowery ⁵ .	1.15 ± 0.19 ^{hijk}	1.89 ± 0.12 ^{fgh}	2.83 ± 0.29 ^{de}	3.10 ± 0.53 ^{cd}	0.78 ± 0.06 ^{ijkl}	1.67 ± 0.23 ^{fghi}	0.98 ± 0.14 ^{ijk}
Phenethyl acetate	103-45-7	Flowery ^{1,5} , roses ⁶ , honey ¹ , Sweet ⁶ , apple ⁶ .	34.09 ± 5.04 ^b	31.16 ± 2.24 ^b	22.47 ± 1.81 ^c	12.04 ± 0.42 ^{efg}	6.21 ± 0.35 ^{ghi}	13.41 ± 0.05 ^{defg}	10.59 ± 1.49 ^{efg}

(E)- β -Farnesene	18794-84-8	Woody ^{3,4} , sweet ⁴ .	28.14 $\pm$ 0.71 ^d	29.46 $\pm$ 3.82 ^d	36.98 $\pm$ 0.73 ^{bc}	32.40 $\pm$ 2.48 ^{cd}	2.60 $\pm$ 0.25 ⁱ	16.04 $\pm$ 2.27 ^{ef}	12.04 $\pm$ 1.47 ^{fg}
α -Curcumene	644-30-4	Herbaceous ² , woody ² .	1.89 $\pm$ 0.13 ^{cde}	2.94 $\pm$ 0.11 ^b	3.03 $\pm$ 0.37 ^b	2.92 $\pm$ 0.30 ^b	n.d.	2.28 $\pm$ 0.27 ^c	2.32 $\pm$ 0.32 ^c
α -Muurolene	10208-80-7	Woody ^{2,4} .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
α -Farnesene	502-61-4	Fruity ³ .	14.20 $\pm$ 0.57 ^{de}	18.44 $\pm$ 2.48 ^{cd}	24.47 $\pm$ 1.23 ^{bc}	22.08 $\pm$ 0.28 ^{bc}	0.89 $\pm$ 0.08 ^h	9.37 $\pm$ 0.98 ^{efg}	11.35 $\pm$ 0.28 ^{ef}
δ -Cadinene	483-76-1	Woody ^{2,3} , sweet ² , medicinal ² , thyme ⁴ , herbaceous ⁴ .	3.29 $\pm$ 0.55 ^{de}	3.73 $\pm$ 0.28 ^{de}	4.37 $\pm$ 0.29 ^d	n.d.	0.58 $\pm$ 0.07 ^{hi}	n.d.	n.d.
α -Calacorene	21391-99-1	Woody ² .	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Nerolidol	7212-44-4	Apple ¹ , citrus ¹ , rose ^{1,7} , green ¹ , flowery ⁴ , woody ⁷ .	96.32 $\pm$ 0.21 ^{fg}	128.56 $\pm$ 11.66 ^{de}	140.02 $\pm$ 6.66 ^{de}	224.44 $\pm$ 4.95 ^{bc}	20.02 $\pm$ 1.09 ^h	121.64 $\pm$ 7.75 ^{ef}	110.40 $\pm$ 7.95 ^{efg}
Bisabolol	515-69-5	Spicy ² , flowery ² , peppery ⁷ .	n.d.	n.d.	0.98 $\pm$ 0.01 ^d	n.d.	n.d.	n.d.	n.d.
Farnesol	4602-84-0	Flowery ¹ , medicinal ⁷ .	3.60 $\pm$ 0.59 ^{efgh}	1.53 $\pm$ 0.23 ^{gh}	4.79 $\pm$ 0.35 ^{cdefg}	14.14 $\pm$ 1.38 ^b	n.d.	6.14 $\pm$ 0.83 ^{cdef}	8.11 $\pm$ 0.19 ^{cd}

COV	CAS	Odor described	Samples (mg/L)						
			UA-15	UM-01	UM-02	UM-03	UM-04	US-01	US-02
Alcohols									
3-Methyl-1-butanol	123-51-3	Fruity ^{3,8} , alcohol ^{5,8} , sweet ⁸ .	369.43 ± 23.38 ^{efg}	497.84 ± 18.51 ^{bc}	498.38 ± 3.09 ^{bc}	607.99 ± 32.37 ^a	94.17 ± 3.21 ^m	260.18 ± 31.92 ^{hij}	385.98 ± 17.32 ^{def}
2-Methyl-1-butanol	137-32-6	Alcohol ^{5,8} , fruity ⁸ , sweet ⁸ .	140.92 ± 5.24 ^{ef}	190.84 ± 4.73 ^{bcd}	183.51 ± 3.89 ^{bcd}	231.67 ± 12.01 ^a	36.38 ± 2.32 ^k	79.17 ± 4.30 ^{ij}	176.77 ± 14.41 ^{cd}
2-Heptanol	543-49-7	Lemon ¹ , orange ¹ .	3.35 ± 0.30 ^g	n.d.	11.21 ± 1.21 ^a	8.72 ± 0.37 ^b	0.84 ± 0.04 ^h	4.39 ± 0.28 ^{efg}	n.d.
1-Octanol	111-87-5	Coconut ⁶ , walnut ⁶ , oily ⁶ .	5.05 ± 0.47 ^{hijk}	6.71 ± 0.79 ^{efgh}	4.98 ± 0.36 ^{hijk}	12.39 ± 0.10 ^c	2.18 ± 0.16 ^{lm}	17.38 ± 0.52 ^b	8.53 ± 0.47 ^{def}
2-Nonanol	628-99-9	Fruity ¹ , green ¹ .	3.01 ± 0.37 ^{hi}	n.d.	8.27 ± 0.32 ^c	n.d.	n.d.	n.d.	n.d.
Phenylethyl Alcohol	60-12-8	Honey ¹ , rose ^{1,7} , flowery ^{5,7} , sweet ⁷ .	69.86 ± 7.16 ^a	40.00 ± 0.44 ^b	16.45 ± 0.78 ^{ef}	3.61 ± 0.25 ^{ij}	0.71 ± 0.06 ^j	11.00 ± 0.91 ^{fgh}	4.61 ± 0.57 ^{hij}
2-Undecanol	1653-30-1	Fruity ¹ .	0.33 ± 0.05 ^{kl}	n.d.	n.d.	4.32 ± 0.24 ^{efg}	0.98 ± 0.15 ^{jkl}	n.d.	2.14 ± 0.18 ^{hijk}
Esters									
Ethyl Acetate	141-78-6	Fruity ^{1,3} , pineapple ^{1,7} .	32.68 ± 1.70 ^j	58.09 ± 2.29 ^{ghi}	132.26 ± 1.99 ^b	33.02 ± 2.15 ^j	5.49 ± 0.33 ^k	107.87 ± 7.78 ^{cde}	41.01 ± 1.74 ^{ij}
Ethyl butanoate	105-54-4	Strawberry ¹ , apple ¹ , pineapple ^{3,7} , fruity ⁵ .	1.71 ± 0.06 ^f	2.96 ± 0.07 ^b	2.73 ± 0.04 ^{bc}	4.34 ± 0.36 ^a	0.30 ± 0.03 ⁱ	1.88 ± 0.06 ^{ef}	4.29 ± 0.42 ^a
Isoamyl acetate	123-92-2	Sweet ¹ , fruity ¹ , banana ^{1,3,5,7} .	11.85 ± 0.75 ^{efg}	12.19 ± 1.05 ^{efg}	28.43 ± 0.66 ^b	12.50 ± 1.86 ^{ef}	4.56 ± 0.10 ^{hi}	3.20 ± 0.22 ⁱ	9.24 ± 0.50 ^{fg}
Ethyl hexanoate	123-66-0	Sweet ^{1,7} , green apple ^{1,6} , fruity ^{5,6,7} , anise ⁷ .	26.97 ± 2.24 ⁱ	53.66 ± 5.10 ^{ef}	69.91 ± 1.35 ^{cd}	79.25 ± 7.23 ^{abc}	9.33 ± 0.83 ^{jk}	85.13 ± 0.86 ^{ab}	49.66 ± 4.68 ^f

