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**PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIA E TECNOLOGIA DE
ALIMENTOS**

EIKE GUILHERME TORRES DE SOUZA

**EFEITO DO RECICLO DE “CHIPS” DAS MADEIRAS CARVALHO (*Quercus sp.*),
CASTANHEIRA (*Bertholletia excelsa*) E AMBURANA (*Amburana cearensis*) PARA
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CASTANHEIRA (*Bertholletia excelsa*) E AMBURANA (*Amburana cearensis*) PARA MATURAÇÃO
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Aos meus pais, Maria Valdete e Maurílio,
Dedico.

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“É no meio da dificuldade que reside a oportunidade.”

- Albert Einstein

RESUMO

A aguardente é um dos destilados mais consumidos mundialmente e, para atender mercados cada vez mais exigentes, a maturação torna-se uma etapa fundamental para a melhoria das características químicas e sensoriais. Tradicionalmente realizada em barris de madeira, esse processo apresenta alto custo e longo tempo, o que motivou o desenvolvimento de métodos rápidos e alternativos, como o uso de chips de madeira. Este trabalho investigou a utilização e a reutilização de chips das madeiras carvalho (*Quercus sp.*), castanheira (*Bertholletia excelsa*) e amburana (*Amburana cearensis*) para maturação acelerada de aguardente, avaliando os efeitos do processo no perfil químico e na bioatividade da bebida. O delineamento experimental foi dividido em duas etapas principais. Na primeira, realizou-se a coleta e caracterização química da matriz alcoólica, obtida de engenho local na Paraíba, monodestilada e sem diluição. Após a confirmação dos parâmetros de identidade e qualidade, o destilado foi utilizado na segunda etapa, que consistiu na maturação acelerada em dois ciclos com chips de carvalho, amburana e castanheira. No primeiro ciclo, a bebida foi armazenada por 28 dias com seis chips por litro, gerando os respectivos tratamentos Q-1, A-1 e B-1. Após essa fase, os chips foram reutilizados para um segundo ciclo, originando os tratamentos Q-2, A-2 e B-2. O acompanhamento periódico foi realizado em intervalos de 7 dias (teor alcoólico, densidade relativa, intensidade de cor, acidez volátil, fenólicos totais e taninos totais), e as bebidas finais foram avaliadas quanto à cor instrumental, atividade antioxidante (DPPH, ABTS e FRAP), perfil de fenólicos, congêneres voláteis e perfil de voláteis, proporcionando sua caracterização química e bioativa. Os resultados indicaram que a primeira utilização dos chips promoveu maior extração de compostos fenólicos, diretamente associados ao aumento da intensidade de cor (amarelo saturado) e da atividade antioxidante. Essas amostras também apresentaram maior diversidade e intensidade de compostos voláteis, especialmente ésteres e terpenos, resultando em perfis característicos em função do tipo de madeira utilizado. Entre as madeiras, o carvalho destacou-se pelo maior conteúdo fenólico e atividade antioxidante. A castanheira apresentou um perfil de fenólicos semelhante ao carvalho, e a amburana apresentou características diferenciadas quanto aos tipos de fenólicos presentes. Por outro lado, a reutilização dos chips revelou-se limitada, com redução na intensidade das características químicas, aproximando as amostras da matriz alcoólica, devido à extração dos compostos da madeira ocorrer de forma mais eficiente no primeiro uso. Os achados sugerem que o uso de chips de madeira é uma alternativa promissora para a produção de aguardentes de alta qualidade, especialmente em mercados que demandam processos mais econômicos e rápidos. Contudo, a prática de reutilização dos chips requer otimizações adicionais para garantir a manutenção da qualidade química ao longo dos ciclos de maturação.

PALAVRAS-CHAVE: Aguardente de cana-de-açúcar. Maturação acelerada. Chips de madeira. Reuso. Perfil fenólico e volátil. Atividade antioxidante.

ABSTRACT

Spirits are one of the most widely consumed distillates in the world and, in order to satisfy increasingly demanding markets, maturation has become a fundamental stage in improving chemical and sensory characteristics. Traditionally carried out in wooden barrels, this process is costly and time-consuming, which has prompted the development of quick and alternative methods, such as the use of wood chips. This study investigated the use and reuse of oak (*Quercus sp.*), chestnut (*Bertholletia excelsa*) and amburana (*Amburana cearensis*) wood chips for the accelerated maturation of brandy, assessing the effects of the process on the chemical profile and bioactivity of the beverage. The experimental design was divided into two main stages. In the first, the alcoholic matrix was collected and chemically characterized, obtained from a local mill in Paraíba, mono-distilled and undiluted. After confirming the identity and quality parameters, the distillate was used in the second stage, which consisted of accelerated maturation in two cycles with oak, amburana, and chestnut chips. In the first cycle, the beverage was stored for 28 days with six chips per liter, generating the treatments Q-1, A-1, and B-1. After this phase, the chips were reused for a second cycle, giving rise to treatments Q-2, A-2, and B-2. Periodic monitoring was carried out at seven-day intervals (alcohol content, relative density, color intensity, volatile acidity, total phenolics, and total tannins). The final drinks were evaluated for instrumental color, antioxidant activity (DPPH, ABTS, and FRAP), phenolic profile, volatile congeners, and volatile profile, providing their chemical and bioactive characterization. The results indicated that the first use of the chips led to greater extraction of phenolic compounds, directly associated with an increase in color intensity (saturated yellow) and antioxidant activity. These samples also showed greater diversity and intensity of volatile compounds, especially esters, and terpenes, resulting in characteristic profiles depending on the type of wood used. Among the woods, oak stood out for its higher phenolic content and antioxidant activity. Chestnut had a similar phenolic profile to oak, while amburana had different characteristics in terms of the types of phenolics present. On the other hand, the reuse of the chips proved to be limited, with a reduction in the intensity of the chemical characteristics, bringing the samples closer to the alcoholic matrix, due to the extraction of the compounds from the wood occurring more efficiently in the first use. The findings suggest that the use of wood chips is a promising alternative for the production of high-quality sugarcane spirits, especially in markets that demand more economical and faster processes. However, the practice of reusing the chips requires additional optimizations to ensure that the chemical quality is maintained throughout the maturation cycles.

KEY-WORDS: Sugarcane spirit. Accelerated maturation. Wood chips. Reuse. Phenolic and volatile profile. Antioxidant activity.

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1. INTRODUÇÃO

A aguardente de cana-de-açúcar tem, em seu processo produtivo, a maturação como uma das etapas mais importantes, impactando diretamente os produtos formados, especialmente em mercados competitivos que exigem alta qualidade (Da-Silva *et al.*, 2023). Atrás da vodca russa e do soju coreano, é o terceiro destilado mais consumido no mundo, e sua exportação representa uma das principais fontes de receita para os produtores (Granato *et al.*, 2014; Serafim *et al.*, 2015).

Tradicionalmente, os fabricantes de aguardente utilizam-se de barris e recipientes apropriados de madeiras para o envelhecimento, onde a bebida permanece acondicionada, por tempo variável, com objetivo de promover e intensificar interações físico-químicas entre a matriz alcoólica e a madeira, acarretando assim em alterações químicas na bebida e consequentemente no perfil sensorial final (Bortoletto *et al.*, 2016). Apesar dos benefícios, esta técnica apresenta fatores limitantes de utilização, como elevado tempo operação, imobilização do capital de giro sob a forma de acondicionamento, além de dificuldades relacionadas ao acompanhamento de parâmetros de processo, custo de aquisição dos barris e conhecimento limitado sobre o efeito da reutilização das madeiras para produção de novas bebidas (Coelho *et al.*, 2021).

Levando em consideração as mudanças propostas na legislação brasileira “(Brasil, 2022b), a adição de constituintes fracionados de madeiras, como os “chips”, tem sido avaliado como método alternativo para otimização do processo de maturação da aguardente de cana, oferecendo à bebida compostos fenólicos e voláteis característicos de envelhecimento, em menor tempo e com menores custos, devido a utilização de pequenas quantidades de madeira por litro de bebida e os “chips” podem ser reutilizados (García-Alcaraz *et al.*, 2020).

Em soma, destaca-se na literatura e no setor produtivo, que o carvalho, especificamente a espécie do gênero *Quercus*, é a principal madeira utilizada na confecção de barris destinados ao armazenamento de destilados, devido aos atributos sensoriais reconhecidos (Fernandes *et al.*, 2019). Entretanto, devido ao alto custo de aquisição desta madeira, produtores buscam alternativas como substituição na maturação de aguardente, como outras madeiras, à exemplo de algumas madeiras tropicais brasileiras, com características sensoriais semelhantes, ou métodos que utilizem uma menor quantidade de madeira, como a aplicação de “chips” supracitada (Coelho *et al.*, 2021).

Dentre as madeiras tropicais brasileiras que apresentam contribuição desejável ao perfil químico e sensorial das bebidas, destaca-se a castanheira (*Bertholletia excelsa*) por apresentar

características similares ao carvalho quanto aos principais compostos de envelhecimento, como voláteis e fenólicos (Coldea *et al.*, 2020) e a amburana (*Amburana cearensis*), devido a sua alta demanda comercial, além de formar espécies químicas em proporções mais elevadas durante a maturação em comparação ao carvalho, como vanilina, ácido vanílico, siringaldeído e sinapaldeído, conferindo perfil aromático específico (Bortoletto, *et al.*, 2021).

A escolha do tipo e da espécie de madeira utilizada possibilita a obtenção de destilados com características químicas, e consequentemente sensoriais, exclusivas e distintas. Isso torna a caracterização dos compostos derivados da interação entre madeira e o destilado, altamente relevante para a compreensão dos impactos dessa etapa no produto final (Bortoletto *et al.*, 2016).

Considerando que a indústria de bebidas destiladas busca constantemente oferecer produtos inovadores, capazes de atender às demandas e preferências dos mercados, torna-se essencial aprofundar o entendimento sobre tecnologias emergentes de maturação e os fatores que impactam as características químicas e bioativas associadas à produção da bebida.

2. OBJETIVOS

2.1 OBJETIVO GERAL

Investigar a utilização e a reutilização de chips das madeiras carvalho (*Quercus sp.*), castanheira (*Bertholletia excelsa*) e amburana (*Amburana cearensis*) para maturação acelerada de aguardente, avaliando os efeitos do processo no perfil químico e bioatividade da bebida.

2.2 OBJETIVOS ESPECÍFICOS

- Realizar o acompanhamento periódico para monitorar a evolução dos compostos marcadores químicos de maturação na aguardente com chips de madeiras;
- Determinar as características físicas de cor das aguardentes maturadas com chips de madeiras;
- Caracterizar o perfil de compostos fenólicos, fenólicos totais e taninos das aguardentes maturadas com chips de madeiras;
- Avaliar o potencial antioxidante das aguardentes maturadas com chips de madeiras;
- Determinar o perfil de compostos voláteis das aguardentes acondicionadas com chips de madeiras.

3. REVISÃO DE LITERATURA

3.1 AGUARDENTE DE CANA-DE-AÇÚCAR

Segundo as disposições legais do Ministério da Agricultura Pecuária e Abastecimento (MAPA), entende-se por aguardente de cana a bebida de graduação alcoólica de 38 a 54% (V/V) a 20 °C, obtida do destilado alcoólico simples de cana-de-açúcar ou pela destilação do mosto fermentado de cana-de-açúcar, podendo ser adicionados até 6 g/L de açúcares (Brasil, 2022b).

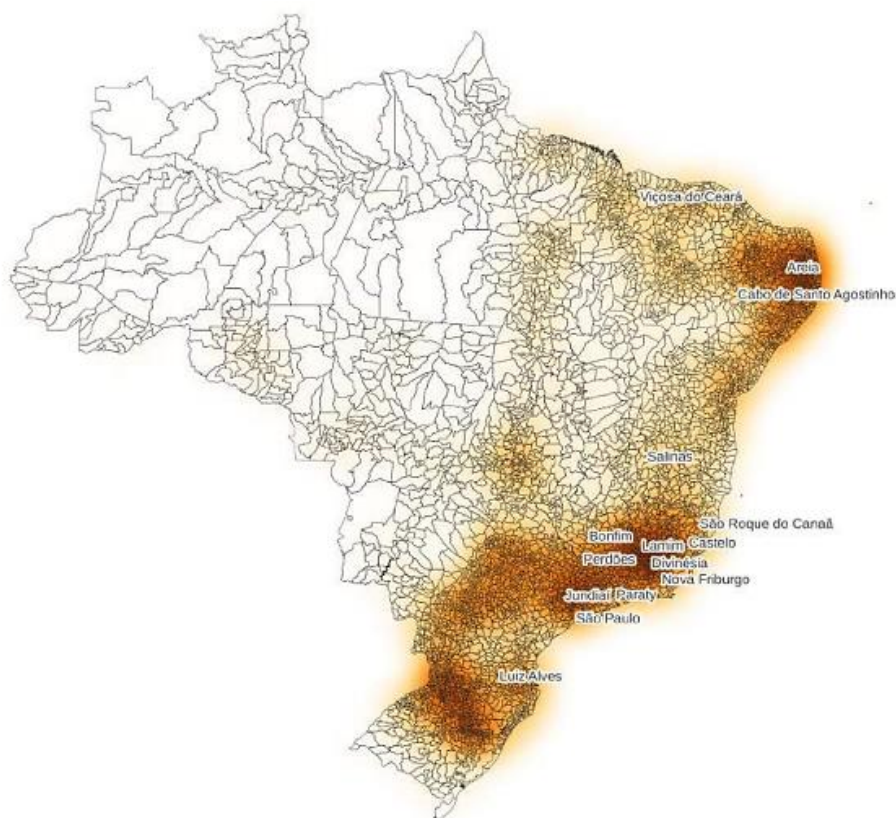
Cachaça é a denominação típica e exclusiva da aguardente de cana produzida no Brasil, com graduação alcoólica de 38 a 48% (V/V) a 20 °C, obtida pela destilação do mosto fermentado do caldo de cana-de-açúcar em alambiques de cobre com características sensoriais peculiares, podendo ser adicionada de açúcares até seis gramas por litro (Brasil, 2022b). Portanto, toda cachaça é aguardente, mas nem toda aguardente pode ser considerada cachaça. Suas principais diferenças estão relacionadas ao teor alcoólico das bebidas, as matérias-primas utilizadas para fermentação, assim como seu local e aparelho de produção.

3.1.1 Setor produtivo

Mesmo com seu consumo destinado majoritariamente ao mercado brasileiro, atualmente, a cachaça está entre as bebidas destiladas mais consumidas no mundo, com uma produção anual estimada em aproximadamente 1,3 bilhão de litros, sendo produzida em todos os estados brasileiros, exceto nos estados do Amapá e Roraima (IBRAC, 2023; Ribeiro-Filho, 2020). Dez estados, incluindo Minas Gerais, São Paulo, Espírito Santo, Rio de Janeiro, Santa Catarina, Rio Grande do Sul, Paraíba, Goiás, Paraná e Bahia, têm o maior número de produtores de cachaça (IBRAC, 2023).

Com relação à concentração de estabelecimentos por município, destaca-se principalmente a alta concentração observada em Viçosa do Ceará/ CE, que abriga 41,7% das cachaçarias do estado, além de Areia/PB, que concentra 28,2% das cachaçarias (Brasil, 2022a). O mapa de calor apresentado na Figura 1 evidencia a concentração das cachaçarias na região sudoeste, sobretudo no estado de Minas Gerais. Observa-se também que embora Salinas/MG seja o município com maior número de estabelecimentos registrados, colorações mais quentes estão presentes em outras regiões como a Paraíba, sobretudo a região do Brejo Paraibano, Pernambuco, Oeste Catarinense e Noroeste Rio-Grandense.

Figura 1. Mapa de calor de cachaçarias com registro de produção ativo no Brasil.



Fonte: Anuário da cachaça 2021, MAPA (Brasil, 2022a).

Após queda nas exportações durante a pandemia, o setor registrou no ano de 2022 um recorde no valor exportado. Foram US\$ 18,47 milhões exportados, aproximadamente US\$ 4 milhões a mais do que em 2019, sendo o maior valor dos últimos 12 anos e 54,74% maior que as exportações de 2021 (IBRAC, 2023). Houve ainda crescimento no volume exportado, com 8,6 milhões de litros seguindo o exterior, promovendo aumento de 30,38% em comparação ao ano de 2021.

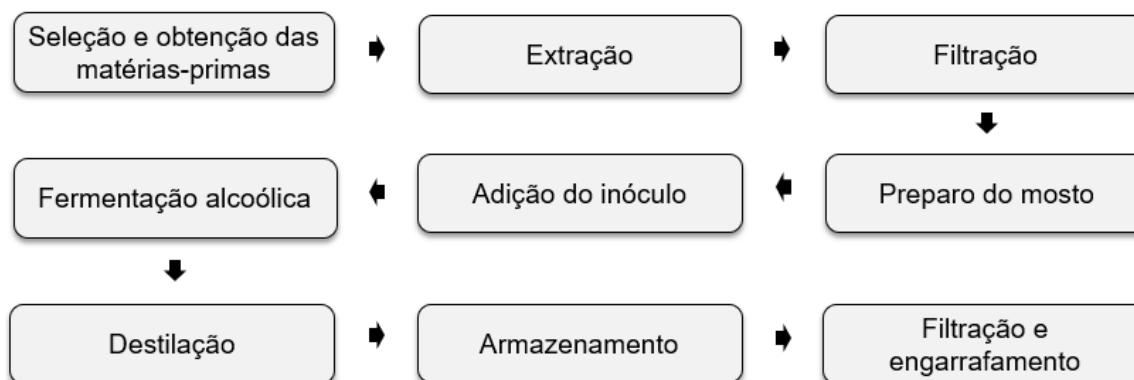
Segundo os dados do anuário da cachaça de 2021 (Brasil, 2022a), a aguardente de cana produzida no Brasil é exportada para 67, somando mais de 7 milhões de litros vendidos e US\$ 13 milhões. Em termos de valor exportado, os principais são os Estados Unidos, Alemanha, Paraguai, Portugal, França e Itália. Este ano trouxe, inclusive, um aumento significativo na participação de alguns desses países, que até então não estavam entre os principais mercados. Assim, para incremento do mercado de exportação e participação ativa de pequenos e médios produtores, faz-se necessário a valorização e elevação da qualidade sensorial e química da bebida, em busca de público internacional mais exigente.

3.1.2 Processo de produção da cachaça (aguardente de cana-de-açúcar)

A cachaça é uma bebida alcoólica produzida exclusivamente no Brasil com teor alcoólico de 38 a 48°GL (% v/v) a 20°C, cujo teor alcoólico é gerado a partir da fermentação do caldo de cana (mosto de cana), seguida da destilação do mosto de cana com características sensoriais adequadas. A cachaça pode ser classificada em: cachaça, cachaça adoçada, cachaça descansada e cachaça envelhecida (incluindo cachaça premium e cachaça extra premium) (Brasil, 2022b). A regulamentação da cachaça é simples, assim como as etapas de produção, que são a prática de campo (plantio de cana-de-açúcar, colheita e transporte) e a prática industrial (extração do suco de recepção, fermentação, destilação, padronização e envelhecimento) (Da-Silva *et al.*, 2023).

A produção de cachaça deve ser monitorada desde a obtenção das matérias primas até o acondicionamento do destilado final. As etapas para obtenção de aguardente podem ser resumidas e visualizadas na Figura 2, de modo que cada parte do processo deve ser seguida no intuito da obtenção de um produto final com qualidade.

Figura 2. Etapas resumidas para o processamento de aguardente.



Fonte: Adaptado de Venturini Filho (2010).

Como uma das etapas impactantes para o destilado final, a fermentação alcoólica é realizada pelas atividades metabólicas das leveduras, que convertem os açúcares em etanol, dióxido de carbono e metabólitos secundários. As leveduras são amplamente utilizadas na produção de cachaça devido à sua característica de resistência à toxicidade, à sua alta capacidade fermentativa, à sua resistência ao estresse e à sua geração de compostos desejáveis (Da-Silva *et al.*, 2023).

Durante a fermentação, as células de levedura produzem uma grande quantidade de substâncias ativas de aroma (moléculas que contêm enxofre, ácidos orgânicos, álcoois superiores, compostos carbonílicos e ésteres voláteis) que afetam a complexidade do sabor das bebidas alcoólicas fermentadas, mesmo que estejam em baixas concentrações (Da-Silva *et al.*, 2023).

Dentre os principais compostos, os ésteres voláteis possuem grande contribuição sensorial, pois são responsáveis pelo caráter frutado desejável em bebidas, como os ésteres de acetato, destacando-se o acetato de etila (aroma solvente), o acetato de isoamila (sabor de banana) e o acetato de feniletila (aroma floral e de rosas). Adicionalmente, ésteres etílicos de ácidos graxos de cadeia média, como o hexanoato de etila e octanoato de etila, que tem aromas de “maçã azeda” também possuem importância no buquê aromático (Ribeiro-Filho *et al.*, 2021).

Após a fermentação, o fermentado cuja concentração alcoólica é de 7 a 8% é enviado ao alambique e aquecido, emitindo vapores com composição rica em compostos voláteis alcoólicos, formando o teor alcoólico das bebidas (Ribeiro-Filho *et al.*, 2021). Pode-se entender por destilação, a operação unitária de separação entre misturas com diferentes pontos de ebulição, assim, utiliza a volatilização de líquidos pelo aquecimento, condensando-os a seguir, com objetivo de formar novos produtos por decomposição das frações “cabeça”, “coração” e “cauda”.

A destilação permite a separação de componentes voláteis (água, álcool etílico, aldeídos, álcoois superiores, ácido acético, etc.) dos componentes não voláteis (células de leveduras, bactérias, sólidos em suspensão, sais minerais, açúcares não fermentescíveis, proteínas, entre outros resíduos), obtendo como produtos finais duas frações líquidas: o destilado rico em etanol, água e outros componentes (flegma) e a vinhaça (ou vinhoto) (Venturini Filho, 2010).

As aguardentes recém-destiladas apresentam-se incolores, com gosto ardente, agressivo, sabor repugnante, além de buquê irregular, não sendo recomendado o seu consumo imediato. Contudo, torna-se necessário a alteração parcial da composição química do produto após sua destilação (Venturini Filho, 2010). Assim, o processo de armazenamento da cachaça se caracteriza em sua maioria na busca por melhorar a qualidade química, e por consequência a sensorial, da bebida.

O armazenamento dos destilados pode ser feito em recipientes inertes, como tanques de aço inoxidável, porém o processo mais tradicional e mais usual é a utilização de recipientes de madeira (tonéis ou pipas) para que ocorra o descanso da bebida. Por consequência das reações de oxidação e esterificação entre o destilado e os componentes da madeira, ocorre a maturação

da bebida e originam-se novas substâncias químicas, como compostos aromáticos, conferindo-lhe boas características sensoriais. Por exemplo, ácidos reagem com álcoois formando ésteres, que são as substâncias mais aromáticas que as anteriores (Alcarde *et al.*, 2010).

3.2 MATURAÇÃO DA AGUARDENTE DE CANA-DE-AÇÚCAR

O processo de maturação é um sistema complexo que envolve numerosas reações fundamentadas principalmente pela extração de moléculas da madeira e aeração controlada do líquido alcoólico. Fenômenos de migração de constituintes da madeira, evolução de compostos fenólicos, aeração/oxidação, estabilização da cor, sabor e o surgimento do caráter amadeirado, contribuem para a riqueza e complexidade do buquê aromático (Ramirez-Ramirez, 2002). O envelhecimento de destilados é o principal fator para a sua caracterização, pois aproximadamente 60% dos compostos aromáticos são oriundos da interação com a madeira, sendo o restante proveniente do processo de produção (Mosedale; Puech, 1998).

Segundo Bortoletto *et al.* (2021) Os efeitos e tempo requeridos para a maturação são variáveis e influenciados principalmente pelo tipo de madeira utilizada. Diversas madeiras possuem potencial para envelhecer a aguardente, tais como acácia, castanheira, carvalho, araruva, jequitibá, grápia, jatobá, freijó, eucalipto, cedro, entre outras (Parazzi *et al.*, 2008).

A caracterização da madeira e do barril está condicionada a fatores ambientais, geográficos, país e floresta de origem, clima e solo, variações inerentes à composição das macromoléculas das árvores e suas individualidades (idade, largura do cerne e composição anatômica), método de obtenção de aduelas, tipo de maturação da madeira ou secagem (natural ou artificial), tempo em que as aduelas foram expostas ao ambiente antes da construção do barril - "seasoning", tempo e temperatura de queima aplicada na produção do barril, tamanho, condições de temperatura e umidade do local de armazenamento dos barris durante o envelhecimento da bebida (Mosedale; Puech, 1998).

3.2.1 Estrutura da madeira

O constituinte vegetal denominado de madeira, possui estrutura física heterogênea, sendo um sistema biológico complexo, constituídas de diversas moléculas, com maior destaque a celulose, hemicelulose e lignina, também denominadas macromoléculas e em menor parte pectinas, proteínas, triglicerídeos e compostos inorgânico, como os minerais (Barrera-García *et al.*, 2007).

A celulose é um homopolissacarídeo linear constituído por monômeros de glicose ligados por ligações glicosídicas β (1-4) e é o principal constituinte da madeira (45% p/p). O segundo polímero de maior proporção presente na madeira é a lignina (25% p/p), sendo constituído por três álcoois fenilpropenóicos (álcool p-cumarílico, álcool coniferílico e álcool sinapílico) e possuindo funcionalidade estrutural de tornar a parede celular vegetal rígida e impermeável. Em terceiro lugar, a hemicelulose (20 % p/p) é formada por polissacarídeos complexos (xilanos, xiloglucanos, fucogalactoxiloglucanos e mananos). Se diferencia da celulose por possuir baixo peso molecular, não ser solúvel em soluções alcalinas e sofrer hidrólise por ácidos. As hemiceluloses podem produzir pentoses por hidrólise e posteriormente furfural, molécula aromática encontrada em bebidas maturadas (Masson; Puech; Moutnet, 1995).

No caso da cachaça e aguardente de cana, o furfural também pode ser formado pela pirogênese da matéria orgânica durante o processo de destilação em alambiques, contribuindo para o sabor ardente da bebida (Asquieri; Silva; Cândido, 2009). A partir do aquecimento para a confecção dos barris comumente utilizados, a hemicelulose gera produtos de caramelização, provenientes da quebra das moléculas de açúcar por desidratação e reação de Maillard, desenvolvendo compostos relevantes para a composição do buquê aromático e da cor característica do destilado (Bortoletto *et al.* 2015). As pentoses formam furfural como principal produto de degradação, já as hexoses formam 5-hidroximetilfurfural (HMF) e outros compostos como 2- hidroxiacetilfurano e maltol (Boidron; Chatonnet; Pons, 1988). A fragmentação da cadeia de carbono destes produtos primários da desidratação forma outros compostos, tais como acetol, acetoina, diacetil e os ácidos láctico, pirúvico, acético, levulínico e fórmico. O surgimento de produtos de caramelização, derivados da quebra de polioses (celulose e hemicelulose), é maximizado pelo maior tempo de maturação da madeira e pela aplicação da queima com intensidade média (Bortoletto *et al.* 2016).