Ethyl heptanoate	106-30-9	Fruity ¹ .	5.77 ± 0.36 ^{efg}	10.55 ± 0.51 ^c	n.d.	18.31 ± 1.79 ^a	1.19 ± 0.04 ^{ij}	8.08 ± 0.85 ^d	4.42 ± 0.72 ^g
Ethyl octanoate	106-32-1	Sweet ^{1,6} , fruity ^{1,6,7} , pear ¹ , apple ⁶ , lemon ⁷ , anise ⁷ .	703.5 ± 38.74 ^d	1581.4 ± 50.76 ^b	1708.0 ± 19.65 ^b	1576.9 ± 90.79 ^b	268.1 ± 17.04 ^e	1151.9 ± 21.94 ^c	1141.2 ± 90.71 ^c
Ethyl phenylacetate	101-97-3	Flowery ⁵ .	2.37 ± 0.40 ^{def}	4.02 ± 0.08 ^b	2.90 ± 0.19 ^{de}	3.92 ± 0.78 ^{bc}	n.d.	7.10 ± 0.20 ^a	1.94 ± 0.15 ^{fgh}
Phenethyl acetate	103-45-7	Flowery ^{1,5} , roses ⁶ , honey ¹ , Sweet ⁶ , apple ⁶ .	88.67 ± 10.46 ^a	18.32 ± 0.43 ^{cde}	21.35 ± 1.07 ^{cd}	n.d.	1.97 ± 0.27 ^{hi}	n.d.	6.91 ± 0.34 ^{fghi}
Ethyl nonanoate	123-29-5	Fruity ¹ , flowery ¹ .	29.17 ± 1.42 ^{ijk}	80.06 ± 7.14 ^d	27.76 ± 1.05 ^{kl}	277.45 ± 15.90 ^a	12.70 ± 0.87 ^l	47.32 ± 0.70 ^{gh}	53.62 ± 6.16 ^{fgh}
Ethyl decanoate	110-38-3	Fruity ¹ , grape ^{1,7} .	2441.0 ± 123.82 ^{gh}	4001.1 ± 108.20 ^{cd}	4306.1 ± 22.16 ^{bc}	3807.3 ± 121.39 ^{cde}	734.2 ± 14.94 ^{kl}	2141.3 ± 20.11 ^{hi}	3663.4 ± 240.97 ^{de}
Isoamyl octanoate	2035-99-6	Sweet ¹ , fruity ¹ , cream ¹ , oily ¹ .	59.10 ± 5.41 ^{fg}	89.31 ± 5.43 ^c	78.26 ± 1.01 ^{cde}	69.33 ± 3.42 ^{def}	17.74 ± 0.57 ^{lm}	25.51 ± 2.15 ^{klm}	69.89 ± 5.52 ^{ef}
Ethyl laurate	106-33-2	Sweet ^{1,5,7} , flowery ^{1,7} , fruity ¹ , cream ¹ , soap ⁷ .	1348.1 ± 86.96 ^{fgh}	2193.4 ± 106.07 ^b	2695.9 ± 53.47 ^a	1795.2 ± 12.91 ^c	426.7 ± 19.37 ^k	1253.5 ± 16.78 ^{gh}	1494.2 ± 12.54 ^{ef}
Ethyl palmitate	628-97-7	Sweet ¹ , fruity ¹ , fatty ^{1,7} , rancid ^{1,7} .	158.67 ± 19.98 ^{ef}	218.03 ± 12.34 ^{cd}	259.24 ± 3.05 ^{bc}	135.14 ± 10.55 ^{ef}	73.69 ± 6.52 ^{gh}	11.27 ± 0.80 ⁱ	18.77 ± 1.33 ⁱ
Ethyl linoleate	544-35-4	Fruity ⁵ , fatty ⁷ .	14.26 ± 1.06 ^b	6.64 ± 0.60 ^{cd}	13.33 ± 0.01 ^b	1.28 ± 0.11 ^{fgh}	1.09 ± 0.10 ^{fgh}	n.d.	n.d.
Aldehydes									
Hexanal	66-25-1	Sweet ¹ , orange ¹ .	0.73 ± 0.01 ^j	1.45 ± 0.03 ^{def}	1.46 ± 0.13 ^{def}	1.19 ± 0.08 ^{efg}	0.24 ± 0.03 ^j	0.80 ± 0.04 ^{ij}	0.95 ± 0.01 ^{ghij}
Nonanal	124-19-6	Flowery ¹ , citrus ^{1,2} , green ² , fatty ² .	11.68 ± 0.24 ^{ijkl}	19.93 ± 1.19 ^{efg}	16.39 ± 1.74 ^{ghi}	25.56 ± 0.54 ^{bcd}	3.25 ± 0.12 ^m	13.40 ± 1.55 ^{ijkl}	14.89 ± 2.03 ^{hijk}
Decanal	112-31-2	Grassy ¹ , orange ¹ .	9.61 ± 0.95 ^{cde}	13.14 ± 0.59 ^{bc}	14.86 ± 1.53 ^b	28.70 ± 4.57 ^a	1.16 ± 0.12 ^k	8.08 ± 0.35 ^{defg}	3.58 ± 0.39 ^{ijk}
Undecanal	112-44-7	Flowery ¹ .	n.d.	2.68 ± 0.03 ^b	n.d.	5.77 ± 0.23 ^a	n.d.	2.04 ± 0.16 ^c	n.d.
Ketones									
2-Nonanone	821-55-6	Fruity ¹ .	1.37 ± 0.24 ^{def}	2.63 ± 0.11 ^c	0.52 ± 0.01 ^f	3.66 ± 0.22 ^b	0.51 ± 0.04 ^f	7.87 ± 1.10 ^a	4.29 ± 0.06 ^b
β-Damascenone	23726-93-4	Sweet ¹ , honey ¹ .	40.13 ± 4.78 ^{bc}	44.68 ± 1.71 ^{bc}	40.40 ± 4.21 ^{bc}	29.45 ± 4.33 ^d	5.02 ± 0.14 ⁱ	22.43 ± 1.11 ^{def}	40.76 ± 3.32 ^{bc}
Geranyl acetone	3796-70-1	Flowery ¹	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
(E)-β-Ionone	79-77-6	Woody ⁴ , flowery ⁴ .	n.d.	1.71 ± 0.26 ^c	n.d.	n.d.	n.d.	n.d.	n.d.
Terpenes									
p-Cymene	99-87-6	Fruity ¹ , sweet ¹ , citrus ² .	5.63 ± 0.64 ^a	5.02 ± 0.25 ^b	n.d.	4.33 ± 0.17 ^c	0.33 ± 0.05 ^f	n.d.	n.d.
Limonene	138-86-3	Citrus ² , fresh ² .	5.49 ± 0.75 ^b	n.d.	n.d.	22.88 ± 2.67 ^a	2.24 ± 0.25 ^{cd}	n.d.	n.d.
Linalool	78-70-6	Sweet ¹ , citrus ^{1,2} , grape ¹ , flowery ^{1,2,3,7,8,9} , lavender ^{2,8,9} .	n.d.	n.d.	5.88 ± 0.09 ^b	n.d.	n.d.	n.d.	n.d.

Menthol	1490-04-6	Minty ⁹ .	6.83 ± 0.56 ^c	10.07 ± 0.76 ^b	n.d.	n.d.	0.21 ± 0.03 ^e	n.d.	n.d.
β-Cyclocitral	432-25-7	Fruity ⁴ , herbaceous ⁴ .	8.56 ± 0.71 ^b	n.d.	n.d.	4.95 ± 0.39 ^c	1.08 ± 0.09 ^e	n.d.	n.d.
Citronellol	106-22-9	Citrus ¹ , sweet ^{1,8} , oily ⁴ , rose ^{4,8} , flowery ⁸ .	8.76 ± 0.79 ^{ab}	5.77 ± 0.38 ^{ef}	5.44 ± 0.26 ^{efg}	9.52 ± 0.59 ^a	0.88 ± 0.06 ^k	4.89 ± 0.15 ^{gh}	5.79 ± 0.23 ^{ef}
β-Elemene	515-13-9	Herbaceous ² , woody ² , green ² , sweet ³ .	n.d.	n.d.	n.d.	2.39 ± 0.28 ^a	1.59 ± 0.21 ^b	n.d.	n.d.
(E)-β-Farnesene	18794-84-8	Woody ^{3,4} , sweet ⁴ .	30.13 ± 3.07 ^d	86.30 ± 5.11 ^a	15.86 ± 0.33 ^{ef}	19.65 ± 0.20 ^e	40.83 ± 3.69 ^b	29.32 ± 1.49	17.31 ± 1.70 ^{ef}
α-Curcumene	644-30-4	Herbaceous ² , woody ² .	1.54 ± 0.07 ^{ef}	4.60 ± 0.19 ^a	1.28 ± 0.18 ^{fg}	1.67 ± 0.19 ^{def}	4.69 ± 0.33 ^a	2.25 ± 0.27 ^{cd}	0.92 ± 0.07 ^{gh}
α-Murolene	10208-80-7	Woody ^{2,4} .	n.d.	4.91 ± 0.33 ^a	n.d.	2.67 ± 0.41 ^b	n.d.	2.67 ± 0.31 ^b	n.d.
α-Farnesene	502-61-4	Fruity ³ .	18.38 ± 3.24 ^{cd}	52.38 ± 6.11 ^a	5.80 ± 0.04 ^{fgh}	9.76 ± 1.67 ^{efg}	25.71 ± 1.83 ^b	20.49 ± 1.25 ^{bc}	8.13 ± 0.91 ^{efg}
δ-Cadinene	483-76-1	Woody ^{2,3} , sweet ² , medicinal ² , thyme ⁴ , herbaceous ⁴ .	1.65 ± 0.27 ^{gh}	6.40 ± 0.40 ^c	4.28 ± 0.19 ^d	38.83 ± 1.60 ^a	4.16 ± 0.10 ^d	11.29 ± 0.73 ^b	1.46 ± 0.07 ^{gh}
α-Calacorene	21391-99-1	Woody ² .	n.d.	n.d.	n.d.	11.85 ± 0.41 ^a	n.d.	6.71 ± 0.12 ^b	n.d.
Nerolidol	7212-44-4	Apple ¹ , citrus ¹ , rose ^{1,7} , green ¹ , flowery ⁴ , woody ⁷ .	211.94 ± 22.74 ^{bc}	538.24 ± 15.23 ^a	119.23 ± 10.05 ^{ef}	138.75 ± 5.11 ^{de}	160.46 ± 8.79 ^d	194.67 ± 0.19 ^c	46.92 ± 3.37 ^h
Bisabolol	515-69-5	Spicy ² , flowery ² , peppery ⁷ .	n.d.	14.83 ± 0.83 ^a	2.71 ± 0.23 ^c	n.d.	n.d.	4.02 ± 0.38 ^b	n.d.
Farnesol	4602-84-0	Flowery ¹ , medicinal ⁷ .	26.18 ± 3.10 ^a	25.83 ± 2.72 ^a	7.33 ± 1.14 ^{cde}	14.45 ± 2.20 ^b	2.58 ± 0.26 ^{fgh}	7.35 ± 0.10 ^{cde}	3.07 ± 0.20 ^{efgh}

UA - Cachaças of Agreste; UM - Cachaças of Mata; US - Cachaças of Sertão. Different letters in the same column differ by tukey test (0.05). n.d. – not determined. References: ¹Welke et al. (2014); ²Costa et al. (2008); ³Zhang et al. (2020); ⁴Wang et al. (2023); ⁵Zhao et al. (2024); ⁶Amorim et al. (2016); ⁷Bortoletto et al. (2021); ⁸Gonzalez-Robles & Cook (2016); ⁹Gabetti, et al. (2021).

Supplementary table 3. Determination of total phenolic compound (TPC) content, colour intensity and antioxidant activity by DPPH radical inhibition for commercial cachaças stored in *Amburana cearensis* barrels.

Sample	Total Phenolic Compounds (mgAGE/ 100 mL)	Color Intensity (Abs 420 nm)	Antioxidant activity - DPPH (%Inhibition)
UA-01	2.17 ± 0.09 ^j	00.079 ± 0.002 ^l	2.1 ± 0.2 ^{hi}
UA-02	11.1 ± 0.5 ^h	0.1187 ± 0.0006 ^j	1.7 ± 0.7 ⁱ
UA-03	17.2 ± 0.7 ^{fg}	0.171 ± 0.001 ^g	3.2 ± 0.7 ^{ghi}
UA-04	11.1 ± 0.3 ^h	0.1 ± 0.0 ^o	3.6 ± 0.5 ^{ghi}
UA-05	9.8 ± 0.9 ^{hi}	0.168 ± 0.002 ^{gh}	1.9 ± 0.6 ⁱ
UA-06	14.4 ± 0.4 ^{gh}	0.137 ± 0.002 ⁱ	2.32 ± 0.09 ^{ghi}
UA-07	0.73 ± 0.01 ^j	0.093 ± 0.002 ^k	1.5 ± 0.2 ⁱ
UA-08	5.2 ± 0.3 ^{ij}	0.0675 ± 0.0005 ⁿ	7.3 ± 0.5 ^{ef}
UA-09	44 ± 2 ^b	0.3 ± 0.0 ^f	23.8 ± 1.7 ^b
UA-10	23 ± 2 ^{de}	0.289 ± 0.002 ^d	15.7 ± 1.8 ^{cd}
UA-11	18.0 ± 1.0 ^{efg}	0.0785 ± 0.0005 ^{lm}	5.0 ± 0.5 ^{fgh}
UA-12	21.1 ± 1.0 ^{ef}	0.433 ± 0.004 ^b	12.9 ± 0.9 ^d
UA-13	10.7 ± 0.9 ^h	0.2 ± 0.0 ^h	6.7 ± 0.5 ^{ef}
UA-14	2.4 ± 0.3 ^j	0.0655 ± 0.0005 ⁿ	2.1 ± 0.2 ^{hi}
UA-15	9.6 ± 0.1 ^{hi}	0.146 ± 0.002 ⁱ	5.3 ± 0.5 ^{fgh}
UM-01	11.4 ± 0.9 ^h	0.1 ± 0.0 ^{mn}	7.1 ± 0.4 ^{ef}
UM-02	1.04 ± 0.02 ^j	0.084 ± 0.001 ^{kl}	3.6 ± 0.2 ^{ghi}
UM-03	35.8 ± 6.0 ^c	0.3305 ± 0.0005 ^c	16.9 ± 1.0 ^c
UM-04	27.9 ± 0.2 ^d	0.271 ± 0.003 ^e	15.7 ± 1.5 ^{cd}
US-01	23 ± 2 ^{de}	0.176 ± 0.003 ^g	9.1 ± 1.2 ^e
US-02	62 ± 3 ^a	0.91 ± 0.01 ^a	35.7 ± 2.4 ^a

Note(s): Different lowercase letters in columns indicate significant differences between samples (Tukey's HSD, teste post-hoc, P <0,05).