A macromolécula responsável pela geração dos compostos denominados marcadores de envelhecimento, é a lignina. Esses compostos são formados a partir da desestruturação desta molécula pela ação do etanol e oxidação promovida pela ação da aeração do barril. Os marcadores de envelhecimento são responsáveis pela caracterização do buquê aromático e evolução da bebida (Ramirez-Ramirez, 2002).

3.2.2 Madeiras utilizadas

3.2.2.1 Carvalho (*Quercus sp.*)

O carvalho é a madeira mais indicada para a fabricação de barris, devido à estrutura das fibras que geram resistência mecânica, flexibilidade, porosidade e dureza, além de conferir características sensoriais peculiares que agregam qualidade à bebida. O carvalho é a principal madeira utilizada no envelhecimento de bebidas alcoólicas, adquirindo supremacia mundial. Modifica as propriedades sensoriais da bebida, pois participa ativamente do "flavor" e viscosidade ou oleosidade, graças à extração de moléculas aromáticas e taninos hidrolisáveis. Porém, demanda longo período de crescimento da árvore, alto custo inicial e a necessidade de importação de países europeus ou norte-americanos (Les Cahiers Itinéraires D'itv France, 2003).

O carvalho é representado por mais de 250 espécies no mundo, situadas majoritariamente nas zonas temperadas no hemisfério norte do planeta. Para a confecção de barris, são utilizadas predominantemente as espécies: carvalho peduncular (*Quercus robur* Linn, *Quercus pedunculata* Ehrh.), carvalho séssil (*Quercus petraea* Liebl, *Quercus sessiliflora* Sm.), carvalho branco americano (*Quercus alba* L.) e o vermelho da América do Norte (*Quercus rubra*) (Chatonnet; Dubourdieu, 1998).

Os fatores que influenciam a composição química da madeira de carvalho estão relacionados com a espécie botânica, origem geográfica, idade da madeira e modo de conduzir a floresta. Esses são considerados parâmetros relevantes na escolha do produtor, pois definem a qualidade da madeira e, conseqüentemente no perfil sensorial da bebida (Marco *et al.*, 1994; Mosedale; Puech, 1998).

3.2.2.2 Madeiras brasileiras

Com utilização comercial já conhecida e aplicada a alguns anos, outras espécies de madeira tropical brasileira têm sido usadas para o envelhecimento de cachaça, incluindo umburana, amendoim bravo, jequitibá, araruva, jequitibá rosa, cerejeira, ipê roxo, castanheira, grápia, pau-pereira e freijó. Os produtores de cachaça ainda utilizam barris de carvalho para envelhecimento; no entanto, o mercado brasileiro e o mercado internacional vêm abrindo novas portas para as cachaças envelhecidas em barris de madeira (Bortoletto; Alcarde, 2013; Bortoletto *et al.*, 2021; Bortoletto *et al.*, 2016; Ribeiro-Filho 2020).

O envelhecimento da cachaça em madeiras tropicais mantém os padrões de autenticidade da cachaça; porém, cada tipo de madeira gera uma composição físico-química e características sensoriais diferentes para a cachaça (Alcarde *et al.*, 2010; Bortoletto *et al.*, 2021). O envelhecimento da cachaça em barris de madeira tropical abriu um novo mercado para os produtores devido à diversidade de barris de madeira tropical e suas possibilidades de blendagem, que podem gerar uma mistura diferente de sabores e características sensoriais para atender a diversos tipos de consumidores (Bortoletto *et al.*, 2021). Cada tipo de barril de madeira influencia a qualidade da cachaça, reduzindo o amargor e aumentando a doçura, resultando em uma bebida suave.

Os barris de Umburana fornecem uma predominância de compostos como curamina, eugenol, louro canela, catequina e ácido vinílico (Dias *et al.*, 1998). Campos *et al.* (2004), analisaram por cromatografia líquida de alta eficiência - HPLC, compostos fenólicos de baixo peso molecular (ácido gálico, 5-hidroximetilfurfural, furfural, ácido vanílico, ácido siríngico, vanilina, siringaldeído, coniferaldeído, sinapaldeído e cumarina) em cachaças envelhecidas, originadas das regiões produtoras do Estado do Ceará e em extratos de Amburana cearenses com o objetivo de estabelecer o perfil químico das cachaças e avaliar o potencial dos extratos de Amburana. O estudo concluiu que o tempo de armazenamento ideal para fornecer maiores concentrações dos compostos fenólicos deve variar de acordo com as características do barril (tipo de madeira, idade e tamanho) e com as condições ambientais do local de armazenamento (temperatura e umidade). Os barris de Castanheira geram como compostos fenólicos principais o ácido gálico e o ácido elágico (Bortoletto *et al.*, 2016; Zacaroni *et al.*, 2011).

3.2.3 Alterações físico-químicas durante a maturação

A maturação é um processo de modificação da composição química do destilado. Seu mecanismo se baseia na extração de moléculas da madeira, sua posterior oxidação e volatilização dos componentes do destilado alcoólico. O álcool favorece reações físico-químicas de extração e degradação de moléculas da madeira.

A transformação do destilado ocorre por vias aditivas e subtrativas. As vias aditivas correspondem à decomposição das macromoléculas da madeira, extração de constituintes minoritários e oxidação de compostos (Bortoletto *et al.*, 2021). As vias subtrativas caracterizam-se pela evaporação de compostos voláteis, adsorção de moléculas pelas fibras da madeira e oxidação de compostos (Mosedale; Puech, 1998).

O sistema físico-químico presente no processo de maturação se caracteriza pela saturação das fibras da madeira e dissolução das moléculas extraídas pela madeira na bebida. A ação de extração de constituintes da madeira, assim como a oxidação do destilado, ocorre no limite de saturação da fibra (Bortoletto *et al.*, 2016). Nos primeiros dias em contato com o destilado e com o passar do tempo, o processo dinâmico confere o retorno de algumas moléculas para o interior do barril. A extração de constituintes da madeira é ocasionada pela sua simples dissolução nas bebidas alcoólicas e por meio de reações de hidrólise, pirólise e oxidações que permitem reduzir o grau de retenção dos compostos e facilitar sua dissolução no líquido (Conner; Paterson; Piggott, 1989; Mosedale; Puech, 1998; Nykanen, 1986; Singleton, 1995).

As macromoléculas estruturais da madeira (celulose, hemicelulose e lignina) são os principais responsáveis da transformação do destilado e responsáveis pela geração dos marcadores de envelhecimento. A lignina é a macromolécula com maior potencial para gerar marcadores. A extração direta de seus principais blocos monoméricos possibilita a geração de moléculas menores com o passar do tempo (Bortoletto *et al.*, 2015).

Duas vias ocorrem simultaneamente, a via do siringil (cuja oxidação origina o sinapaldeído, o siringaldeído e o ácido siríngico) e a via do guaiaxil (gerando o coniferaldeído, a vanilina e o ácido vanílico). Os mecanismos que envolvem a extração desses compostos são propostos de duas principais formas, uma delas é a simples extração desses compostos fenólicos presentes na madeira, que são incorporados na bebida, e a outra é a partir da extração da lignina da madeira mediante ação do etanol, formando um composto etanol-lignina que é posteriormente degradado em compostos fenólicos simples. Pelas vias de formação, a oxidação do sinapaldeído origina o siringaldeído, o qual, por sua vez, pode ser oxidado a ácido siríngico. A oxidação do coniferaldeído forma a vanilina, a qual pode ser oxidada a ácido vanílico (Aylott; Mackenzie, 2010; Dias; Maia; Nelson, 1998; Puech, 1981; Puech; Jouret; Goffinet, 1985).

Demais marcadores de envelhecimento podem ser oriundos da celulose e hemicelulose, tal como o furfural e o 5-hidroximetilfurfural. Taninos hidrolisáveis, presentes na madeira, são responsáveis pela origem do ácido gálico e ácido elágico. Lactonas são ésteres cíclicos presentes nas madeiras, que impactam positivamente o destilado envelhecido. Algumas madeiras contêm ácidos graxos de cadeia longa, que derivam isômeros *cis* e *trans* da β -metil- γ -octalactona. A oxidação térmica dos ácidos graxos gera a whiskylactona (Nykanen, 1986). Apesar do destilado envelhecido ser caracterizado pela adição de componentes da madeira, há também a redução de alguns compostos, promovida pelo sistema em ação. As principais vias subtrativas ocorrem por meio de oxidação, evaporação e adsorção (Conner; Reid; Jack, 2003).

3.2.4 Características sensoriais da aguardente maturada

O tipo e espécie da madeira utilizada para maturação e envelhecimento possibilita obtenção de destilados com aspectos sensoriais distintos (Francis, 1992). Análises sensoriais e físico-químicas podem demonstrar as importantes diferenças entre a origem e espécie da madeira que personalizam o caráter amadeirado devido à cinética de extração dos compostos da madeira ser diferentes (Masson; Guichard, 1995).

Ácido gálico, 5-hidroximetilfurfural, furfural, ácido vanílico, ácido sirínico, vanilina, siringaldeído, coniferaldeído, sinapaldeído, cumarina, whisky-lactona, furfural, hidroximetilfurfural e cumarina são compostos fenólicos de baixo peso molecular extraídos da madeira mediante mecanismos de degradação da celulose, hemicelulose e da lignina e participam ativamente do perfil sensorial do destilado envelhecido, assim como outros compostos minoritários, tais como açúcares, taninos, lipídeos, etc (Aylott; Mackenzie, 2010; Dias; Maia; Nelson, 1998; Puech, 1981; Puech; Jouret; Goffinet, 1985).

O processo de queima da madeira, é um dos principais responsáveis pelos descritores sensoriais em bebidas. Essa etapa realizada durante a produção dos barris tem a finalidade de dar forma às aduelas, auxiliando na envergadura. Outro processo de queima é realizado após as etapas de produção, e contribui para modificar e modular as estruturas das moléculas da madeira. Esse processo causa a degradação de polímeros, como polissacarídeos e polifenóis, e permite o surgimento de novas substâncias aromáticas, que conferem sabor diferenciado ao produto (Leão, 2006). O nível de degradação térmica da madeira pode também influenciar as características físicas da madeira, pois aumenta a superfície de contato com o líquido; as características químicas, mediante degradação térmica dos compostos, formação de novos componentes químicos e aumento do teor de compostos suscetíveis de serem extraídos. Consequentemente afetam as características sensoriais da bebida, aumentando sua complexidade aromática (Chatonnet, 1999).

A madeira não queimada possui poucas quantidades de furfural e traços de álcool furfúrilico. Esses compostos são encontrados somente após a queima final, etapa que produz importantes quantidades de aldeído furânicos a partir de açúcares. Assim, as hexoses, componentes da celulose são transformadas em hidroximetil-5-furfural (5HMF) e em metil-5-furfural, e as pentoses componentes da hemicelulose são transformadas em furfural. Esses compostos são oriundos da reação de Maillard e por desidratação (reação catalisada por ácido acético). As notas aromáticas associadas a esses compostos são “queimado”, “caramelo” e “amêndoas grelhadas”. Entre os polifenóis formados a partir da queima, destacam-se também

os originados da série guaiacil e siringil, que são responsáveis pelas sensações organolépticas de “defumado” e “especiarias” (Alcarde *et al.*, 2010; Bortoletto; Alcarde, 2013; Bortoletto *et al.* 2015).

Outros compostos de destaque são os aldeídos fenólicos, composto presente em maior quantidade na madeira queimada. Destacam-se os aldeídos benzóicos (vanilina e siringaldeído) e os aldeídos hidroxicinâmicos (coniferaldeído e sinapaldeído). Possuem notas aromáticas associadas a “fumaça”, “especiarias” e “fenólico” (Chatonnet; Dubourdieu, 1998). A maior complexidade aromática é produzida por volta de 250°C com a vanilina, formando aroma de baunilha, queimado e amêndoas. Vanilina é o maior ativador de flavor derivado da quebra da lignina (Singleton, 1995).

3.2.5 Acondicionamento com constituintes de madeira

O uso de fragmentos de madeiras é considerado acelerador de envelhecimento em bebidas (Singleton, 1995). Outras metodologias, tais como a adição de extratos ou constituintes de madeiras, têm sido utilizadas para diminuir o período de maturação e oferecer à bebida características de envelhecimento (Mosedale; Puech, 1998).

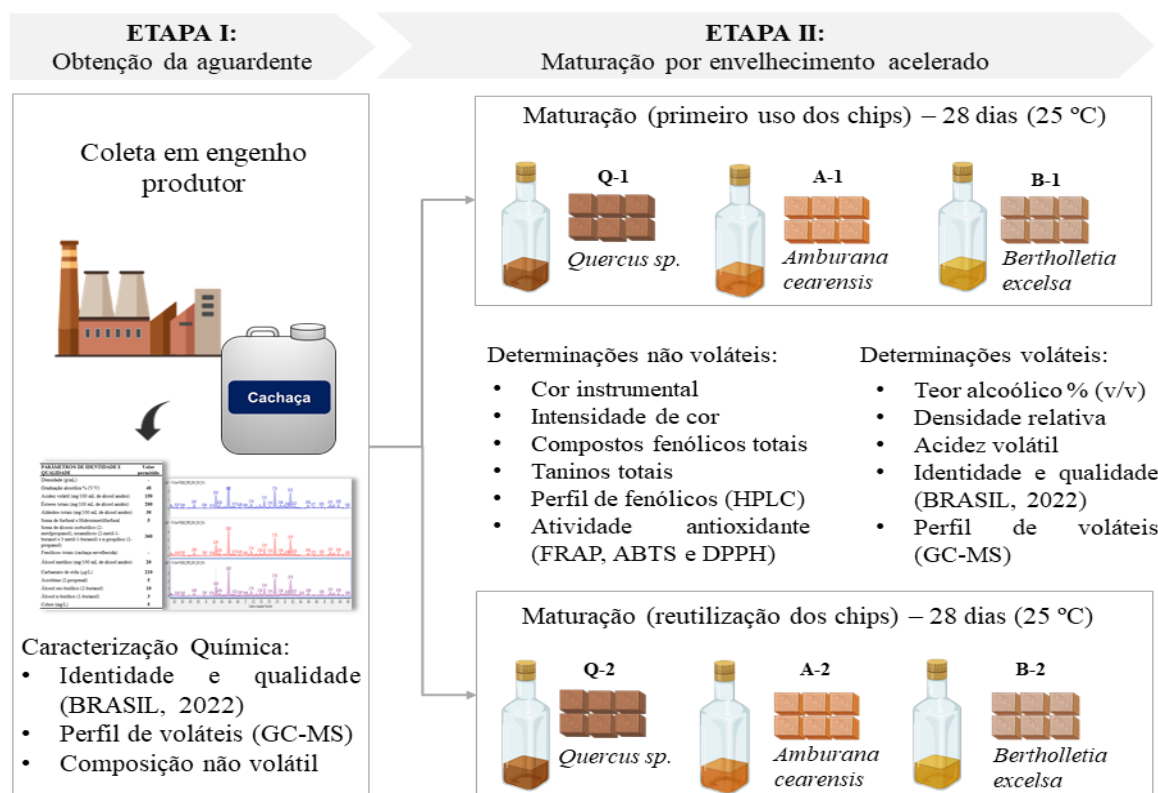
Alguns estudos com uso de lascas, chips ou extratos de madeiras (Abreu-Lima *et al.*, 2005; Borragini; Faria, 2010; Bortoletto; Alcarde, 2015; Castro Neto *et al.*, 2005), avaliaram o impacto sensorial da adição de extratos de diferentes madeiras em interação com cachaça, e compararam com cachaça envelhecida de maneira tradicional em barril de carvalho em teste sensorial de aceitação. Resultados favoráveis foram encontrados para a cachaça com extrato de bálsamo e a cachaça comercial envelhecida em ipê amarelo e destaque negativo para amostras com extratos de louro-canela e jequitibá rosa. A aplicação de circulação forçada de ar dentro de um recipiente contendo a cachaça proporcionou uma maior extração dos compostos da madeira do que o processo tradicional (Borragini; Faria, 2010; Castro Neto *et al.*, 2005).

Desta forma, persiste uma lacuna científica e tecnológica na avaliação dos impactos da utilização e subsequente reutilização de constituintes fracionados de madeira (chips) nas características químicas, bem como em sua influência nos aspectos sensoriais das bebidas.

4. DELINEAMENTO EXPERIMENTAL

A coleta de dados foi realizada por meio de pesquisa experimental para a obtenção de seis diferentes aguardentes maturadas com três tipos de “chips” de madeira (a partir de uma amostra controle – aguardente tradicional) submetidas a mais de uma utilização. O estudo verificou, além da potencialidade bioativa, o perfil de compostos voláteis e não voláteis. O delineamento experimental, apresentado na Figura 4, resume a metodologia para o desenvolvimento do projeto em duas principais etapas.

Figura 4. Delineamento experimental.



Fonte: Autor (2024).

4.1 ETAPA I: OBTENÇÃO DA AGUARDENTE

A primeira etapa do esquema representado na Figura 3 (Etapa I) representa a obtenção da amostra que foi disponibilizada por engenho produtor de cachaça, localizado no estado da paraíba, seguindo da caracterização química quanto às análises de identidade e qualidade

preconizadas na legislação brasileira (Brasil, 2022) e também para o perfil de compostos voláteis e fenólicos.

4.2 ETAPA II: MATURAÇÃO POR ENVELHECIMENTO ACELERADO

A segunda etapa do experimento (Etapa II) esquematiza o processo de maturação acelerada da aguardente em dois ciclos. Para realização da primeira maturação, o destilado coletado (sem diluição) foi homogeneizado e distribuído igualmente em três recipientes de vidro de 5 litros, adicionado junto a 6 unidades de chips por litro de bebida (recomendação do fabricante do chips) das madeiras selecionadas: carvalho (*Quercus sp.*), amburana (*Amburana cearensis*) e castanheira (*Bertholletia excelsa*), dando origem aos respectivos tratamentos Q-1, A-1 e B-1, como mostra a Tabela 1.

Tabela 1. Tratamentos para maturação com diferentes chips de madeiras.

Chips de madeira adicionado	Tratamentos	
	Primeira utilização	Segunda utilização
Carvalho (<i>Quercus sp.</i>)	Q-1	Q-2
Amburana (<i>Amburana cearensis</i>)	A-1	A-2
Castanheira (<i>Bertholletia excelsa</i>)	B-1	B-2

Após adição dos chips, os recipientes foram levados para estufa B.O.D com controle de temperatura (25 ± 1 °C) por quatro semanas (28 dias), ficando acondicionados de forma estática. Aliquotas de análise (140 mL) foram retiradas semanalmente para acompanhamento cinético da evolução química da bebida mediante as análises descritas na Tabela 2.

Tabela 2. Pontos de coleta para acompanhamento da maturação com diferentes chips de madeiras.

Ponto de coleta	Tempo de interação com chips	Análises de acompanhamento cinético
P(1)	1 dia	Teor alcoólico, densidade relativa, intensidade de cor, acidez volátil, fenólicos totais e taninos totais.
P(2)	7 dias	Teor alcoólico, densidade relativa, intensidade de cor, acidez volátil, fenólicos totais e taninos totais.

P(3)	14 dias	Teor alcoólico, densidade relativa, intensidade de cor, acidez volátil, fenólicos totais e taninos totais.
P(4)	21 dias	Teor alcoólico, densidade relativa, intensidade de cor, acidez volátil, fenólicos totais e taninos totais.
P(5)	28 dias	Teor alcoólico, densidade relativa, cor instrumental, intensidade de cor, acidez volátil, fenólicos totais, taninos totais, atividade antioxidante (DPPH, ABTS e FRAP), perfil de fenólicos, congêneres voláteis e perfil de voláteis.

Após o final da primeira maturação, as bebidas resultantes foram removidas e acondicionadas em garrafas de vidro, tampadas e levadas ao armazenamento longe da luz e com o mesmo controle de temperatura realizado durante a maturação, até realização da análise sensorial. Os chips de madeiras remanescentes da primeira maturação foram reutilizados (imediatamente) adicionando novo volume de aguardente, promovendo a segunda utilização desses constituintes, dando origem aos tratamentos Q-2, A-2 e B-2 como também é mostrado na Tabela 1. Assim, o processo de maturação foi repetido para as mesmas condições de estocagem, tempo e análises de acompanhamento cinético realizadas nas aguardentes Q-1, A-1 e B-1, afim de verificar a influência da reutilização dos chips para os mesmos parâmetros analíticos em mesmas condições de maturação.

5. RESULTADOS

Os resultados obtidos na pesquisa desenvolvida estão apresentados na forma de artigo científico em atendimento a Norma Complementar nº 01/2024 do Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos da Universidade Federal da Paraíba (PPGCTA/UFPB).

5.1 ARTIGO I - ACCELERATED AGING OF BRAZILIAN SUGARCANE SPIRIT: IMPACT OF WOOD CHIPS REUSE ON THE PHENOLIC AND VOLATILE PROFILE OF THE BEVERAGE

O Artigo I, publicado na revista científica Food Chemistry (fator de impacto 8.5), explorou o impacto do uso de chips de diferentes madeiras no envelhecimento acelerado de aguardente de cana-de-açúcar, focando nos ciclos de maturação e suas contribuições para o perfil químico das bebidas. O primeiro ciclo demonstrou maior extração de compostos fenólicos e voláteis, resultando em uma bebida mais complexa, com maior intensidade de cor e atividade antioxidante. O carvalho destacou-se por seu conteúdo fenólico e bioatividade superior, enquanto amburana e castanheira exibiram perfis únicos de compostos.

Foi identificado que a reutilização dos chips reduz a concentração de compostos bioativos e a complexidade química, embora mantenha os padrões legais de qualidade. O estudo concluiu que os chips são uma alternativa promissora para agregar qualidade às bebidas, especialmente em mercados com restrições de recursos. Contudo, a prática de reutilização dos chips apresenta limitações, especialmente na intensidade das características que definem as bebidas envelhecidas, o que leva a pesquisas futuras explorar estratégias quanto à otimização dessa tecnologia emergente.



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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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have investigated the impact of using woodchips in sugarcane spirits, an area still little explored given the change in Brazilian legislation allowing the use of the technique (Brasil. Ministério da Agricultura, 2022).

Research into the feasibility of reusing chips is scarce. However, it is crucial to understand how this practice affects the chemical composition of the beverage. Given the high cost and time involved in traditional aging, exploring alternatives that can maintain or even improve the chemical characteristics of sugarcane spirits using faster and more accessible techniques has a significant impact on both the industry and consumers (Caldeira et al., 2017; Krüger et al., 2022).

It should also be noted that oak, specifically the genus *Quercus*, is the main wood used for storing distillates, due to its recognized sensory attributes (Fernandes et al., 2020). Nonetheless, in countries where it does not occur naturally, such as Brazil, it is pertinent to evaluate the use of local woods (Coelho et al., 2021). Among the Brazilian tropical woods that make a significant contribution to the chemical profile of beverages, amburana (*Amburana cearensis*) and chestnut (*Bertholletia excelsa*) stand out. Amburana is a wood known for generating high concentrations of compounds such as vanillic acid and vanillin, as well as providing a characteristic aromatic profile (Bortoletto & Alcarde, 2013). Chestnut, on the other hand, also stands out, as it has aging compounds comparable to those found in oak (Coldea et al., 2020).

To characterize the key compounds in the maturation of distilled spirits, analytical techniques play a fundamental role, allowing for the identification and quantification of these constituents. Spectrophotometry and colorimetry are often applied to evaluate general parameters, while more robust methods, such as high-performance liquid chromatography with diode array detection (HPLC-DAD), allow for detailed phenolic characterization (Plaza et al., 2018). In addition, gas chromatography coupled with flame ionization detection (GC-FID) and mass spectrometry (GC-MS) provide a comprehensive view of the volatile profile, highlighting the impact of maturation conditions on the final composition of the beverage (Ribeiro et al., 2023).

This research aims to compare the effects of the first and second use of chips from different woods (oak, amburana, and chestnut) on migration and to elucidate the formation of phenolic and volatile compounds during the accelerated maturation of sugarcane spirits.

2. Material and methods

2.1. Obtaining the alcoholic matrix

The spirit used for maturation (alcoholic matrix) was made from sugar cane, monodistilled, stored in a stainless steel tank and did not pass through a process of dilution, standardization or filtering with active materials. It was made available by a cachaça-producing mill located in the state of Paraíba, Brazil. After collection, the beverage was transported in an inert, closed container, where it was stored under temperature control (25 ± 1 °C) until it began to mature.

2.2. Accelerated aging of sugarcane spirit

Wood chips were obtained commercially. A unit of this constituent is characterized by being cubic (1 cm^3), with an average weight of 0.90 g, and by undergoing a toasting process at 200/230 °C for 10 to 15 min.

Two maturation processes were carried out to evaluate the first and second use of the woodchips. To carry out the first use/maturation, the collected distillate (50 % ethanol, V/V) was homogenized and distributed in three inert containers containing 5 l of the spirit, added together with 6 units of chips per liter (commercial recommendations) from the selected woods: oak (*Quercus* sp.), amburana (*Amburana cearensis*) and chestnut (*Bertholletia excelsa*), giving rise to the respective treatments Q-1, A-1 and B-1.

After adding the woodchips, the containers were closed and taken to a temperature-controlled oven (25 ± 1 °C) for 28 days. Aliquots of 140

mL were taken weekly from each container containing the matured spirits to monitor the kinetic evolution of the beverage by analyzing the color intensity and total phenolics as described in section 2.4.

At the end of the first maturation cycle (28 days), the beverages obtained were bottled, and the remaining wood chips were immediately reused without undergoing drying or any additional processing. These chips were directly placed into new containers with 5 l of sugarcane spirit (alcoholic matrix) under the same conditions described for the first maturation cycle (storage, time, and monitoring analyses). This process resulted in the treatments Q-2, A-2, and B-2, corresponding to the spirits matured with chips of oak (*Quercus* sp.), amburana (*Amburana cearensis*), and chestnut (*Bertholletia excelsa*), respectively.

2.3. Identity and quality analysis of the brandies

The alcoholic beverages were assessed for standard identity and quality, as established by Brazilian legislation (Brasil. Ministério da Agricultura, 2022). The relative density, alcoholic strength (ABV % V/V), and total and volatile acidity were evaluated according to the methodologies proposed by the Ministry of Agriculture and Livestock (Brasil. Ministério da Agricultura, 2005).