6. CONSIDERAÇÕES FINAIS

Foi realizado o mapeamento dos estabelecimentos produtores de cachaças da Paraíba, e identificado a produção de cachaça em três mesorregiões (Mata Paraibana, Agreste e Sertão Paraibano), onde foram coletadas 26 cachaças não envelhecidas (tradicional) e 21 cachaças armazenadas em barris de Amburana.

Com relação a qualidade química das cachaças paraibanas, os resultados demonstraram que a maioria das cachaças não envelhecidas (tradicional) atenderam aos parâmetros legais exigidos, refletindo boas práticas de produção por parte dos produtores; no entanto, os resultados das cachaças armazenadas em barris de Amburana, evidenciaram uma grande variabilidade na composição química, refletindo as diferenças nos métodos de produção, tempo de armazenamento e condições dos barris, com a presença de carbamato de etila em concentrações superiores ao permitido em quase metade das amostras analisadas, reforçando a necessidade de maior controle sobre o processo produtivo, desde a qualidade da cana-de-açúcar até a destilação e o armazenamento.

A análise dos compostos voláteis das cachaças paraibanas não envelhecidas (tradicional), revelou um perfil aromático diversificado, diretamente influenciado pelas condições edafoclimáticas e pela localização geográfica das unidades produtoras. As diferenças significativas entre os perfis aromáticos das cachaças das mesorregiões da Mata Paraibana, do Agreste e do Sertão Paraibano, confirmam a existência de um *terroir* para as cachaças paraibanas. Essas variações contribuem para a riqueza sensorial do produto, agregando valor comercial e fortalecendo a identidade regional da cachaça.

O perfil de compostos voláteis e não-voláteis das cachaças paraibanas armazenadas em barris de Amburana, identificou uma diversidade dos compostos voláteis que influenciam diretamente a percepção sensorial da bebida, sendo impactada pelo tempo de envelhecimento e reutilização dos barris. Esses fatores destacam a importância de regulamentações mais claras quanto ao armazenamento da cachaça, visando garantir não apenas a segurança e a qualidade do produto final, mas também preservar sua identidade sensorial e agregando valor ao produto.

Dessa forma, compreender todo o processo de produção é essencial para o aprimoramento da qualidade, e assim produzir uma bebida com qualidade, e com perfil aromático e sensorial que caracterize a cachaça paraibana.

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8 PRINCIPAIS CONTRIBUIÇÕES TECNICO-CIENTÍFICAS

IMPLANTAÇÃO DE METODOLOGIAS OFICIAIS PARA ANÁLISE DE CACHAÇA EM ATENDIMENTO AO PROJETO FAPESQ-PB Nº 04/2021 – APOIO A PESQUISAS PARA ARRANJOS PRODUTIVOS LOCAIS

- Participação na implantação das metodologias para as análises de identidade e qualidade de cachaça e aguardente de cana, exigidas pela Portaria MAPA Nº 539/2022, no Laboratório de *Flavor*/DEA/UFPB, bem como a realização das análises das cachaças paraibanas, em cumprimento ao projeto aprovado pelo edital FAPESQ-PB Nº 04/2021 – Apoio a pesquisas para arranjos produtivos locais.

APRESENTAÇÃO DE PESQUISAS EM EVENTOS E INTERAÇÃO COM OS PRODUTORES DE CACHAÇA E CONSUMIDORES.

- Evento Brasil Cachaças – Uma Lapada de Ciência: Participação como palestrante, apresentando resultados da pesquisa sobre: Voláteis das Cachaças Tradicionais Paraibanas, promovendo a interação entre academia, produtores e consumidores.
- Evento Brasil Cachaças – I Concurso de Sobremesas com Cachaça: Premiada em 2º lugar na categoria estudante, com a sobremesa “Noivinha Arretada” produzida com diferentes cachaças paraibanas (tradicional, armazenadas em barris de Amburana e de Carvalho) do Engenho Nobre. A construção da sobremesa reuniu o conhecimento químico e gastronômico, onde cada tipo de cachaça utilizada na preparação, foi pensada, levando em consideração o perfil aromático de cada bebida, agregando aromas e sabores, além de memória afetiva para essa sobremesa.
- Evento "Conexão Cachaça – Universidade em Parceria com a Indústria": Foram apresentados resultados da pesquisa sobre o Perfil Aromático das Cachaças Paraibanas.

PUBLICAÇÕES E APRESENTAÇÕES DE TRABALHOS CIENTÍFICOS.

- Apresentação de trabalho intitulado "Perfil aromático das cachaças tradicionais da Paraíba" no 15º Simpósio Latino-Americano de Ciência de Alimentos e Nutrição (SLACAN).
- Participação em artigos científicos de mesma temática, contribuindo com as metodologias implantadas e fornecimento de amostras coletadas.

Artigo publicado no periódico *Food Chemistry*, em 2025, sob o título *Accelerated aging of Brazilian sugarcane spirit: Impact of wood chips reuse on the phenolic and volatile profile of the beverage*. <https://doi.org/10.1016/j.foodchem.2025.143163>

Artigo publicado no periódico *Food Chemistry*, em 2025, sob o título *Quantification of alcohol content and identification of fraud in traditional cachaças using NIR spectroscopy*. <https://doi.org/10.1016/j.foodchem.2025.143809>

IMPACTO SOCIAL DO PROJETO

A pesquisa contribuiu para uma maior interação da universidade junto ao setor produtivo de cachaça da Paraíba, desde a coleta das amostras onde foi possível compreender as dificuldades e reais necessidades dos produtores paraibanos, que buscam por atender as exigências da legislação, encontrando parceria com universidade com a implantação das metodologias oficiais.

Os resultados desse estudo favorecem o arranjo produtivo local de cachaça, com informações que contribuem para a valorização da cachaça paraibana, um produto de grande destaque e relevância econômica e cultural no Brasil.

A apresentação dos dados científicos junto ao público geral durante os eventos, promoveu impacto positivo junto aos consumidores, consolidando a cachaça como um produto de alta qualidade, agregando valor a bebida e fortalecendo sua identidade no mercado.



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Accelerated aging of Brazilian sugarcane spirit: Impact of wood chips reuse on the phenolic and volatile profile of the beverage

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ABSTRACT

The study investigated the impact of reusing wood chips in the maturation of sugarcane spirits on the chemical profile (non-volatile and volatile) of the beverage. Chips of oak (*Quercus* sp.), amburana (*Amburana cearensis*), and chestnut (*Bertholletia excelsa*) were used in two maturation cycles. The first use of the chips resulted in greater extraction of phenolic and volatile compounds (especially esters and terpenes), increasing color intensity and antioxidant activity, promoting more complex beverages. Oak stood out for its higher phenolic content and greater antioxidant activity, while amburana and chestnut had different phenolic profiles. Compounds such as vanillin, vanillic acid and procyanidin-B2 were confirmed as markers of the woods studied. Reusing the chips reduced the concentration of antioxidant compounds, although it maintained the legal quality standards. Thus, the use of woodchips is a promising technique for adding aging markers in a short time, although their reuse is a limited practice.

1. Introduction

The aging process is essential for the development of desirable sensory characteristics in fermented and distilled beverages, such as spirits, cachaça, whiskey, and cognac, adding color, aromatic complexity, and flavor, which make the beverages more attractive and allow them to be marketed with greater added value (Bortoletto et al., 2016; Da Silva Cruz et al., 2020).

During aging, distillates interact with the container, traditionally wooden barrels, which contribute significantly to the final profile of the beverage, adding compounds derived from plant structures, such as phenolic and volatile compounds, which characterize aged distillates (Silvello et al., 2021). However, despite the benefits, the traditional process of aging in barrels is considered slow and expensive, due to the

gradual nature of extraction, immobilization of working capital in the form of packaging, as well as acquisition costs, and limited knowledge about the effect of reusing wood to produce new drinks (Krüger et al., 2022).

Studies have explored alternatives to traditional aging, focusing on methods that speed up the process without compromising the quality of the beverage. One of these alternatives is the use of chips, which are fragments of wood in the form of cubes or splinters, where they are immersed in the product (Krüger et al., 2022). This technique has been applied to fermented and distilled beverages, with promising results in terms of reducing costs and maintaining chemical quality, as the increased contact surface between the wood and the liquid promotes faster extraction of phenolic and volatile compounds that are markers of aging (Caldeira et al., 2017). Despite the growing interest, few studies

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Quantification of alcohol content and identification of fraud in traditional cachaças using NIR spectroscopy

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ABSTRACT

Cachaça, a traditional Brazilian alcoholic beverage, plays a significant economic role in Brazil. Traditional quality control methods are expensive, requiring specialized labor and extended response times. This study proposes innovative and eco-friendly approaches using near-infrared (NIR) spectroscopy for alcohol content quantification and fraud detection, eliminating the need for chemical reagents. A dataset of 462 cachaças from the Brejo microregion of Paraíba, Brazil, was analyzed using variable selection algorithms: PLS, MCUVE-PLS, CARS-PLS, and iSPA-PLS. The MCUVE-PLS-DA/OFF model achieved 97.3 % accuracy in fraud detection, while the iSPA-PLS-DA/OFF model reached an RMSEP of 0.16 v/v, RPD of 59.04 and r of 0.99 for alcohol content determination. These models provide a sustainable and cost-effective alternative to traditional methods, enhancing interpretability and reducing computational costs while ensuring regulatory compliance and consistency in cachaça production.

1. Introduction

Cachaça is the specific and exclusive designation for an alcoholic beverage produced exclusively in Brazil through the fermentation of sugarcane juice, possessing distinct sensory characteristics. This beverage has an alcohol content that ranges from 38 % to 48 % v/v at 20 °C and displays unique organoleptic attributes. Furthermore, Brazilian legislation permits the addition of up to 6 g/L of sugars in its composition (Fernandes et al., 2019; Oliveira et al., 2019; Oliveira et al., 2023).

In Brazil, cachaça is widely consumed and plays a significant economic role. It ranks third among the most consumed distilled beverages in the world (Lima et al., 2024). In 2023, Brazil registered an approximate production of 226 million liters of cachaça, distributed among 10,526 distinct brands, produced by 1217 distilleries registered with the Ministry of Agriculture and Livestock (MAPA). Additionally, Brazil exported 8,618,832 L of cachaça to 76 countries, generating a total revenue of USD 20,242,453, the highest value ever recorded (Brasil, 2024).

Cachaça can be produced in copper pot stills or stainless-steel columns (Serafim, Galinaro, et al., 2012). In the pot still process, the distillate is fractionated into “head” (containing excessive volatile compounds soluble in ethanol), “heart” (the actual cachaça), and “tail” (with a higher concentration of water-soluble compounds, such as acetic acid and 5-HMF) (Borges et al., 2014; Oliveira et al., 2020; Serafim, Galinaro, et al., 2012; Serafim, Silva, et al., 2012). In contrast, industrial production using stainless-steel columns achieves higher distillation efficiency and does not require fraction separation.