The levels of total aldehydes, total esters, methyl alcohol, and higher alcohols (n-propyl, isobutyl and isoamyl) were determined using a Gas Chromatograph with Flame Ionization Detector (GC-FID) (Agilent model HP6890) following a methodology adapted from Bortoletto et al. (2016). The standards used were acetaldehyde, ethyl acetate, methanol, 1-propanol, isobutanol, isoamyl alcohol, sec-butanol, 1-butanol, acrolein, furfural and 5-HMF (Merck - Darmstadt, Germany), all of chromatographic grade with purity >99 %. The samples were added with the internal standard (3-pentanol) to quantify the analytes. Separation was carried out using a polar capillary column (CP-Wax 52CB), 60 m $\times$ 0.25 mm $\times$ 0.25 μm . The detector and injector temperatures were set at 250 °C and 245 °C, respectively, and the manual injection mode with a split flow rate of 1:25, an injection volume of 1.0 μL of the sample, in triplicate and a temperature ramp starting at 40 °C (4 min), increasing to 120 °C at a rate of 20 °C/min for 1 min and increasing at 30 °C/min to 170 °C for 4 min.

2.4. Non-Volatile characterizations

2.4.1. Instrumental color and color intensity

The instrumental color of the matured spirits and the control (alcoholic matrix) was determined using a Minolta digital colorimeter (Model CR-300, Minolta, Mahwah/New Jersey, USA) to read the parameters L^* (luminosity), a^* (red/green intensity), b^* (yellow/blue intensity), C^* (chroma) and h° (hue angle) according to the specifications of the Commission Internationale de L'éclairage (CIE, 2004). The color intensity of the spirits was determined using spectrophotometry, by directly reading the absorbance at 420 nm (Bortoletto et al., 2016).

2.4.2. Total phenolic compounds and total tannins content

The total content of phenolic compounds was assessed by spectrophotometry (at 765 nm), after reaction with Folin-Ciocalteu reagent (Amerine & Ough, 1980) using gallic acid as a standard for constructing the analytical curve in a 40 % (V/V) ethanol solution. The final concentration was expressed in mg gallic acid equivalent per 100 mL. Total tannins were also quantified by UV/VIS spectrophotometry by the colorimetric reaction of the sample with the Folin-Ciocalteu reagent, quantified at 725 nm following the methodology of Petchidurai et al. (2019), using tannic acid as a standard for constructing the analytical curve in a 40 % (V/V) ethanol solution.

2.4.3. Antioxidant activity of the sugarcane spirits

2.4.3.1. Radical Scavenging Activity 2,2-difenil-1picrilhidrazila (DPPH).

The distillates' DPPH radical scavenging capacity was determined by UV-VIS spectrophotometry (Quimis, Q798U, São Paulo, Brazil) at 515 nm according to the methodology described by Brand-Williams et al. (1995). The calibration curve was prepared with Trolox (20–1200 μM), and the results were expressed as a percentage of radical oxidation inhibition.

2.4.3.2. Radical Scavenging Activity 2,2'-azino-bis (3-ethylbenzothiazolyl)-6-ácido sulfônico (ABTS). The ABTS⁺ radical scavenging activity was determined according to the antioxidant capacity of the extracts and checked at 734 nm in a UV-VIS spectrophotometer (Quimis, Q798U, São Paulo, Brazil) according to the methodology proposed by Re et al. (1999). The standard curve was made with Trolox (20–1600 μM), and the results were expressed as a percentage of inhibition, based on the decrease in absorbance about the blank at time zero.

2.4.3.3. Iron Reducing Antioxidant Power (FRAP). The antioxidant potential of the samples through their ability to reduce iron (Fe^{3+}) to the ferrous form (Fe^{2+}) was checked at 595 nm in a UV-VIS spectrophotometer (Quimis, Q798U, São Paulo, Brazil) using the methodology described by Benzie and Strain (1999), with adaptations. Based on the calibration curve prepared with different concentrations of Trolox (50–1000 μM), the results were expressed as a percentage of iron reduction.

2.4.4. Phenolic compounds profile

The individual phenolic compounds were determined following the methodology validated by Padilha et al. (2017), using an Agilent 1260 Infinity LC System Liquid Chromatograph (Agilent Technologies, Santa Clara - USA) coupled to a Diode Array Detector (DAD) (model G1315D). The data was processed using OpenLAB CDS ChemStation Edition software (Agilent Technologies, Santa Clara - USA). The column used was Zorbax Eclipse Plus RP-C18 (100 $\times$ 4.6 mm, 3.5 μm) and the pre-column was Zorbax C18 (12.6 $\times$ 4.6 mm, 5 μm) (Zorbax, USA). The oven temperature was 35 °C and the injection volume was 20 μL of the sample, previously diluted in phase A (phosphoric acid solution) and filtered through a 0.45 μm membrane (Millex Millipore, Barueri, SP, Brazil). The solvent flow rate was 0.8 mL per minute. The gradient used in the separation was 0–5 min: 5 % B; 5–14 min: 23 % B; 14–30 min: 50 % B; 30–33 min: 80 % B where solvent A was a solution of phosphoric acid (0.1 M, pH = 2.0) and solvent B was methanol acidified with 0.5 % H_3PO_4 . The compounds were detected at 220, 280, 320, 360 and 520 nm, and identified and quantified by comparison with external standards. The samples were previously diluted (1:1 V/V) with phosphoric acid solution (0.52 %) and filtered using a filtrate filter with a PVDF membrane (diameter 13 mm, pore size 0.45 μm).

2.5. Volatile compounds profile

The samples were prepared by adding 3.6 mL of ultrapurified water, 0.4 μL of the alcoholic beverage, and 4 μL of the internal standard (3-pentanol at a concentration of 1 mg/mL) in 15 mL vials. Headspace solid-phase microextraction (HS-SPME) was carried out in a water bath at 45 °C, leaving the 50/30 μm PDMS/Carboxen/DVB SPME fiber (Supelco, Bellefonte, PA, USA) exposed without equilibrium for 50 min, activated according to the manufacturer's recommendations (270 °C/60 min), with desorption lasting 5 min.

The compounds were separated using a 7890B gas chromatograph coupled to an Agilent Technologies 5977B mass spectrometer (Little Falls, DE, USA). The column used was HP-5MS (60 m $\times$ 0.25 mm $\times$ 0.25 μm). The following conditions from the methodology of Zacaroni et al. (2017) were used: oven temperature from 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 the carrier gas at a flow rate of 1.78 mL/min in a 1:4 split-mode injection system. The interface temperature of the detector and

ion source remained at 240 °C and 200 °C, respectively. The mass spectrometer was operated in electron impact mode (70 eV) and the mass scan range was 50 to 400 m/z at 4.44 scan.s⁻¹.

Compound identification was carried out using the NIST library (2014) and confirmed using the linear retention index (LRI) calculated from the injection of a series of C6-C20 n-alkanes, with the results expressed in concentration ($\mu\text{g/L}$).

2.6. Statistical analysis

To investigate the influence of the type of wood (W), the number of uses of the chips (N) and the interaction between these variables (WxN), two-way ANOVA with multiple comparisons using Tukey's post hoc test was used. For comparisons with the alcohol matrix (control), ANOVA was used with Dunnett's post hoc test at a 5 % significance level. All analyses of variance were carried out using XLSTAT software (version 2014.5.03, Addinsoft, New York, USA).

Principal component analysis, PCA-Biplot, was carried out for the non-volatile characterization of spirits using XLSTAT software, with the data pre-processed (auto-scaling). For the chemometric analysis of the profile of volatile compounds, a PCA was carried out, along with a heat map and hierarchical clustering. These analyses were conducted, respectively, using the Chemometrics Web App (Darzé et al., 2023), developed in the R programming language, and the TBools-II v2.105 software (Chen et al., 2023), using autoscaled data.

3. Results and discussion

3.1. Identity and quality standards for the distillates obtained

The parameters required by Brazilian legislation were evaluated (Table 1) and it can be seen that the process of accelerated maturation through the use of chips, independently of the wood species, has a significant influence on the increase in the congener coefficient, represented by the sum of total aldehydes (in acetaldehyde), total esters (in ethyl acetate), higher alcohols (1-propanol, isobutanol and isoamyl), furfural and hydroxymethylfurfural and volatile acidity (in acetic acid) compared to the control.

This behavior is to be expected and is also evidenced in traditional aging processes in wooden barrels, as the distillate-wood interaction promotes extraction, degradation and oxidation phenomena of chemical species present in plant constituents, resulting in higher concentrations of congeners (Bortoletto et al., 2021). However, even though this influence has a significant effect on quality compounds, it results in distillates that remain within the legal limits of identity and quality (Brasil. Ministério da Agricultura, 2022). Quality assurance of spirits is of great industrial importance due to the control and safety of the beverage for sale.

The presence of wood (chips) in contact with the distillate was enough to increase the amount of total esters. Esters are mainly produced during fermentation by yeasts but are also formed during aging by the esterification of acids with ethanol or acetic acid. This process occurs more slowly in inert containers than in barrels, where the presence of oxygen accelerates the production of aromatic esters (Bortoletto et al., 2021; Lima et al., 2022).

On the other hand, total aldehydes, which are also present in greater abundance in spirits that interact with woodchips compared to the matrix, are influenced by the reuse of these constituents (*p*-value 0.0008), so that the highest concentrations of aldehydes are found in spirits A-1, Q-1, B-1 and A-2, which do not differ from each other, but with Q-2 and B-2 being the samples with the lowest concentrations. Aldehydes result from the oxidation of ethanol and acids, as well as the degradation of phenolic compounds normally extracted from wood during the maturation process (Castro et al., 2023). Maturation with chips is enough to raise the aldehyde content by rapid interaction, since in barrels this process is slower - as in amburana - where there was no

Table 1

Physico-chemical characterizations of sugar cane spirits aged for 28 days with chips reused from different woods.

Items	Maximum levels***	Non-aged spirit (control)	Quercus sp.		Amburana cearensis		Bertholletia excelsa		p-value		
			First use	Second use	First use	Second use	First use	Second use	Wood Chips (W)	N° of Use (N)	W x N
Legal parameters											
ABV % (V/V)	54 to 38	50.5 ± 0.2	50.1 ± 0.1 ^a	50.4 ± 0.2 ^a	50.2 ± 0.3 ^a	50.4 ± 0.4 ^a	50.1 ± 0.1 ^a	50.0 ± 0.1 ^a	0.2180	0.2787	0.2667
Volatile acidity (acetic acid)**	150	18.0 ± 0.7	41.9 ± 0.6 ^{c,*}	51.1 ± 1.2 ^{ab,*}	33.3 ± 2.2 ^{d,*}	48.5 ± 2.5 ^{b,*}	38.2 ± 2.2 ^{cd,*}	55.7 ± 1.2 ^{a,*}	0.00013	< 0,0001	0.00484
Total aldehydes (acetaldehyde)**	30	5.7 ± 0.1	8.3 ± 0.6 ^{ab,*}	7.4 ± 0.3 ^{b,*}	8.7 ± 0.6 ^{ab,*}	7.5 ± 0.4 ^{ab,*}	7.9 ± 0.4 ^{ab,*}	7.1 ± 0.4 ^{b,*}	0.1055	0.0008	0.7110
Methyl alcohol**	20	0.63 ± 0.07	0.87 ± 0.06 ^a	0.5 ± 0.3 ^a	0.8 ± 0.1 ^a	0.2 ± 0.2 ^a	0.6 ± 0.5 ^a	0.8 ± 0.3 ^a	0.5003	0.0900	0.1202
Total esters (ethyl acetate)**	200	35.3 ± 0.3	46.4 ± 1.2 ^{ab,*}	42.9 ± 1.8 ^{ab,*}	46.9 ± 2.4 ^{ab,*}	43 ± 3 ^{ab,*}	43.8 ± 1.2 ^{ab,*}	42.9 ± 5.1 ^{ab,*}	0.5977	0.0594	0.6123
Higher alcohols (Sum of isobutyl, isoamyl and n-propyl alcohols)**	360	118.6 ± 0.9	160.0 ± 9.1 ^{ab,*}	149.3 ± 7.7 ^{ab,*}	167 ± 12 ^{ab,*}	149.3 ± 8.2 ^{ab,*}	155.4 ± 8.5 ^{ab,*}	147.9 ± 7.1 ^{ab,*}	0.4606	0.0143	0.6005
Isobutyl alcohol**	–	65.6 ± 0.4	88.3 ± 4.8 ^{ab,*}	82.4 ± 4.8 ^{ab,*}	92 ± 6 ^{ab,*}	82.0 ± 4.6 ^{ab,*}	85.7 ± 4.7 ^{ab,*}	81.3 ± 3.9 ^{ab,*}	0.498	0.012	0.605
Isoamyl alcohol**	–	20.1 ± 0.3	26.9 ± 1.5 ^{ab,*}	25.4 ± 1.2 ^{ab,*}	28.0 ± 2.6 ^{ab,*}	25.5 ± 1.5 ^{ab,*}	26.2 ± 1.2 ^{ab,*}	25.1 ± 1.5 ^{ab,*}	0.519	0.047	0.742
n-Propyl alcohol**	–	32.9 ± 0.2	44.8 ± 2.8 ^{ab,*}	41.5 ± 1.8 ^{ab,*}	47.3 ± 3.5 ^{ab,*}	41.9 ± 2.1 ^{ab,*}	43.5 ± 2.6 ^{ab,*}	41.5 ± 1.7 ^{ab,*}	0.3603	0.0104	0.5144
n-butyl alcohol (1-butanol)**	3	0.48 ± 0.06	0.49 ± 0.06 ^a	0.47 ± 0.03 ^a	0.6 ± 0.343	0.55 ± 0.08 ^a	0.6 ± 0.1 ^a	0.44 ± 0.02 ^a	0.4870	0.3499	0.6809
Sec-butyl alcohol (2-butanol)**	10	0.368 ± 0.004	0.360 ± 0.002 ^a	0.356 ± 0.006 ^a	± 0.009 ^a	0.352 ± 0.009 ^a	0.39 ± 0.06 ^a	0.357 ± 0.008 ^b	< 0,0001	0,0001	0,0001
Sum of furfural and hydroxymethylfurfural**	5	0.09 ± 0.02	0.86 ± 0.05 ^{ab,*}	0.12 ± 0.02 ^c	0.27 ± 0.02 ^{b,*}	0.12 ± 0.02 ^c	0.37 ± 0.08 ^{b,*}	0.14 ± 0.04 ^c	< 0,0001	< 0,0001	< 0,0001
Acrolein**	5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	–	–	–

Different lowercase letters in the rows indicate significant differences between the aged spirits (Tukey's HSD, post-hoc test, $P < 0.05$). *Post hoc Dunnett ($p < 0.05$); one asterisk indicates a statistically significant difference in relation to the control (non-aged spirit). **mg/100 mL anhydrous alcohol. *** Limits of quality and identity standards for spirits (Brasil, 2022).

increase in the total aldehyde content as found by Bortoletto and Alcarde (2013).

The first use of the chips led to a significant increase in the sum of furfural and hydroxymethylfurfural (5-HMF) compared to the control. This can be attributed to the fact that the chips undergo a toasting process and furfural and 5-HMF are formed by the thermal degradation of pentoses and hexoses, respectively, derived from cellulose and lignin present in wood (Bortoletto & Alcarde, 2013).

Spirit Q-1 had the highest concentrations of furfural and 5-HMF, followed by spirits A-1 and B-1, which did not differ from each other. This behavior was also reported by Bortoletto and Alcarde (2013), since among the ten wood barrels analyzed, oak had the highest content of these components. This indicates that even though they come from the same type of toast, oak favors greater extraction of furfural compounds due to its fibrous structure, which offers greater permeability to ethanol (Perez-Coello et al., 1999; Puech, 1987). However, the reuse of these chips significantly reduced these compounds in Q-2, A-2 and B-2, which did not differ from the control or from each other, with an average concentration of 0.126 mg/100 mL. Because they have already been through an initial extraction process, the reused wood has less availability of derivative compounds, which explains why the furfuryl compounds did not increase in concentration.

In addition, the maturation of spirits using the chips was also significant in increasing the volatile acidity of the beverages, with all batches starting at 18.04 ± 0.66 mg/100 mL (alcoholic matrix) and ending maturation with acidity varying between 33 and 55 mg/100 mL, showing a significant effect of the interaction between the type of wood and the number of uses of the chips (W x N).

Volatile acidity is one of the most important parameters for process control in distilled beverages and increasing the number of uses of chips promotes distillates with higher volatile acidity. Aging adds non-volatile organic acids that also favor increased acidity in the beverage, such as those derived from phenolic compounds (gallic, tannic, syringic and

vanillic). In addition, the oxidation of ethanol to acetaldehyde leads to the formation of acetic acid, but it can also be formed due to the degradation of hemicellulose. Thus, acidity is generated both during the production of spirits and during aging (Bortoletto & Alcarde, 2013; Castro et al., 2023).

Methanol, 1-butanol and 2-butanol and acrolein were not influenced by the maturation process, neither by the use of the chips (W) nor by the number of uses (N). These compounds are formed mainly during the fermentation process and are recovered by distillation (Da Silva et al., 2023). They are controlled due to their negative health implications and their formation is controlled during fermentation. Therefore, good management and control of the production process implies reduced quantities of these parameters.

One of the characteristics of distillates aged in barrels is that over a long period of time, even years of interaction, there is a reduction in the alcohol content and a decrease in the volume of the distillate (Da Silva et al., 2023), which explains their high commercial cost. Alternatively, the process of accelerated maturation using chips does not result in distillates with significant differences in alcohol content (ABV%), thus implying constancy in the volume of the beverage and advantages for producers by reducing volumetric loss, and directly, costs.

3.2. Kinetic monitoring of accelerated aging

The maturation process of the spirits was monitored through the release of total phenolic compounds (TPC) and color intensity, with Fig. 1 (A), (B) and (C) presenting a detailed view of how this process develops over the interaction time, considering the type of woodchips and their uses.

In the graphs in Fig. 1, the bars represent the TPC, while the area represents the color intensity of the drinks. It can be seen that the reuse of the chips results in a significant reduction in phenolic compounds at each kinetic point, from the 1st to the 28th day of interaction,

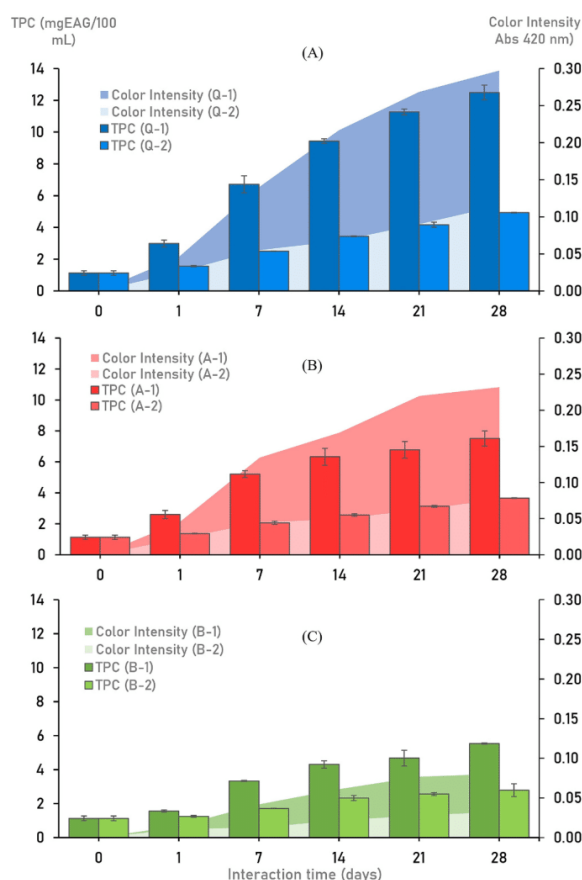


Fig. 1. Increase in total phenolic compounds (TPC) and color intensity of brandies stored with *Quercus* sp. chips (A), *Amburana cearensis* chips (B), and *Bertholletia excelsa* chips (C). Note (s): TPC- Total Phenolic Compounds (mg EAG/100 mL); Q-1: sugarcane spirit aged with *Quercus* sp.chips - first use. Q-2: sugarcane spirit aged with *Quercus* sp.chips - second use. A-1: sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use.

accompanied by a decrease in color intensity (lighter areas). In the first use, all types of chips showed an exponential increase in total phenolic compounds and color intensity, while in the second use, the evolution followed a linear pattern (Table S1).

The spirit packaged with oak chips showed the highest content of TPC from the 14th day of interaction compared to the other woods, also resulting in greater color intensity. The potential of oak to add a higher amount of TPC is noteworthy since the beverage resulting from its reuse (Q-2) is equivalent to the beverage from the first use of the chestnut (B-1). On the other hand, spirits packaged with amburana chips showed intermediate results compared to oak and chestnut in the two uses of the chips. Smailagić et al. (2021) reported that the greatest extraction of polyphenols, the main compounds responsible for antioxidant activity, occurs in the first 30 days of aging.

The results obtained for the total phenolic content corroborate the trend of increased phenolic compounds proportional to the maturation time, as also observed by Nie et al. (2023), who evaluated the effect of adding oak chips to persimmon spirits. Comparatively, the phenolic contents of samples Q-1 (12.5 mg GAE/100 mL) and Q-2 (4.9 mg GAE/100 mL) exceeded those reported for oak barrels (4.87 mg GAE/100

mL), as noted by Bortoletto and Alcarde (2013). These authors also observed a total phenolic content (TPC) of 5.49 mg GAE/100 mL, an intermediate value between those found for samples A-1 (7.6 mg GAE/100 mL) and A-2 (3.7 mg GAE/100 mL) in the present study. It is noteworthy that the total phenolic content obtained for all accelerated maturation beverages is comparable to that of barrel aging.

Similarly, the color intensity results obtained for samples using oak chips (Absorbance of 0.297) and amburana chips (Absorbance of 0.232) were also higher than those reported by Bortoletto and Alcarde (2013) for woods of the same genus when used in barrels over 36 months. In the cited study, samples aged in oak barrels (*Quercus sessilis*) and amburana barrels (*Amburana cearensis*) exhibited respective color intensity values (Abs 420 nm) of 0.197 and 0.127.

Although the castanheira wood provided the lowest color intensity among the woods evaluated in this study (0.080), its results were still superior to those observed for other wood barrels, such as Amendoim (*Pterogyne nitens*) and Araruva (*Centrolobium tomentosum*), which showed final color intensity values of 0.036 and 0.051, respectively (Bortoletto & Alcarde, 2013). These values are comparable to those of the reused chip samples, such as Q-2, A-2, and B-2, which showed color intensities of 0.115, 0.077, and 0.033, respectively. These findings reinforce that the accelerated maturation method with chips can promote faster color formation compared to traditional barrel aging.

Additionally, the mathematical correlation between phenolic compounds and color intensity is shown in Table S1, proposing a linear model with a correlation coefficient (R^2) above 0.9. This indicates an adequate fit between the data and suggests a prediction model that allows producers to predict and pause ripening based on direct absorbance readings (spectrophotometry), providing accurate estimates of phenolic content without the need to carry out the reaction with the Folin-Ciocalteu reagent, making the process more practical for the day-to-day industry.

In addition, Wimalasiri et al. (2024) highlighted the influence of phenolic compounds on the stability and sensory quality of beverages such as wines, correlating with current findings that relate color intensity to phenolic content. The reduction in phenolic compounds in the second use of the chips can be explained by the decreased availability of these compounds after the first maturation cycle, as described by Castro et al. (2023), who observed a decrease in the extraction of phenolic compounds in reused oak barrels.

3.3. Non-volatile characterization

In addition to color intensity, the beverages were characterized in terms of instrumental color parameters, with the results shown in Fig. 2. The colorimetric analysis showed a pattern of behavior among the samples, as all the beverages resulting from the interaction with woodchips resulted in a decrease in L^* and a^* , along with an increase in the b^* , hue and C^* parameters.

The decrease in luminosity (L^*) in the samples shows that the spirits have become darker due to the incorporation of compounds from the wood chips. This phenomenon is common in aging processes, as the wood is responsible for contributing a darker color (González-Saiz et al., 2014). On the other hand, the increase in b^* points to a more yellowish coloration of the beverages, and together with the C^* parameter (saturation), reinforces that the samples tended to become more saturated, with a more pronounced yellow color. The increase in these variables is due to the phenolic compounds, which promote the formation of chromophores that intensify the color in the samples (Moya et al., 2012).

The same behavior of variables, by the decrease in L^* with an increase in yellow color (b^*) was also evidenced by Nie et al. (2023) and Yan et al. (2024) with maturations of distillates using wood chips. This characteristic was more present in samples Q-1 and A-1, while samples Q-2, A-2 and B-1 had equivalent coloration. B-2 was closer to the alcoholic matrix (control).

The a^* and hue parameters showed less significant variations in the

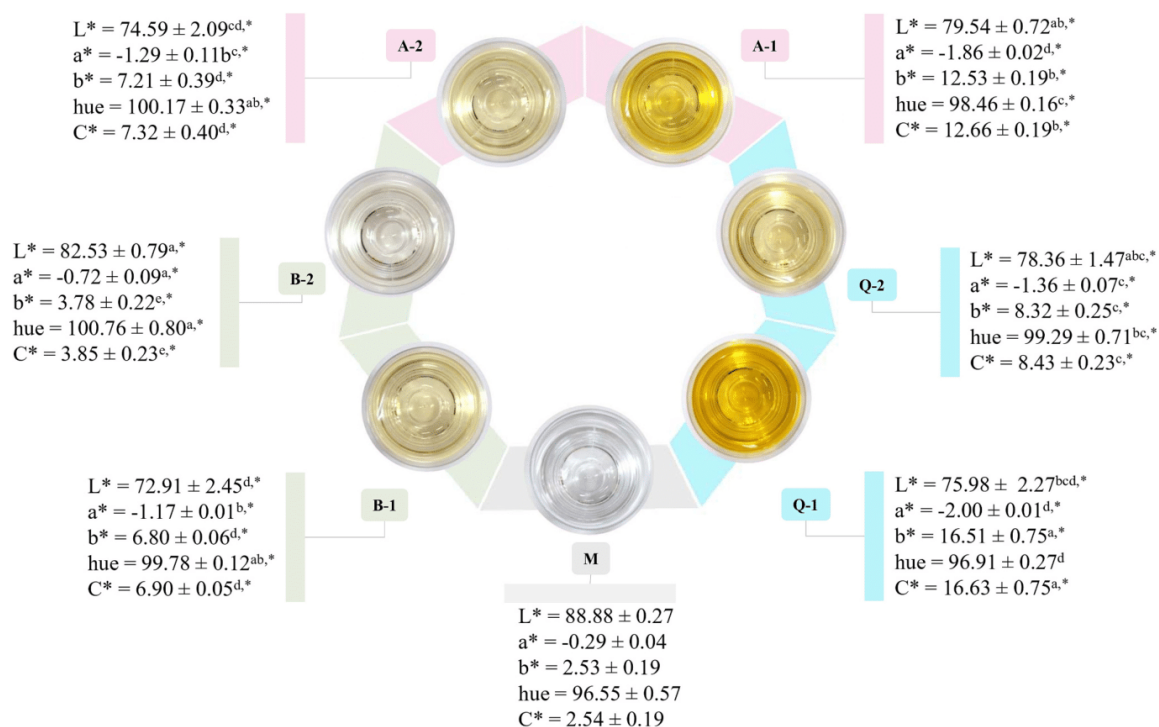


Fig. 2. Instrumental color of spirits obtained through the accelerated maturation process with different wood chips. Note (s): Different lowercase letters for each instrumental color parameter (L^* , a^* , b^* , hue and C^*) indicate significant differences between the aged distillates (Tukey's HSD, post-hoc test, $P < 0.05$). *An asterisk in the mean values indicates a statistically significant difference from the control (unaged distillate). A-1: Sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: Sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use. Q-1: sugarcane spirit aged with *Quercus* sp. chips - first use. Q-2: sugarcane spirit aged with *Quercus* sp. chips - second use.

samples. The a^* variable showed negative initial values in all the samples, indicating a tendency towards slightly greenish tones as it decreased. This coloration is subtle, however, as the main change observed is on the yellow-blue axis, where the increase in b^* predominates.