As with any alcoholic beverage, cachaça must meet high-quality standards to be well-accepted in the market (Hao-Jie et al., 2023; Xiao-Na et al., 2017). The beverage must comply with the parameters established by Regulation No. 539/2022 of MAPA, the Brazilian Ministry of Agriculture and Livestock (Pereira et al., 2024). These include alcohol content at 20 °C, volatile acidity, total esters, total aldehydes, furfural and hydroxymethylfurfural, higher alcohols, and toxic metals (Brasil, 2022; Oliveira et al., 2020; Onça et al., 2020; Pereira et al., 2024; Tenorio et al., 2023).

The quality of cachaça directly influences its price in the market,

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APÊNDICES

APÊNDICE A – Compostos voláteis das cachaças paraibanas não envelhecidas (tradicionais).

COV	CAS	IR Standard	IR Samples	RT (min)	Área								
					CA-01	CA-02	CA-03	CA-04	CA-05	CA-06	CA-07	CA-08	CA-09
Ethyl Acetate	141-78-6	612	651	2.06	7.10E+06	2.51E+06	1.16E+07	9.35E+06	2.53E+07	5.02E+06	2.57E+06	9.20E+06	5.68E+06
3-Methyl-1-butanol	123-51-3	736	728	3.43	3.45E+07	1.79E+07	3.90E+07	2.91E+07	2.65E+07	2.64E+07	2.77E+07	2.25E+07	2.38E+07
2-Methyl-1-butanol	137-32-6	739	733	3.49	1.16E+07	6.39E+06	1.46E+07	9.37E+06	8.65E+06	8.78E+06	1.00E+07	7.77E+06	8.34E+06
Hexanal	66-25-1	800	799	4.73	8.56E+04	5.21E+04	1.07E+05	5.06E+04	1.64E+05	2.49E+05	2.49E+05	1.60E+05	1.23E+05
Ethyl butanoate	105-54-4	802	802	4.80	4.32E+05	1.84E+05	3.06E+05	2.21E+05	2.55E+05	1.13E+05	1.26E+05	1.13E+05	1.14E+05
Isoamyl acetate	123-92-2	876	875	6.83	1.24E+06	4.37E+05	2.45E+06	1.37E+06	3.48E+06	9.63E+05	1.72E+06	1.17E+06	1.09E+06
Ethyl pentanoate	539-82-2	900	900	7.55	n.d.	1.92E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Heptanal	111-71-7	901	901	7.56	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2-Heptanol	543-49-7	901	902	7.57	6.31E+05	n.d.	1.05E+06	6.16E+05	4.96E+05	7.91E+05	6.70E+05	5.02E+05	5.68E+05
1-Octen-3-ol	3391-86-4	980	979	10.22	n.d.	n.d.	8.89E+04	n.d.	7.24E+04	n.d.	1.07E+05	5.74E+04	1.00E+05
3-Octanone	106-68-3	986	989	10.47	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl hexanoate	123-66-0	1000	1000	10.93	4.02E+06	4.13E+06	5.66E+06	5.55E+06	4.26E+06	1.30E+06	1.93E+06	2.40E+06	2.16E+06
p-Cymene	527-84-4	1022	1024	11.73	n.d.	n.d.	5.76E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Limonene	138-86-3	1030	1026	11.88	2.45E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
1-Octanol	111-87-5	1071	1070	13.51	7.22E+05	1.22E+05	8.34E+05	1.06E+06	6.72E+05	5.80E+05	5.53E+05	5.12E+05	8.99E+05
p-Cymenene	1195-32-0	1090	1087	14.08	1.33E+05	6.50E+04	1.51E+06	2.93E+05	n.d.	n.d.	6.31E+04	2.00E+05	1.79E+05
2-Nonanone	821-55-6	1092	1091	14.26	7.84E+04	2.52E+04	6.31E+04	9.90E+04	6.02E+04	8.37E+04	7.22E+04	3.57E+04	n.d.
Ethyl heptanoate	106-30-9	1097	1098	14.50	5.82E+05	4.20E+05	7.10E+05	5.67E+05	8.18E+05	2.97E+05	5.23E+05	4.53E+05	5.31E+05
2-Nonanol	628-99-9	1101	1100	14.57	4.29E+05	1.13E+05	1.02E+06	3.52E+05	4.69E+05	5.07E+05	4.62E+05	5.67E+05	4.60E+05
Nonanal	124-19-6	1104	1103	14.67	2.26E+06	3.55E+05	1.62E+06	1.03E+06	1.16E+06	1.69E+06	1.86E+06	2.17E+06	1.33E+06
Phenylethyl Alcohol	60-12-8	1116	1114	15.09	1.63E+06	1.21E+06	2.95E+06	1.41E+06	1.76E+06	5.33E+05	1.74E+06	9.37E+05	1.42E+06
Menthol	1490-04-6	1169	1163	16.81	3.74E+05	n.d.	1.80E+06	3.60E+05	n.d.	n.d.	6.28E+05	5.15E+05	n.d.
L-menthol	2216-51-5	1175	1171	17.11	4.79E+06	n.d.	1.36E+07	3.15E+06	n.d.	3.14E+06	5.14E+06	4.61E+06	n.d.
Ethyl octanoate	106-32-1	1196	1197	18.04	1.06E+08	7.11E+07	8.78E+07	8.73E+07	6.66E+07	4.33E+07	7.21E+07	8.16E+07	5.13E+07
Decanal	112-31-2	1206	1204	18.29	5.26E+05	1.62E+05	4.46E+05	3.48E+05	3.42E+05	2.91E+05	3.23E+05	2.77E+05	1.95E+05

β-Cyclocitral	432-25-7	1220	1221	18.78	3.71E+05	n.d.	2.73E+05	3.52E+05	n.d.	3.50E+05	1.27E+05	2.60E+05	3.06E+05
Citronellol	106-22-9	1228	1228	19.11	7.88E+05	2.60E+05	1.08E+06	3.76E+05	2.77E+05	7.08E+05	4.84E+05	5.13E+05	4.61E+05
Phenethyl acetate	103-45-7	1258	1256	20.08	n.d.	1.10E+06	3.09E+06	1.94E+06	1.65E+06	1.02E+06	1.65E+06	2.23E+06	1.57E+06
2-Undecanone	112-12-9	1294	1293	21.32	1.25E+06	n.d.	n.d.	n.d.	1.37E+06	8.15E+05	4.83E+05	1.03E+06	n.d.
Ethyl nonanoate	123-29-5	1296	1295	21.43	6.91E+06	2.89E+06	4.43E+06	1.75E+06	2.50E+06	2.45E+06	3.07E+06	5.49E+06	2.59E+06
2-Undecanol	1653-30-1	1308	1300	21.58	6.34E+05	n.d.	4.63E+05	1.45E+05	4.45E+05	3.52E+05	3.16E+05	5.24E+05	3.80E+05
Undecanal	112-44-7	1307	1306	21.76	4.00E+05	1.89E+05	n.d.	1.30E+05	n.d.	n.d.	2.25E+05	n.d.	n.d.
δ-Ellemene	20307-84-0	1338	1336	22.76	3.27E+05	4.54E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Eugenol	97-53-0	1357	1357	23.78	n.d.	n.d.	2.36E+05	4.21E+05	n.d.	6.35E+04	3.21E+05	n.d.	1.10E+05
β-Damascenone	23726-93-4	1386	1383	24.31	2.06E+06	1.06E+06	3.96E+06	2.18E+06	1.83E+06	1.58E+06	2.23E+06	3.04E+06	2.56E+06
β-Elemene	515-13-9	1391	1390	24.53	1.52E+06	1.37E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	3.13E+08	1.90E+08	2.31E+08	1.47E+08	1.24E+08	1.44E+08	1.85E+08	2.30E+08	1.40E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	6.56E+06	3.38E+06	5.11E+06	1.80E+06	8.17E+05	1.33E+06	2.86E+06	6.57E+06	1.69E+06
(E)-β-Farnesene	18794-84-8	1457	1456	26.56	1.41E+06	1.27E+06	1.77E+06	5.23E+05	7.25E+05	4.67E+05	1.08E+06	1.34E+06	6.48E+05
1-Dodecanol	112-53-8	1473	1473	27.09	9.74E+05	n.d.	1.23E+06	6.68E+05	9.19E+05	3.10E+05	5.46E+05	6.10E+05	4.13E+05
γ-Muurolene	30021-74-0	1477	1477	27.17	n.d.	3.13E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
α-Curcumene	644-30-4	1483	1481	27.36	3.85E+05	7.54E+06	1.98E+04	n.d.	5.84E+04	n.d.	5.59E+04	n.d.	n.d.
Ethyl undecanoate	627-90-7	1494	1494	27.74	n.d.	n.d.	1.65E+06	5.25E+05	8.56E+05	8.26E+05	1.06E+06	2.40E+06	1.02E+06
α-Muurolene	10208-80-7	1499	1499	27.89	8.51E+06	5.16E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cuparene	16982-00-6	1505	1504	28.06	4.60E+05	3.09E+05	2.25E+05	n.d.	n.d.	n.d.	n.d.	1.20E+05	n.d.
α-Farnesene	502-61-4	1508	1507	28.13	1.06E+06	1.07E+06	1.07E+06	2.68E+05	3.90E+05	1.43E+05	4.30E+05	3.35E+05	1.48E+05
γ-Cadinene	39029-41-9	1513	1513	28.31	5.78E+05	5.40E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
δ-Cadinene	483-76-1	1524	1522	28.59	3.42E+07	2.61E+07	n.d.	n.d.	4.85E+05	n.d.	n.d.	n.d.	n.d.
α-Calacorene	21391-99-1	1542	1542	29.16	3.91E+06	4.15E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	1.75E+07	1.05E+07	1.91E+07	5.97E+06	9.75E+06	5.48E+06	1.45E+07	8.22E+06	7.80E+06
gleenol		1587	1584	30.4	1.81E+06	2.32E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl laurate	106-33-2	1595	1594	30.68	1.60E+08	1.26E+08	1.42E+08	6.88E+07	5.57E+07	7.08E+07	9.54E+07	1.06E+08	8.42E+07
α-Corocalene	20129-39-9	1623	1624	31.47	2.38E+06	1.86E+06	1.28E+05	n.d.	n.d.	n.d.	n.d.	1.01E+05	6.68E+04
Epicubenol	19912-67-5	1627	1631	31.68	2.74E+06	2.42E+06	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Cadalene	483-78-3	1674	1679	32.92	2.58E+07	2.41E+07	6.69E+05	n.d.	1.02E+06	n.d.	4.62E+05	3.13E+05	n.d.
2,3-Dihydrofarnesol	51411-24-6	1696	1696	33.34	4.87E+06	3.28E+06	3.03E+06	8.30E+06	9.26E+05	2.58E+06	1.71E+06	1.74E+06	1.11E+06
trans-Farnesol	4602-84-0	1722	1727	34.24	1.66E+06	1.45E+06	2.40E+06	5.38E+05	6.49E+05	7.39E+05	1.14E+06	1.29E+06	6.75E+05
Farnesol formate	917105-98-7	1737	1745	34.74	1.97E+06	1.03E+06	1.50E+06	n.d.	1.52E+06	n.d.	5.56E+05	7.75E+05	5.04E+05
Ethyl myristate	124-06-1	1794	1793	36.08	3.37E+07	1.05E+07	1.95E+07	2.70E+06	7.87E+06	9.53E+06	8.04E+06	1.89E+07	1.14E+07
2,3-Dihydro farnesyl acetate	58130-58-8	1823	1808	36.47	6.88E+05	1.05E+06	n.d.	3.70E+05	n.d.	3.94E+05	3.63E+05	7.05E+05	1.78E+05
Farnesyl acetate, (E,E)-	4128-17-0	1843	1841	37.31	n.d.	1.97E+06	3.75E+05	7.57E+05	1.02E+05	6.21E+05	1.03E+06	1.21E+06	7.04E+05
Ethyl palmitate	628-97-7	1993	1992	41.03	4.26E+07	2.13E+07	5.25E+07	2.86E+06	2.59E+07	1.10E+07	2.45E+06	2.40E+07	1.67E+07
Ethyl linoleate	544-35-4	2162		44.84	2.73E+06	6.54E+05	5.92E+06	3.20E+05	8.30E+05	6.36E+05	2.08E+05	2.62E+06	1.42E+06
Ethyl Oleate	111-62-6	2173		44.97	1.74E+06	3.20E+05	2.14E+06	n.d.	1.12E+06	n.d.	n.d.	9.93E+05	5.20E+05