The increase in hue (hue angle) suggests that the samples have become more vibrant compared to the alcoholic matrix, showing colorations with similar hues in the sample groups: B-2, A-2 and B-1; and Q-2, A-1 and Q-1. The similarity in hue values between the sample groups suggests that, despite the differences in color intensity, the final hue stabilizes in similar ranges after interaction with the chips.

These changes in the samples indicate that the interaction with the wood chips plays a crucial role in the evolution of the spirits' coloration, promoting desirable color characteristics that enhance the aging process. In addition, the variations observed between the samples raise questions about the chemical species responsible for these changes, which can be better understood by determining and analyzing the profile of phenolic compounds present.

3.3.1. Phenolic compounds profile

The phenolic compound profile of the spirits obtained (Table 2) revealed a total of 16 compounds, distributed into three main classes: phenolic acids and lignin derivatives, flavonoids and stilbenes. Flavonoids make up the bulk of the phenolic compounds found and can be subdivided into three subgroups: flavonones, flavonols, and condensed tannins or proanthocyanidins. Our findings on the influence of wood on the release of phenolic compounds align with previous studies, such as Smailagić et al. (2021), who demonstrated significant variations in chemical profiles depending on the wood species and aging duration.

Among the compounds of interest that signal positive chemical quality for aged distillates, adding complexity of flavor, the compounds derived from lignin stand out, especially as a result of the oxidation of coniferyl (Vanillin and Vanillic Acid), sinapyl (Syringic acid) and p-coumaric (p-coumaric acid) alcohols (Bortoletto & Alcarde, 2013; Cerinišev, 2017).

Chestnut wood stood out for its presence of vanillin, being the only type of chip that, even when reused, continued to release this compound. Comparatively, the average vanillin concentration for B-1 of 2.4 (mg/L) is higher than that found by Yan et al. (2024), who presented results in the range of 0.58–2.17 (mg/L) for different beverages matured with chips and oak barrels.

In contrast, although amburana had a lower concentration of vanillin on first use, it was four times more expressive in the presence of vanillic acid than in the other woods. This suggests that, for amburana, the conversion of vanillin into vanillic acid occurs more quickly than for the other woods during interaction with the distillate. The absence of vanillin in A-2 and Q-2, together with the low concentration in B-2 and the non-detection of vanillic acid in all the reuse spirits, suggests that the guaiacyl pathway (degradation of coniferyl alcohol) is practically completed after the first use of the woods.

Syringic acid, resulting from the syringyl pathway, was found in all the samples, varying in concentration according to the species of wood and the number of uses of the chips. The presence of syringic acid suggests that as a result of lignin degradation, syringaldehyde is rapidly oxidized to the corresponding benzoic acid (Cabrita et al., 2011). Spirit Q-1 had an average concentration of syringic acid (3147 mg/L) approximately twice as high as B-1 (1779 mg/L) and three times as high as A-1 (1139 mg/L). In reuse, all the spirits had concentrations below 1

Table 2

phenolic compounds of sugarcane spirit aged for 28 days with reused chips from different woods.

Phenolics compounds	Non-aged spirit (control)	Quercus sp.		Amburana cearensis		Bertholletia excelsa		Two-way ANOVA		
		First use	Second use	First use	Second use	First use	Second use	Wood Chips (W)	N° of Use (N)	W x N
Phenolic acids and lignin derivatives										
Vanillic acid**	n.d	1.393 ± 0.002 ^{b,*}	n.d	5.813 ± 0.003 ^{a,*}	n.d	0.89 ± 0.03 ^{c,*}	n.d	< 0,0001	< 0,0001	< 0,0001
Syringic acid**	n.d	3.147 ± 0.003 ^{a,*}	0.352 ± 0.004 ^{d,*}	1.139 ± 0.003 ^{c,*}	0.29 ± 0.01 ^{e,*}	1.779 ± 0.004 ^{b,*}	0.352 ± 0.006 ^{d,*}	< 0,0001	< 0,0001	< 0,0001
p-coumaric acid**	n.d	0.491 ± 0.003 ^{a,*}	n.d	n.d	n.d	0.363 ± 0.005 ^{b,*}	n.d	< 0,0001	< 0,0001	< 0,0001
trans-cinnamic acid**	0.18 ± 0.03	0.214 ± 0.001 ^{a,*}	0.1835 ± 0.0001 ^d	0.194 ± 0.002 ^{bc,*}	0.201 ± 0.005 ^{b,*}	0.193 ± 0.006 ^{bcd,*}	0.185 ± 0.002 ^{cd}	0.00108	< 0,0001	< 0,0001
Vanillin**	n.d	1.973 ± 0.001 ^{b,*}	n.d	1.373 ± 0.006 ^{c,*}	n.d	2.400 ± 0.008 ^{a,*}	0.520 ± 0.003 ^{d,*}	< 0,0001	< 0,0001	< 0,0001
Flavonoids										
Flavonones										
Naringenin**	n.d	n.d	n.d	3.534 ± 0.006 ^{a,*}	1.026 ± 0.001 ^{b,*}	n.d	n.d	< 0,0001	< 0,0001	< 0,0001
Hesperitin**	n.d	n.d	n.d	0.85 ± 0.01 ^{a,*}	n.d	n.d	n.d	< 0,0001	< 0,0001	< 0,0001
Flavonols										
Catechin**	1.47 ± 0.01	1.25 ± 0.02 ^{c,*}	1.461 ± 0.004 ^{ab}	1.497 ± 0.003 ^a	1.45 ± 0.04 ^b	1.067 ± 0.003 ^{d,*}	1.453 ± 0.003 ^{ab}	< 0,0001	< 0,0001	< 0,0001
Epicatechin**	n.d	0.748 ± 0.004 ^{a,*}	n.d	n.d	0.294 ± 0.006 ^{c,*}	0.42 ± 0.04 ^{b,*}	0.290 ± 0.008 ^{c,*}	< 0,0001	< 0,0001	< 0,0001
Epigallocatechin**	n.d	0.897 ± 0.001 ^{a,*}	n.d	n.d	n.d	n.d	n.d	< 0,0001	< 0,0001	< 0,0001
Epicatechin gallate (ECG)**	2.914 ± 0.002	3.128 ± 0.009 ^{a,*}	2.950 ± 0.009 ^{c,*}	3.0731 ± 0.0002 ^{b,*}	2.942 ± 0.001 ^{c,*}	2.945 ± 0.001 ^{c,*}	2.91 ± 0.02 ^d	< 0,0001	< 0,0001	< 0,0001
Epigallocatechin gallate (EGCG)**	n.d	0.577 ± 0.006 ^{b,*}	n.d	2.622 ± 0.002 ^{a,*}	n.d	0.28 ± 0.04 ^{c,*}	n.d	< 0,0001	< 0,0001	< 0,0001
Quercetin 3-glucoside**	n.d	2.811 ± 0.007 ^{b,*}	1.60 ± 0.02 ^{d,*}	n.d	n.d	3.0 ± 0.1 ^{a,*}	1.77 ± 0.02 ^{c,*}	< 0,0001	< 0,0001	< 0,0001
Condensed tannins										
Procyanidin B1**	n.d	0.045 ± 0.002 ^{a,*}	n.d	n.d	n.d	n.d	n.d	< 0,0001	< 0,0001	< 0,0001
Procyanidin B2**	n.d	0.779 ± 0.003 ^{a,*}	n.d	0.588 ± 0.005 ^{b,*}	n.d	0.496 ± 0.002 ^{c,*}	n.d	< 0,0001	< 0,0001	< 0,0001
Stilbenes										
trans-resveratrol**	n.d	0.330 ± 0.001 ^{b,*}	n.d	n.d	n.d	0.37 ± 0.01 ^{a,*}	0.225 ± 0.002 ^{c,*}	< 0,0001	< 0,0001	< 0,0001

Different lowercase letters in the rows indicate significant differences between the aged spirits (Tukey's HSD, post-hoc test, $P < 0.05$). *Post hoc Dunnett ($p < 0.05$); one asterisk indicates a statistically significant difference in relation to the control (non-aged spirit). **mg/L.

mg/L. Sample Q-1 has a sirinic acid concentration in the same range as that found by a study that also matured oak chips, with an average value of 3.8 (mg/L), corroborating the literature (Yan et al., 2024).

Thus, in maturation with chips, the syringyl pathway is predominantly formed in oak and chestnut, resulting in syringic acid as the main benzoic acid, while the guaiacyl pathway is favored in amburana, with vanillic acid being the main benzoic acid. This behavior corroborated the study by Bortoletto and Alcarde (2013), where cachaça stored for 36 months in Amburana barrels was found to be expressive for vanillic acid compared to other woods, and was highlighted as a chemical marker for this species.

In addition, average amounts of 0.491 and 0.363 mg/L of p-coumaric acid were found only in Q-1 and B-1, a compound also derived from lignin and present in small concentrations in distillates aged for years (Puech, 1987). The presence of p-coumaric acid and syringic and vanillic acids are quality indicators in aged beverages, and especially for first-use spirits, accelerated maturation was considered positive, highlighting the potential of the chips to confer aging characteristics in a short period of interaction.

trans-Cinnamic acid was the only phenolic acid found in the alcoholic matrix (control). The presence of this compound in unaged distillate

comes from fermentation, as yeast (*S. cerevisiae*) is capable of producing this compound and its derivatives (Gottardi et al., 2017). However, during maturation, there was an increase in the concentration of trans-cinnamic acid in spirits Q-1, A-1, A-2, and B-1 compared to the control. This behavior is expected since trans-cinnamic acid can also be formed by the oxidation of hydroxycinnamic acids from wood lignin (Cernişev, 2017).

In addition to phenolics derived from lignin, other compounds such as stilbenes, flavonoids, and tannins can also be extracted from the cellular structures of wood and can give a unique chemical profile to the distillates obtained. Trans-Resveratrol was the only stilbene quantified and was present in spirits B-1, B-2 and Q-1. The addition of wood chips from the *Quercus* genus to beverages increases the trans-resveratrol content, since oak is known to release significant amounts of this compound due to its chemical composition rich in stilbenes (Gortzi et al., 2013; Jung et al., 2016). The chestnut can also release trans-resveratrol, especially in the first use of the chips, when the levels of phenolic compounds are still high. In the second use, the concentration tends to be lower, but still relevant (0.225 ± 0.002 mg/L).

The presence of flavonones, represented by Naringenin and Hesperitin, was found exclusively in the beverages used with amburana

chips. Naringenin was present in spirits A-1 and A-2, with a reduction of approximately 70 % in its concentration due to the reuse of the chips. Hesperitin was only found in the spirit resulting from the first use of the chips (A-1). In addition to their antioxidant function, these compounds are also found in citrus peels (Liga et al., 2023), which may give distillates that interact with this type of wood their distinctive sensory characteristics.

About quantified tannins, sample Q-1 stands out for its higher concentration of Procyanidin B2 (0.779 mg/L), compared to A-1 (0.588 mg/L) and B-1 (0.496 mg/L), and was the only sample to show minimal concentrations of Procyanidin B1, showing that the use of oak chips results in distillates with a higher concentration of tannins. Tannins are phenolic compounds associated with plant defense, being an antioxidant and antimicrobial agent (Geissman, 1963). However, they also play a role in adding color and developing flavor in distillates, since they are also associated with increased astringency and bitterness (De Simón et al., 2014).

Among the flavonols present in the spirit samples (including the alcoholic matrix), there were concentrations of Catechin and Epicatechin gallate (ECG) above 1.0 mg/L and 2.9 mg/L, respectively. These compounds may come from the beverage production process, since the extraction of sugarcane juice can add compounds derived from its plant walls that can migrate to the distillate, in addition to sucrose (mostly) (Cernișev, 2017).