COV	CAS	IR Standard	IR Samples	RT (min)	Área								
					CA-10	CA-11	CA-12	CA-13	CA-14	CA-15	CA-16	CA-17	CA-18
Ethyl Acetate	141-78-6	612	651	2.06	4.81E+06	4.18E+06	7.01E+06	3.48E+06	1.86E+06	9.53E+06	1.01E+07	3.68E+06	3.42E+06
3-Methyl-1-butanol	123-51-3	736	728	3.43	2.02E+07	3.91E+07	1.89E+07	1.62E+07	1.49E+07	1.88E+07	8.94E+06	3.24E+07	9.86E+06
2-Methyl-1-butanol	137-32-6	739	733	3.49	6.25E+06	1.86E+07	7.58E+06	6.18E+06	8.03E+06	6.79E+06	8.91E+06	1.24E+07	6.63E+04
Hexanal	66-25-1	800	799	4.73	1.79E+05	1.81E+05	5.57E+04	2.95E+04	5.54E+04	2.38E+05	2.77E+04	1.14E+05	4.21E+04
Ethyl butanoate	105-54-4	802	802	4.80	2.83E+05	2.42E+05	1.46E+05	7.52E+04	3.00E+04	2.82E+05	1.94E+05	1.27E+05	1.36E+05
Isoamyl acetate	123-92-2	876	875	6.83	2.23E+06	5.17E+06	1.54E+06	6.19E+05	4.72E+05	1.02E+06	1.22E+06	1.22E+06	2.13E+06
Ethyl pentanoate	539-82-2	900	900	7.55	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.39E+05
Heptanal	111-71-7	901	901	7.56	n.d.	7.55E+05	n.d.	1.72E+05	2.44E+05	n.d.	n.d.	3.15E+05	n.d.
2-Heptanol	543-49-7	901	902	7.57	7.77E+05	n.d.	7.34E+05	n.d.	n.d.	5.44E+05	n.d.	n.d.	n.d.
1-Octen-3-ol	3391-86-4	980	979	10.22	8.22E+04	1.60E+05	5.25E+04	4.12E+04	n.d.	5.19E+04	n.d.	n.d.	n.d.
3-Octanone	106-68-3	986	989	10.47	5.19E+04	1.13E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl hexanoate	123-66-0	1000	1000	10.93	4.08E+06	4.79E+06	3.95E+06	9.97E+05	3.55E+06	3.58E+06	1.04E+06	3.56E+06	4.98E+06
p-Cymene	527-84-4	1022	1024	11.73	n.d.	n.d.	n.d.	4.47E+04	n.d.	n.d.	1.19E+06	n.d.	7.11E+04
Limonene	138-86-3	1030	1026	11.88	3.82E+05	n.d.	n.d.	n.d.	3.40E+04	n.d.	1.15E+06	n.d.	4.59E+04
1-Octanol	111-87-5	1071	1070	13.51	7.02E+05	7.45E+05	3.23E+05	4.41E+05	3.57E+05	2.32E+05	3.43E+05	2.75E+05	2.41E+05

p-Cymenene	1195-32-0	1090	1087	14.08	1.66E+05	3.94E+05	n.d.	2.99E+05	6.26E+04	n.d.	9.01E+05	n.d.	1.02E+05
2-Nonanone	821-55-6	1092	1091	14.26	1.11E+05	1.12E+05	3.68E+04	4.08E+04	3.69E+04	5.64E+04	n.d.	2.68E+04	2.15E+04
Ethyl heptanoate	106-30-9	1097	1098	14.50	6.69E+05	7.77E+05	3.05E+05	2.15E+05	2.47E+05	4.02E+05	3.95E+05	3.99E+05	3.36E+05
2-Nonanol	628-99-9	1101	1100	14.57	4.92E+05	4.21E+05	6.54E+05	3.65E+05	2.46E+05	5.36E+05	1.19E+05	1.10E+05	1.05E+05
Nonanal	124-19-6	1104	1103	14.67	1.87E+06	2.29E+06	3.40E+05	9.55E+05	6.37E+05	7.97E+05	5.38E+05	1.91E+06	1.28E+06
Phenylethyl Alcohol	60-12-8	1116	1114	15.09	2.15E+05	3.43E+06	n.d.	1.05E+06	4.95E+04	7.67E+05	5.88E+06	7.61E+05	2.46E+06
Menthol	1490-04-6	1169	1163	16.81	n.d.	1.76E+05	n.d.	4.05E+05	1.10E+05	n.d.	1.43E+06	n.d.	3.48E+05
L-menthol	2216-51-5	1175	1171	17.11	n.d.	n.d.	n.d.	3.53E+06	1.79E+06	1.25E+06	6.08E+06	n.d.	3.41E+06
Ethyl octanoate	106-32-1	1196	1197	18.04	9.33E+07	8.53E+07	5.74E+07	4.49E+07	1.99E+07	6.89E+07	3.65E+07	9.49E+07	8.59E+07
Decanal	112-31-2	1206	1204	18.29	2.64E+05	3.97E+05	1.37E+05	1.77E+05	1.49E+05	2.81E+05	1.58E+05	2.77E+05	3.74E+05
β -Cyclocitral	432-25-7	1220	1221	18.78	2.71E+05	2.82E+05	n.d.	3.13E+05	n.d.	n.d.	n.d.	1.33E+05	1.11E+05
Citronellol	106-22-9	1228	1228	19.11	5.49E+05	6.64E+05	3.20E+05	3.61E+05	5.86E+05	3.90E+05	4.20E+05	4.07E+05	n.d.
Phenethyl acetate	103-45-7	1258	1256	20.08	1.36E+06	5.53E+06	n.d.	9.47E+05	7.26E+05	7.56E+05	3.32E+07	9.57E+05	1.72E+06
2-Undecanone	112-12-9	1294	1293	21.32	9.46E+05	8.15E+05	n.d.	4.27E+05	1.68E+05	5.29E+05	1.25E+05	8.63E+05	n.d.
Ethyl nonanoate	123-29-5	1296	1295	21.43	6.88E+06	6.90E+06	8.30E+05	3.97E+06	1.76E+06	2.21E+06	1.53E+06	6.59E+06	3.93E+06
2-Undecanol	1653-30-1	1308	1300	21.58	5.59E+05	4.46E+05	1.73E+05	5.85E+05	1.89E+05	5.53E+05	n.d.	n.d.	1.86E+05
Undecanal	112-44-7	1307	1306	21.76	3.04E+05	n.d.	n.d.	n.d.	n.d.	1.93E+05	n.d.	n.d.	1.43E+05
δ -Elemene	20307-84-0	1338	1336	22.76	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Eugenol	97-53-0	1357	1357	23.78	n.d.	n.d.	1.30E+05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
β -Damascenone	23726-93-4	1386	1383	24.31	2.49E+06	3.00E+06	1.62E+06	1.25E+06	1.88E+06	1.04E+06	2.27E+06	2.93E+06	6.37E+06
β -Elemene	515-13-9	1391	1390	24.53	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	2.13E+08	2.19E+08	1.15E+08	1.44E+08	8.17E+07	1.57E+08	9.40E+07	2.27E+08	2.24E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	5.35E+06	4.92E+06	9.83E+05	3.36E+06	1.14E+06	1.21E+06	1.39E+06	8.49E+06	7.00E+06
(E)- β -Farnesene	18794-84-8	1457	1456	26.56	7.72E+05	1.61E+06	5.62E+05	1.13E+06	2.93E+06	8.09E+05	9.04E+05	1.56E+06	1.65E+06
1-Dodecanol	112-53-8	1473	1473	27.09	8.37E+05	1.38E+06	n.d.	n.d.	n.d.	2.40E+05	n.d.	n.d.	n.d.
γ -Muurolene	30021-74-0	1477	1477	27.17	n.d.	n.d.	n.d.	n.d.	n.d.	1.60E+05	n.d.	4.40E+05	n.d.
α -Curcumene	644-30-4	1483	1481	27.36	n.d.	6.85E+04	n.d.	9.20E+04	2.93E+05	3.93E+04	3.21E+04	7.52E+04	5.25E+05
Ethyl undecanoate	627-90-7	1494	1494	27.74	1.97E+06	2.03E+06	3.23E+05	1.70E+06	n.d.	1.27E+06	7.75E+05	2.19E+06	1.66E+06
α -Muurolene	10208-80-7	1499	1499	27.89	n.d.	n.d.	n.d.	n.d.	n.d.	2.64E+05	n.d.	n.d.	n.d.

Cuparene	16982-00-6	1505	1504	28.06	4.54E+05	n.d.	n.d.	2.55E+05	n.d.	n.d.	n.d.	2.38E+05	n.d.
α -Farnesene	502-61-4	1508	1507	28.13	4.78E+05	9.48E+05	3.37E+05	7.61E+05	1.88E+06	4.98E+05	5.72E+05	8.06E+05	1.10E+06
γ -Cadinene	39029-41-9	1513	1513	28.31	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
δ -Cadinene	483-76-1	1524	1522	28.59	n.d.	n.d.	n.d.	1.16E+05	n.d.	7.41E+05	n.d.	2.64E+06	n.d.
α -Calacorene	21391-99-1	1542	1542	29.16	n.d.	n.d.	n.d.	n.d.	n.d.	2.18E+05	n.d.	5.89E+05	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	1.04E+07	1.32E+07	4.20E+06	3.87E+06	7.37E+06	7.14E+06	6.58E+06	1.30E+07	3.69E+06
gleenol		1587	1584	30.4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl laurate	106-33-2	1595	1594	30.68	9.98E+07	1.12E+08	6.11E+07	7.15E+07	3.70E+07	8.19E+07	4.13E+07	1.17E+08	1.15E+08
α -Corocalene	20129-39-9	1623	1624	31.47	9.54E+04	1.46E+05	6.59E+04	n.d.	n.d.	7.08E+04	3.35E+04	2.88E+05	n.d.
Epicubenol	19912-67-5	1627	1631	31.68	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6.71E+05	n.d.
Cadalene	483-78-3	1674	1679	32.92	2.21E+05	n.d.	1.36E+05	2.99E+05	n.d.	1.86E+06	1.53E+05	3.58E+06	3.08E+05
2,3-Dihydrofarnesol	51411-24-6	1696	1696	33.34	9.53E+05	2.07E+06	8.90E+05	n.d.	1.76E+06	5.48E+05	6.22E+05	1.99E+06	1.13E+06
trans-Farnesol	4602-84-0	1722	1727	34.24	7.37E+05	7.40E+05	3.50E+05	n.d.	6.72E+05	6.36E+05	5.41E+05	1.07E+06	3.05E+05
Farnesol formate	917105-98-7	1737	1745	34.74	8.02E+05	1.05E+06	4.07E+05	4.25E+05	4.15E+05	4.63E+05	5.91E+05	1.01E+06	5.40E+05
Ethyl myristate	124-06-1	1794	1793	36.08	3.61E+04	9.54E+06	4.65E+06	1.22E+07	1.37E+06	8.69E+06	1.81E+06	1.34E+07	9.61E+06
2,3-Dihydro farnesyl acetate	58130-58-8	1823	1808	36.47	2.66E+05	2.20E+06	1.57E+05	n.d.	7.26E+05	1.24E+05	4.98E+04	n.d.	6.07E+05
Farnesyl acetate, (E,E)-	4128-17-0	1843	1841	37.31	1.43E+06	4.33E+06	2.78E+05	n.d.	1.03E+06	6.73E+05	3.88E+05	n.d.	1.75E+06
Ethyl palmitate	628-97-7	1993	1992	41.03	1.64E+07	3.06E+06	1.23E+07	6.30E+06	6.47E+05	1.20E+07	7.32E+06	3.51E+07	1.12E+07
Ethyl linoleate	544-35-4	2162		44.84	8.39E+05	3.13E+05	6.15E+05	2.21E+05	7.17E+04	1.43E+05	7.93E+05	9.28E+05	2.58E+05
Ethyl Oleate	111-62-6	2173		44.97	2.89E+05	n.d.	n.d.	n.d.	n.d.	1.02E+05	2.14E+05	5.37E+05	1.33E+05
COV	CAS	IR Standard	IR Samples	RT (min)	Área								
					CA-19	CA-20	CM-01	CM-02	CM-03	CM-04	CS-01	CS-02	
Ethyl Acetate	141-78-6	612	651	2.06	6.23E+06	7.23E+06	2.07E+06	1.99E+06	1.49E+06	3.54E+06	1.69E+06	1.44E+06	
3-Methyl-1-butanol	123-51-3	736	728	3.43	2.09E+07	2.40E+07	2.97E+07	3.50E+07	1.71E+07	2.78E+07	1.47E+07	2.01E+07	
2-Methyl-1-butanol	137-32-6	739	733	3.49	7.61E+06	8.30E+06	1.00E+07	1.62E+07	7.02E+06	1.05E+07	6.05E+06	7.76E+06	
Hexanal	66-25-1	800	799	4.73	9.89E+04	9.94E+04	2.07E+05	8.44E+04	3.80E+04	1.08E+05	2.92E+04	1.38E+04	
Ethyl butanoate	105-54-4	802	802	4.80	4.15E+05	4.61E+05	1.50E+05	5.20E+05	1.03E+05	1.75E+05	1.78E+05	4.57E+04	

[illegible]

β -Damascenone	23726-93-4	1386	1383	24.31	5.24E+05	1.31E+06	2.17E+06	1.80E+06	1.45E+06	2.22E+06	1.06E+06	1.43E+06
β -Elemene	515-13-9	1391	1390	24.53	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	8.25E+07	1.60E+08	1.89E+08	2.20E+08	1.21E+08	1.45E+08	1.27E+08	1.57E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	4.15E+05	2.33E+06	4.23E+06	8.95E+06	3.25E+06	3.25E+06	1.56E+06	3.37E+06
(E)- β -Farnesene	18794-84-8	1457	1456	26.56	1.82E+05	9.27E+05	2.81E+06	2.33E+06	9.85E+05	2.96E+06	7.44E+05	1.67E+06
1-Dodecanol	112-53-8	1473	1473	27.09	7.74E+04	n.d.	n.d.	5.63E+05	n.d.	n.d.	n.d.	n.d.
γ -Muurolene	30021-74-0	1477	1477	27.17	n.d.	4.06E+05	6.70E+05	n.d.	n.d.	3.92E+05	n.d.	n.d.
α -Curcumene	644-30-4	1483	1481	27.36	n.d.	5.46E+04	2.63E+05	1.13E+05	7.59E+04	1.74E+05	8.01E+04	1.69E+05
Ethyl undecanoate	627-90-7	1494	1494	27.74	3.16E+05	1.89E+06	3.81E+06	3.52E+06	1.30E+06	2.68E+06	5.55E+05	1.07E+06
α -Muurolene	10208-80-7	1499	1499	27.89	n.d.	2.69E+05	n.d.	n.d.	n.d.	4.91E+05	n.d.	n.d.
Cuparene	16982-00-6	1505	1504	28.06	n.d.	1.89E+05	6.99E+05	n.d.	4.90E+05	n.d.	n.d.	n.d.
α -Farnesene	502-61-4	1508	1507	28.13	n.d.	4.80E+05	2.26E+06	1.24E+06	8.74E+05	2.21E+06	6.24E+05	1.01E+06
γ -Cadinene	39029-41-9	1513	1513	28.31	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
δ -Cadinene	483-76-1	1524	1522	28.59	n.d.	1.03E+06	6.91E+05	n.d.	n.d.	n.d.	7.17E+04	n.d.
α -Calacorene	21391-99-1	1542	1542	29.16	n.d.	3.79E+05	6.58E+05	3.76E+05	2.78E+05	9.58E+05	n.d.	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	1.52E+06	7.44E+06	2.46E+07	1.50E+07	2.67E+06	2.41E+07	4.70E+06	4.94E+06
gleenol		1587	1584	30.4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl laurate	106-33-2	1595	1594	30.68	5.40E+07	8.34E+07	1.32E+08	1.09E+08	6.63E+07	7.29E+07	5.51E+07	9.54E+07
α -Corocalene	20129-39-9	1623	1624	31.47	n.d.	1.46E+05	2.30E+05	n.d.	8.11E+04	3.05E+05	5.78E+04	n.d.
Epicubenol	19912-67-5	1627	1631	31.68	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cadalene	483-78-3	1674	1679	32.92	1.17E+05	1.14E+06	4.92E+05	3.60E+05	2.10E+05	3.44E+05	1.56E+05	2.18E+05
2,3-Dihydrofarnesol	51411-24-6	1696	1696	33.34	8.33E+04	8.73E+05	4.75E+05	5.92E+06	n.d.	1.81E+06	3.37E+06	n.d.
trans-Farnesol	4602-84-0	1722	1727	34.24	1.40E+05	4.77E+05	5.03E+05	2.26E+06	n.d.	9.34E+05	1.63E+05	4.81E+05
Farnesol formate	917105-98-7	1737	1745	34.74	2.05E+05	1.02E+06	1.58E+06	1.07E+06	n.d.	2.47E+06	4.46E+05	8.70E+05
Ethyl myristate	124-06-1	1794	1793	36.08	2.69E+07	1.08E+07	3.19E+07	1.56E+07	8.87E+06	6.93E+06	1.11E+06	5.71E+06
2,3-Dihydro farnesyl acetate	58130-58-8	1823	1808	36.47	n.d.	3.06E+05	1.15E+05	3.89E+06	1.92E+05	9.60E+04	1.49E+06	3.78E+05
Farnesyl acetate, (E,E)-	4128-17-0	1843	1841	37.31	n.d.	1.51E+06	9.24E+05	4.68E+06	1.51E+06	4.95E+05	2.93E+05	n.d.

Ethyl palmitate	628-97-7	1993	1992	41.03	3.54E+07	2.35E+07	5.01E+07	1.84E+07	2.94E+07	1.75E+07	1.86E+05	9.38E+06
Ethyl linoleate	544-35-4	2162		44.84	3.07E+06	3.42E+05	3.62E+06	1.02E+06	4.36E+05	9.57E+05	n.d.	1.88E+05
Ethyl Oleate	111-62-6	2173		44.97	1.37E+06	3.56E+05	1.62E+06	4.50E+05	8.40E+05	3.48E+05	n.d.	1.55E+05

n.d. – not determined

APÊNDICE B – Compostos voláteis das cachaças paraibanas armazenadas em barris de Amburana (*Amburana cearenses*).

COV	CAS	IR Standard	IR Samples	RT (min)	Área						
					UA-01	UA-02	UA-03	UA-04	UA-05	UA-06	UA-07
Ethyl Acetate	141-78-6	612	651	2.06	3.50E+06	2.05E+06	3.88E+06	3.59E+06	4.80E+06	8.28E+06	4.89E+06
3-Methyl-1-butanol	123-51-3	736	728	3.43	1.89E+07	2.00E+07	1.53E+07	6.25E+06	1.81E+07	1.29E+07	1.32E+07
2-Methyl-1-butanol	137-32-6	739	733	3.49	6.17E+06	7.24E+06	5.11E+06	3.46E+04	6.75E+06	3.67E+06	5.14E+06
Hexanal	66-25-1	800	799	4.73	8.25E+04	8.09E+04	6.94E+04	5.67E+04	1.17E+05	8.66E+04	3.40E+04
Ethyl butanoate	105-54-4	802	802	4.80	5.56E+04	5.17E+04	7.03E+04	6.51E+04	1.58E+05	1.63E+05	1.07E+05
Isoamyl acetate	123-92-2	876	875	6.83	3.69E+05	5.90E+05	5.92E+05	5.40E+05	1.40E+06	1.36E+06	1.11E+06
2-Heptanol	543-49-7	901	901	7.56	4.13E+05	2.73E+05	3.46E+05	3.56E+05	4.51E+05	6.26E+05	4.60E+05
Ethyl hexanoate	123-66-0	1000	1000	10.93	1.02E+06	4.32E+05	1.42E+06	1.45E+06	2.33E+06	3.37E+06	3.29E+06
p-Cymene	99-87-6	1022	1022	11.74	n.d.	n.d.	n.d.	n.d.	8.87E+04	n.d.	n.d.
Limonene	138-86-3	1030	1026	11.88	n.d.	n.d.	n.d.	n.d.	3.28E+05	n.d.	n.d.
1-Octanol	111-87-5	1071	1070	13.51	2.65E+05	2.42E+05	4.12E+05	5.10E+05	3.12E+05	2.61E+05	2.55E+05
2-Nonanone	821-55-6	1092	1091	14.26	6.54E+04	1.39E+05	6.54E+04	3.83E+04	8.34E+04	6.05E+04	2.71E+04
Ethyl heptanoate	106-30-9	1097	1098	14.50	2.63E+05	n.d.	3.17E+05	3.28E+05	3.86E+05	3.23E+05	1.97E+05
Linalool	78-70-6	1099	1099	14.52	n.d.	3.05E+05	n.d.	n.d.	n.d.	n.d.	n.d.
2-Nonanol	628-99-9	1101	1100	14.57	3.07E+05	2.39E+05	1.89E+05	2.08E+05	2.91E+05	4.89E+05	4.26E+05
Nonanal	124-19-6	1104	1103	14.67	9.53E+05	1.15E+06	1.31E+06	8.79E+05	1.28E+06	1.77E+06	3.56E+05
Phenylethyl Alcohol	60-12-8	1116	1114	15.09	8.29E+05	1.42E+06	2.97E+05	9.34E+05	1.08E+06	4.54E+05	1.31E+05
Menthol	1490-04-6	1169	1163	16.81	n.d.	2.01E+05	1.33E+05	n.d.	2.04E+05	n.d.	n.d.
Ethyl octanoate	106-32-1	1196	1197	18.04	3.92E+07	3.95E+07	5.37E+07	4.32E+07	6.61E+07	7.27E+07	5.74E+07
Decanal	112-31-2	1206	1204	18.29	1.82E+05	2.86E+05	3.36E+05	2.89E+05	4.76E+05	3.83E+05	3.12E+05
β-Cyclocitral	432-25-7	1220	1221	18.78	n.d.	n.d.	n.d.	2.50E+05	1.20E+06	n.d.	n.d.
Citronellol	106-22-9	1228	1228	19.11	3.13E+05	4.19E+05	3.10E+05	3.00E+05	2.62E+05	1.84E+05	2.16E+05
Ethyl phenylacetate	101-97-3	1246	1244	19.67	8.09E+04	6.45E+04	7.96E+04	9.00E+04	1.35E+05	4.34E+04	8.01E+04
Phenethyl acetate	103-45-7	1258	1256	20.08	9.65E+05	7.51E+05	5.26E+05	6.55E+05	7.04E+05	6.29E+05	4.84E+05
Ethyl nonanoate	123-29-5	1296	1295	21.43	2.50E+06	2.46E+06	3.69E+06	2.08E+06	4.52E+06	4.38E+06	1.07E+06

2-Undecanol	1653-30-1	1308	1300	21.58	2.71E+05	2.79E+05	2.26E+05	2.03E+05	4.09E+05	3.14E+05	1.65E+05
Undecanal	112-44-7	1307	1306	21.76	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
β -Damascenone	23726-93-4	1386	1383	24.31	1.38E+06	1.62E+06	2.33E+06	1.73E+06	1.73E+06	1.95E+06	1.08E+06
β -Elemene	515-13-9	1391	1390	24.53	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	1.29E+08	1.35E+08	1.67E+08	1.15E+08	1.57E+08	1.70E+08	1.27E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	1.16E+06	1.91E+06	3.93E+06	9.27E+05	2.71E+06	2.20E+06	1.65E+06
Geranyl acetone	3796-70-1	1453	1452	26.45	9.96E+04	1.73E+05	1.96E+05	n.d.	n.d.	n.d.	n.d.
(E)- β -Farnesene	18794-84-8	1457	1456	26.56	6.09E+05	1.85E+06	8.51E+05	7.81E+05	9.61E+05	3.73E+05	4.89E+05
α -Curcumene	644-30-4	1483	1481	27.36	7.41E+04	1.33E+05	3.72E+04	3.35E+04	3.62E+04	n.d.	n.d.
(E)- β -Ionone	79-77-6	1486	1486	27.47	2.17E+04	2.73E+04	1.31E+05	2.46E+04	1.27E+05	1.96E+05	1.81E+04
α -Muurolene	10208-80-7	1499	1499	27.89	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
α -Farnesene	502-61-4	1508	1507	28.13	3.22E+05	1.22E+06	3.87E+05	5.18E+05	6.36E+05	2.81E+05	3.70E+05
δ -Cadinene	483-76-1	1524	1522	28.59	n.d.	n.d.	1.93E+05	9.33E+04	1.88E+05	1.21E+05	1.04E+05
α -Calacorene	21391-99-1	1542	1542	29.16	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	4.74E+06	1.29E+07	4.00E+06	5.76E+06	7.84E+06	2.31E+06	3.06E+06
Ethyl laurate	106-33-2	1595	1594	30.68	4.89E+07	6.03E+07	8.35E+07	5.77E+07	6.94E+07	6.85E+07	6.61E+07
Bisabolol	515-69-5	1684	1683	33.17	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Farnesol	4602-84-0	1722	1727	34.24	4.35E+05	9.80E+05	4.36E+05	3.07E+05	2.85E+05	8.24E+04	1.34E+05
Ethyl palmitate	628-97-7	1993	1992	41.03	3.54E+06	1.50E+06	1.34E+07	7.66E+06	8.59E+06	1.04E+07	1.01E+07
Ethyl linoleate	544-35-4	2162		44.84	2.06E+05	7.67E+04	8.98E+05	3.11E+05	1.87E+05	3.66E+05	1.92E+05
COV	CAS	IR Standard	IR Samples	RT (min)	Área						
					UA-08	UA-09	UA-10	UA-11	UA-12	UA-13	UA-14
Ethyl Acetate	141-78-6	612	651	2.06	3.63E+06	2.82E+06	3.35E+06	3.72E+06	7.19E+06	5.23E+06	6.80E+06
3-Methyl-1-butanol	123-51-3	736	728	3.43	1.12E+07	1.21E+07	9.16E+06	2.19E+07	1.15E+07	2.45E+07	1.11E+07
2-Methyl-1-butanol	137-32-6	739	733	3.49	5.30E+06	4.49E+06	4.29E+06	8.33E+06	3.75E+06	9.04E+06	3.92E+06
Hexanal	66-25-1	800	799	4.73	4.59E+04	2.60E+04	3.99E+04	6.65E+04	6.39E+04	7.55E+04	1.47E+05
Ethyl butanoate	105-54-4	802	802	4.80	5.16E+04	4.46E+04	6.00E+04	9.27E+04	1.98E+05	2.97E+05	1.87E+05
Isoamyl acetate	123-92-2	876	875	6.83	4.93E+05	4.88E+05	8.01E+05	8.54E+05	7.71E+05	2.47E+05	4.61E+04

2-Heptanol	543-49-7	901	901	7.56	2.87E+05	1.21E+05	1.69E+05	2.49E+05	3.51E+05	4.01E+05	3.53E+05
Ethyl hexanoate	123-66-0	1000	1000	10.93	1.59E+06	3.88E+05	1.60E+06	3.13E+06	1.01E+06	4.06E+06	3.34E+06
p-Cymene	99-87-6	1022	1022	11.74	n.d.	n.d.	3.96E+04	n.d.	7.58E+04	n.d.	n.d.
Limonene	138-86-3	1030	1026	11.88	3.11E+04	n.d.	3.05E+04	1.00E+05	6.35E+04	n.d.	n.d.
1-Octanol	111-87-5	1071	1070	13.51	3.25E+05	4.50E+05	1.66E+05	1.72E+05	7.64E+04	6.42E+05	1.33E+05
2-Nonanone	821-55-6	1092	1091	14.26	3.89E+04	4.91E+04	5.02E+04	2.82E+04	8.39E+04	1.07E+05	4.70E+04
Ethyl heptanoate	106-30-9	1097	1098	14.50	1.55E+05	1.61E+05	n.d.	3.36E+05	6.04E+05	1.01E+06	3.19E+05
Linalool	78-70-6	1099	1099	14.52	n.d.	n.d.	2.00E+05	n.d.	n.d.	n.d.	n.d.
2-Nonanol	628-99-9	1101	1100	14.57	3.48E+05	2.73E+05	1.58E+05	7.30E+04	2.69E+05	1.72E+05	3.36E+05
Nonanal	124-19-6	1104	1103	14.67	6.31E+05	9.11E+05	7.91E+05	1.20E+06	1.37E+06	1.22E+06	7.56E+05
Phenylethyl Alcohol	60-12-8	1116	1114	15.09	5.05E+05	2.86E+05	5.36E+05	1.34E+06	1.23E+06	2.39E+06	8.89E+05
Menthol	1490-04-6	1169	1163	16.81	9.53E+04	3.14E+05	4.24E+04	n.d.	2.99E+05	1.72E+05	n.d.
Ethyl octanoate	106-32-1	1196	1197	18.04	4.29E+07	2.72E+07	3.38E+07	7.47E+07	3.10E+07	7.43E+07	6.02E+07
Decanal	112-31-2	1206	1204	18.29	2.88E+05	1.14E+05	1.64E+05	4.96E+05	6.09E+05	5.67E+05	5.27E+05
β -Cyclocitral	432-25-7	1220	1221	18.78	n.d.	2.14E+05	n.d.	1.20E+05	1.24E+05	1.11E+05	n.d.
Citronellol	106-22-9	1228	1228	19.11	2.85E+05	1.76E+05	1.28E+05	2.90E+05	1.46E+05	2.64E+05	2.24E+05
Ethyl phenylacetate	101-97-3	1246	1244	19.67	3.71E+04	5.24E+04	5.74E+04	9.48E+05	8.11E+04	1.07E+05	4.80E+04
Phenethyl acetate	103-45-7	1258	1256	20.08	9.49E+05	6.88E+05	4.56E+05	8.24E+05	6.25E+05	9.24E+05	5.17E+05
Ethyl nonanoate	123-29-5	1296	1295	21.43	1.78E+06	2.25E+06	1.43E+06	5.03E+06	2.31E+06	7.67E+06	2.17E+06
2-Undecanol	1653-30-1	1308	1300	21.58	2.67E+05	3.18E+05	n.d.	2.81E+05	1.42E+05	3.38E+05	3.84E+05
Undecanal	112-44-7	1307	1306	21.76	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
β -Damascenone	23726-93-4	1386	1383	24.31	1.42E+06	9.90E+05	7.72E+05	2.10E+06	6.47E+05	1.71E+06	8.47E+05
β -Elemene	515-13-9	1391	1390	24.53	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	1.33E+08	8.84E+07	8.70E+07	1.87E+08	6.12E+07	1.81E+08	1.37E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	3.29E+06	1.29E+06	1.43E+06	5.80E+06	1.75E+05	2.90E+06	1.15E+06
Geranyl acetone	3796-70-1	1453	1452	26.45	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
(E)- β -Farnesene	18794-84-8	1457	1456	26.56	1.01E+06	6.51E+05	7.51E+05	1.51E+06	2.84E+05	1.20E+06	5.88E+05
α -Curcumene	644-30-4	1483	1481	27.36	3.88E+04	5.75E+04	5.41E+04	1.07E+05	n.d.	1.37E+05	7.74E+04
(E)- β -Ionone	79-77-6	1486	1486	27.47	3.34E+04	n.d.	n.d.	2.55E+05	n.d.	n.d.	n.d.

α -Muurolene	10208-80-7	1499	1499	27.89	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
α -Farnesene	502-61-4	1508	1507	28.13	5.10E+05	4.07E+05	4.97E+05	9.54E+05	9.80E+04	8.25E+05	4.62E+05
δ -Cadinene	483-76-1	1524	1522	28.59	1.06E+05	8.25E+04	8.87E+04	n.d.	4.79E+04	n.d.	n.d.
α -Calacorene	21391-99-1	1542	1542	29.16	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	3.09E+06	2.84E+06	2.84E+06	8.20E+06	2.16E+06	9.99E+06	5.39E+06
Ethyl laurate	106-33-2	1595	1594	30.68	6.32E+07	3.40E+07	5.07E+07	9.04E+07	2.63E+07	9.19E+07	6.93E+07
Bisabolol	515-69-5	1684	1683	33.17	n.d.	n.d.	4.01E+04	n.d.	n.d.	n.d.	n.d.
Farnesol	4602-84-0	1722	1727	34.24	1.81E+05	3.14E+04	9.73E+04	7.06E+05	n.d.	5.35E+05	3.58E+05
Ethyl palmitate	628-97-7	1993	1992	41.03	2.27E+06	6.23E+06	2.57E+06	2.33E+07	2.14E+06	1.55E+07	2.93E+06
Ethyl linoleate	544-35-4	2162		44.84	1.26E+05	7.21E+04	n.d.	9.21E+05	4.98E+04	2.62E+05	n.d.
COV	CAS	IR Standard	IR Samples	RT (min)	Área						
					UA-15	UM-01	UM-02	UM-03	UM-04	US-01	US-02
Ethyl Acetate	141-78-6	612	651	2.06	2.61E+06	1.98E+06	3.76E+06	1.63E+06	9.12E+05	6.28E+06	1.63E+06
3-Methyl-1-butanol	123-51-3	736	728	3.43	2.37E+07	1.70E+07	1.42E+07	2.93E+07	1.56E+07	1.52E+07	1.53E+07
2-Methyl-1-butanol	137-32-6	739	733	3.49	1.39E+07	6.51E+06	5.22E+06	1.27E+07	6.04E+06	5.14E+06	7.01E+06
Hexanal	66-25-1	800	799	4.73	4.48E+04	4.95E+04	4.15E+04	6.29E+04	3.98E+04	4.68E+04	3.44E+04
Ethyl butanoate	105-54-4	802	802	4.80	1.28E+05	1.01E+05	7.76E+04	2.45E+05	5.04E+04	1.09E+05	1.70E+05
Isoamyl acetate	123-92-2	876	875	6.83	1.02E+06	3.76E+05	8.09E+05	6.49E+05	7.57E+05	1.86E+05	3.67E+05
2-Heptanol	543-49-7	901	901	7.56	2.75E+05	n.d.	3.19E+05	4.83E+05	2.72E+05	2.55E+05	n.d.
Ethyl hexanoate	123-66-0	1000	1000	10.93	1.77E+06	1.83E+06	1.99E+06	4.42E+06	1.55E+06	4.96E+06	1.97E+06
p-Cymene	99-87-6	1022	1022	11.74	4.36E+05	1.71E+05	n.d.	2.83E+05	5.47E+04	n.d.	n.d.
Limonene	138-86-3	1030	1026	11.88	4.19E+05	n.d.	n.d.	1.36E+06	4.24E+05	n.d.	n.d.
1-Octanol	111-87-5	1071	1070	13.51	3.88E+05	2.29E+05	1.42E+05	7.67E+05	3.62E+05	1.01E+06	3.06E+05
2-Nonanone	821-55-6	1092	1091	14.26	9.63E+04	1.23E+05	1.32E+04	1.97E+05	8.44E+04	4.58E+05	1.70E+05
Ethyl heptanoate	106-30-9	1097	1098	14.50	4.95E+05	3.60E+05	n.d.	1.05E+06	2.31E+05	4.71E+05	1.75E+05
Linalool	78-70-6	1099	1099	14.52	n.d.	n.d.	1.67E+05	n.d.	n.d.	n.d.	n.d.
2-Nonanol	628-99-9	1101	1100	14.57	2.84E+05	n.d.	2.35E+05	n.d.	n.d.	n.d.	n.d.
Nonanal	124-19-6	1104	1103	14.67	1.00E+06	6.80E+05	4.66E+05	1.40E+06	5.40E+05	7.80E+05	5.91E+05

Phenylethyl Alcohol	60-12-8	1116	1114	15.09	6.43E+06	1.65E+06	7.77E+05	1.95E+05	8.21E+04	5.76E+05	1.74E+05
Menthol	1490-04-6	1169	1163	16.81	5.00E+05	3.70E+05	n.d.	n.d.	3.54E+04	n.d.	n.d.
Ethyl octanoate	106-32-1	1196	1197	18.04	4.90E+07	5.39E+07	4.86E+07	7.53E+07	4.45E+07	6.71E+07	4.53E+07
Decanal	112-31-2	1206	1204	18.29	8.53E+05	3.90E+05	4.89E+05	1.38E+06	1.53E+05	5.86E+05	1.31E+05
β -Cyclocitral	432-25-7	1220	1221	18.78	6.58E+05	n.d.	n.d.	1.74E+05	1.79E+05	n.d.	n.d.
Citronellol	106-22-9	1228	1228	19.11	8.29E+05	1.97E+05	1.55E+05	5.16E+05	1.69E+05	3.14E+05	2.30E+05
Ethyl phenylacetate	101-97-3	1246	1244	19.67	3.11E+05	1.26E+05	8.25E+04	2.20E+05	n.d.	4.13E+05	6.98E+04
Phenethyl acetate	103-45-7	1258	1256	20.08	7.15E+06	6.25E+05	5.56E+05	n.d.	4.35E+05	n.d.	3.71E+05
Ethyl nonanoate	123-29-5	1296	1295	21.43	2.43E+06	2.73E+06	7.90E+05	1.39E+07	2.11E+06	2.76E+06	2.13E+06
2-Undecanol	1653-30-1	1308	1300	21.58	6.64E+04	n.d.	n.d.	2.24E+05	1.63E+05	n.d.	8.50E+04
Undecanal	112-44-7	1307	1306	21.76	n.d.	9.16E+04	n.d.	3.16E+05	n.d.	1.19E+05	n.d.
β -Damascenone	23726-93-4	1386	1383	24.31	3.30E+06	1.41E+06	1.15E+06	1.63E+06	8.34E+05	1.31E+06	1.62E+06
β -Elemene	515-13-9	1391	1390	24.53	n.d.	n.d.	n.d.	8.06E+04	2.65E+05	n.d.	n.d.
Ethyl decanoate	110-38-3	1396	1397	24.73	1.63E+08	1.36E+08	1.23E+08	1.77E+08	1.22E+08	1.25E+08	1.45E+08
Isoamyl octanoate	2035-99-6	1446	1445	26.24	4.97E+06	3.05E+06	2.23E+06	3.55E+06	2.95E+06	1.49E+06	2.54E+06
Geranyl acetone	3796-70-1	1453	1452	26.45	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
(E)- β -Farnesene	18794-84-8	1457	1456	26.56	3.01E+06	2.94E+06	4.51E+05	1.03E+06	6.78E+06	1.57E+06	6.87E+05
α -Curcumene	644-30-4	1483	1481	27.36	2.85E+05	1.31E+05	3.23E+04	1.15E+05	7.79E+05	1.31E+05	4.62E+04
(E)- β -Ionone	79-77-6	1486	1486	27.47	n.d.	4.74E+04	n.d.	n.d.	n.d.	n.d.	n.d.
α -Muurolene	10208-80-7	1499	1499	27.89	n.d.	2.20E+05	n.d.	2.01E+05	n.d.	1.55E+05	n.d.
α -Farnesene	502-61-4	1508	1507	28.13	1.81E+06	1.79E+06	2.22E+05	5.83E+05	4.27E+06	1.19E+06	3.23E+05
δ -Cadinene	483-76-1	1524	1522	28.59	1.81E+05	2.18E+05	1.22E+05	1.99E+06	6.91E+05	6.57E+05	5.80E+04
α -Calacorene	21391-99-1	1542	1542	29.16	n.d.	n.d.	n.d.	1.96E+05	n.d.	3.91E+05	n.d.
Nerolidol	7212-44-4	1564	1563	29.77	1.64E+07	1.84E+07	3.39E+06	6.87E+06	2.67E+07	1.07E+07	1.86E+06
Ethyl laurate	106-33-2	1595	1594	30.68	9.28E+07	7.48E+07	7.67E+07	8.28E+07	7.09E+07	7.30E+07	5.93E+07
Bisabolol	515-69-5	1684	1683	33.17	n.d.	5.06E+05	7.70E+04	n.d.	n.d.	2.34E+05	n.d.
Farnesol	4602-84-0	1722	1727	34.24	2.48E+06	8.81E+05	2.08E+05	6.00E+05	3.74E+05	3.78E+05	1.62E+05
Ethyl palmitate	628-97-7	1993	1992	41.03	1.25E+07	7.44E+06	6.72E+06	6.45E+06	1.22E+07	6.56E+05	8.44E+05
Ethyl linoleate	544-35-4	2162		44.84	7.01E+05	2.58E+05	3.36E+05	7.74E+04	1.81E+05	n.d.	n.d.

ANEXOS

ANEXO A – Comprovante de aceite para publicação do artigo I

Fwd: Journal of the Institute of Brewing - message regarding JIB.20251630 [email ref: HE-1-a]

De: Prof.º Normando Mendes Ribeiro Filho (normandomrfilho@gmail.com)

Para: vanessapsv@yahoo.com.br

Data: domingo, 22 de junho de 2025 às 21:35 BRT

11-Jun-2025

JIB.20251630 - Chemical Composition and Sensory Diversity of Colourless Cachaças from Paraíba: Influence of Geographic Origin and Production Practices

Dear Dr. Ribeiro Filho,

I'm pleased to tell you that your MS has been accepted for publication in the Journal of the Institute of Brewing.

This is 'soft' accept as I will copy edit your MS in readiness for publication. This will be shared with you on formally accepting your MS via the 'system'. You can review/edit the resulting draft pdf prior to publication.

It will take a bit of time as there are four papers ahead of yours to copy edit/publish.

Cheers,
David

Dr David Quain
EiC, Journal of the Institute of Brewing
Honorary Professor of Fermentation and Dispense Technology at the University of Nottingham
Profº. Normando Mendes Ribeiro Filho
Fermentação, leveduras e química de aromas
Laboratório de Tecnologia de Produtos Agropecuários
Departamento de Solos e Engenharia Rural
Centro de Ciências Agrárias - Campus II - Areia - PB
Universidade Federal da Paraíba

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Fone: (83) 3362-2300 - Ramal: 3289

ANEXO B – Comprovante de submissão do artigo II

Wood Science and Technology - Receipt of Manuscript 'Influence of Amburana...'

De: Wood Science and Technology (sruthi.lakshmi@springernature.com)

Para: vanessapsv@yahoo.com.br

Data: terça-feira, 20 de maio de 2025 às 10:56 BRT

Ref: Submission ID 70b145f8-4d8d-4450-8fc3-f2b2c9032937

Dear Dr Silva,

Please note that you are listed as a co-author on the manuscript "Influence of Amburana cearensis Barrels on the Sensory Profile and Complexity of Cachaça During Maturation", which was submitted to Wood Science and Technology on 20 May 2025 UTC.

If you have any queries related to this manuscript please contact the corresponding author, who is solely responsible for communicating with the journal.

Kind regards,

Editorial Assistant
Wood Science and Technology