The decrease in the concentration of Catechin in spirit Q-1 can be explained by the formation of Procyanidin B1 in this sample since this tannin is the result of the condensation of catechin with its isomer epicatechin. Unlike Catechin, during maturation, there was an increase in GCE due to the use of the woods, except B-2, which did not differ from the control. ECG is an esterified form of epicatechin with gallic acid. These compounds are important antioxidants and may also be involved in oxidation reactions that alter the taste of aged distillates (Méndez & Mato, 1997).

Epicatechin was found in samples Q-1, B-1, B-2 and A-2. The presence of epicatechin in beverages matured by the reuse of amburana and chestnut chips may be associated with the degradation of procyanidin B2, which was present in A-1 and B-1 since the latter is the result of the condensation of two epicatechin molecules. On the other hand, the presence of Epicatechin in Q-1 and B-1 may indicate a similar phenolic composition, as Epicatechin is present in addition to tannins.

The only sample to show Epigallocatechin was Q-1, which may be characteristic of this type of plant species or may have come from the de-

esterification of Epigallocatechin gallate (EGCG). The EGCG compound was only found in the samples from the first use of the chips, with A-1 standing out among the others for having four times higher concentrations of EGCG than Q-1 and nine times higher than B-1. However, the reuse of the chips was significant for the absence of Epigallocatechin gallate and Epigallocatechin in the spirits.

The flavonol Quercetin 3-glucoside was identified in spirits matured with oak and chestnut chips. It is noteworthy that the type of wood influences the presence of this phenolic, as in both uses, spirits derived from chestnut form more Quercetin 3-glucoside than those from oak, with the first use of these species exhibiting higher concentrations of the flavonol. Quercetin 3-glucoside results from the glycosylation of quercetin on a glucose molecule. This compound has antioxidant properties and contributes to the color and stability of the final product, and is also associated with a chemical marker of matured beverages (De Simón et al., 2014).

3.3.2. Antioxidant activity of the sugarcane spirits

The evaluation of the antioxidant capacity of the samples is shown in Fig. 3, which illustrates the percentage inhibition of the ABTS^{•+} and DPPH[•] radicals and the FRAP potential of spirits subjected to the accelerated maturation process using chips. It is noteworthy that all the distillates that interacted with wood showed an increase in antioxidant activity compared to the control, regardless of the method of analysis, which can be explained by the fact that the antioxidant capacity of beverages is proportional to the content of phenolic compounds (Silvello et al., 2021).

The first use of the chips resulted in beverages with greater inhibition of the ABTS^{•+} and DPPH[•] radicals and a higher FRAP potential when compared to the samples obtained by the second use of the chips. In addition, the spirits exhibited different behaviors depending on the type of wood used.

Oak stood out for giving the final distillates a higher antioxidant activity than amburana and chestnut, with Q-1 showing 53 % and 42 % inhibition respectively for ABTS and DPPH and 70 % for FRAP reducing power. Previous studies have identified oak as a wood rich in antioxidant phenolic compounds (De Rosso et al., 2009; Jung et al., 2016), which explains the better performance of oak when compared to the other species. Amburana proved to be intermediate for antioxidant activity, being superior to chestnut for ABTS inhibition and Fe³⁺ reduction (FRPA). However, A-2 and B-2 were equivalent in terms of antioxidant capacity and did not differ for the three methods.

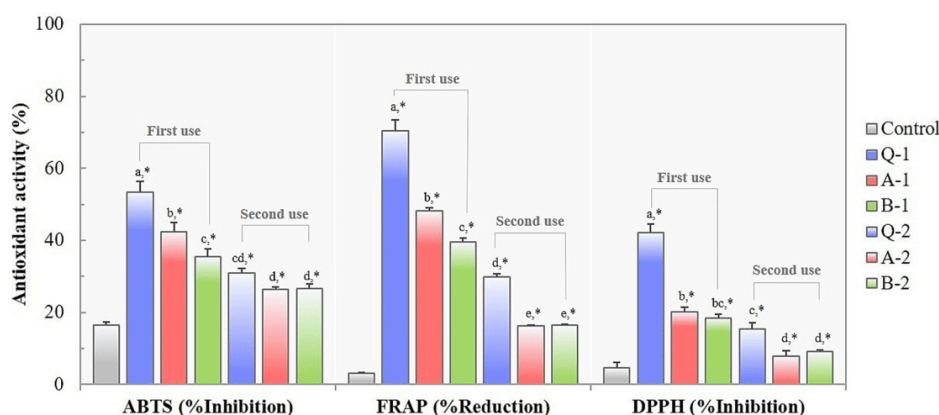


Fig. 3. Antioxidant activity of spirits matured with chips from different woods using the ABTS, FRAP and DPPH methods. Note (s): Different lowercase letters on the separate bars in each quadrant indicate significant differences between the aged distillates (Tukey's HSD, post-hoc test, $P < 0.05$). *Dunnett's post hoc ($p < 0.05$); an asterisk indicates a statistically significant difference from the control (non-aged spirit). Q-1: sugarcane spirit aged with *Quercus* sp.chips - first use. Q-2: sugarcane spirit aged with *Quercus* sp.chips - second use. A-1: sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use.

As for the principle of antioxidant action of the beverages obtained by interacting with chips, the first use of the woods provides distillates with better performance for reducing iron, since the FRAP method achieved better results in reducing power for Q-1 (70.37 %), A-1 (48.17 %) and B-1 (39.58 %) than the other methods. On the other hand, when reused, samples Q-2, A-2, and B-2 performed better for the ABTS method, with inhibition percentages of 30.98 %, 26.38 %, and 26.68 %, respectively. As for the DPPH method, only Q-1 showed satisfactory results, with an inhibition percentage of over 40 %.

In terms of DPPH radical inhibition, the results obtained for samples A-1 and B-1 are similar to the findings of Nie et al. (2023), with inhibition of less than 30 % up to 30 days of maturation at average chip concentrations of 5 g/L. However, the results for Q-1 were higher than those of the aforementioned study, demonstrating the greater antioxidant potential of oak.

The methods for determining antioxidant activity can be classified into two main groups: the first is based on the capture of free radicals, as in the ABTS and DPPH tests; and the second, on determining the oxidation of a target molecule, as in the FRAP assay (Kumara et al., 2018). The results obtained suggest that the different mechanisms that confer antioxidant capacity are linked to the type of phenolic compounds present in the samples. Thus, chemometric correlations (Fig. 4) can elucidate how these specific compounds are associated with antioxidant tests.

3.3.3. Principal component analysis

The main cluster analysis for the non-volatile characterization of the distillates (Fig. 4) explained 82.75 % of the variability of the data in the PC1 (60.61 %) and PC2 (22.14 %) dimensions, with PC1 being more representative. The Bi-plot shows that most of the variables show an increasing correlation with the positive side of PC1, except for the color parameters L^* , a^* , hue, and Catechin, which are on the negative side of PC1.

Spirits Q-1, B-1, and A-1 appear on the positive side of PC1, standing out for the presence of total phenolics, total tannins, and most of the

compounds identified in the phenolic profile, which correlate with greater color intensity, especially in the b^* (yellow color) and C^* (color saturation) parameters. This increase in color and the migration of phenolic compounds also correlated with greater antioxidant activity in the beverages. Spirits Q-2, A-2, and B-2 were on the negative side of PC1, close to the characteristics of the control, showing lighter colors, lower antioxidant activity, and lower amounts of phenolics.

The Bi-plot was effective in relating the profile of phenolics formed by the woods. Samples Q-1 and B-1 were grouped together due to the similarity of the extracted compounds, especially phenolic acids and lignin derivatives, which correlated with iron reduction (FRAP). Phenolic acids, such as gallic acid and p-coumaric acid, play a prominent role in the antioxidant effect of wood, as evidenced in previous studies (Smailagić et al., 2019; Smailagić et al., 2020). In isolation, sample A-1 showed singularity in the formation of phenolics, especially due to the presence of Epigallocatechin gallate, flavonones such as hesperitin, naringenin, and vanillic acid.

In the positive quadrant of PC1 and the positive quadrant of PC2, there was a positive correlation with total phenolics, total tannins, especially procyanidin B2, and the inhibition of ABTS and DPPH radicals, suggesting that the free radical inhibition mechanism is favored by chemical compounds with a higher electron-donating capacity, such as tannins.

In quantitative terms, the use of oak chips results in a distillate with higher phenolic content, followed by amburana and, more subtly, chestnut. However, qualitatively, the profile of phenolic compounds formed by the chestnut is similar to that of oak, especially flavonols and stilbenes, possibly conferring similar sensory characteristics. Although both have similar phenolic profiles, oak stands out due to its high antioxidant activity. This can be explained by two factors: 1) the amount of phenolics extracted from the chestnut was not sufficient to confer significant antioxidant potential; 2) oak-specific phenolics, such as procyanidin B1 and epigallocatechin, increase its ability to inhibit radical oxidation.

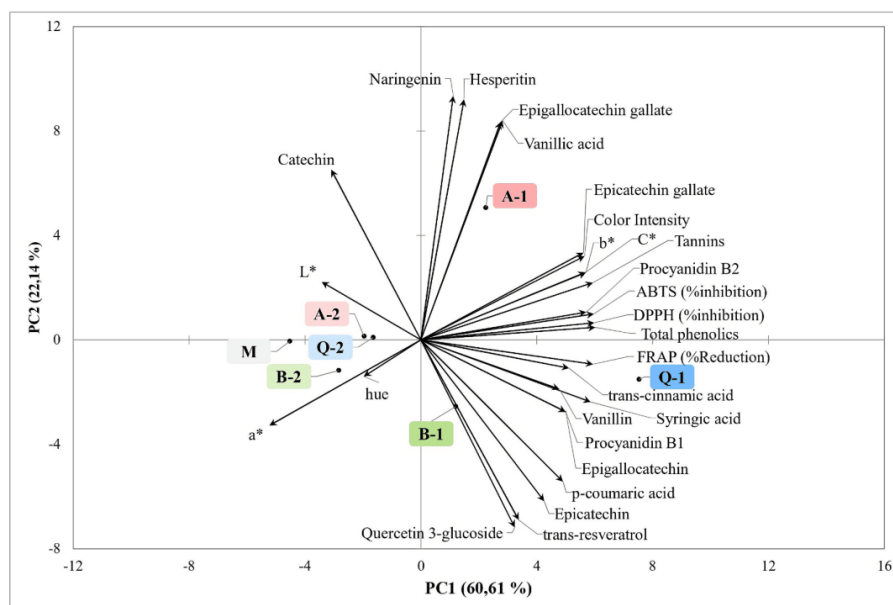


Fig. 4. Principal component analysis (Bi-plot) for non-volatile characterization, phenolic profile and antioxidant activity of sugarcane spirits aged with reused wood chips. Note (s): M: alcoholic matrix (non-aged spirits; control). Q-1: sugarcane spirit aged with *Quercus sp.* chips - first use. Q-2: sugarcane spirit aged with *Quercus sp.* chips - second use. A-1: sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use.

3.4. Volatile compounds profile

One hundred and twenty-two (122) volatile compounds were identified and classified according to their functional chemical characteristics (alcohols, acids, esters, aldehydes, ketones, terpenes, and others) (Table S2) (Fig. 5).

Fig. 5 (A) shows the Principal Component Analysis generated with all the volatile compounds obtained, with components 1 and 3 explaining 49.14 % and 9.01 % of the variability in the data set, respectively. Component 1 shows that there was a well-defined separation between the alcoholic matrix (M) and the beverages matured according to the number of uses of the wood, as there was no intersection between the ellipses representing the different groupings; however, the first use of the wood chips shows greater variability between the samples matured with different woods.

In this way, the distillate-wood interaction makes it possible to alter the profile of the volatile compounds present, but as far as the diversity of volatiles between the types of wood is concerned, only the first use of the chips makes it possible to form distillates with specific odor characteristics, while the reuse of these woods tends to present similar distillates in terms of aroma at the same interaction time, even though they come from different woods.

A breakdown of the volatile compounds in the distillates, according to the type of wood used and their respective uses, is shown in Fig. 6 (A, B, C, D). However, the main classes in terms of the number of compounds identified in the beverages (Fig. 5 B) include esters, terpenes, and alcohols, with the highest number of compounds obtained for the distillates from the first use of the woods. Although the total amount of compounds is similar between the samples, the differences stand out in terms of the intensity of each compound obtained.

The heat map showing the alcohol profile of spirits conditioned with wood (Fig. 6 A) shows 12 compounds, including short-chain alcohols, compounds with an aromatic ring, and long-chain aliphatic alcohols. Samples A-1 and Q-1 form a hierarchical cluster, as do samples A-2, B-2, and Q-2, while B-1 is intermediate between the clusters.

For short-chain alcohols, Isobutanol, Isoamyl alcohol, and the isomer 2-methyl-1-butanol stand out. These compounds were in higher concentrations in the second-use samples of the chips, coming from the control distillate. Their presence in distillates confers a fruity, banana, alcoholic, sweet, and pungent aroma (Portugal et al., 2016; Ribeiro-Filho et al., 2021). It is worth noting that Isoamyl alcohol is a precursor

to the formation of Isoamyl acetate, which is observed (Fig. 6. C) in the samples in a similar behavior to the precursor alcohol.

Among the alcohols that have an aromatic ring in their structure, Phenylethyl Alcohol is known to have a positive impact on the aroma of distillates, conferring sweet notes, such as floral, roses, and honey (Portugal et al., 2016). The use of amburana for maturation, especially in the first use, promotes a higher concentration of Phenylethyl Alcohol compared to oak and chestnut.

The other aromatic ring alcohols present were cerulignol and 4-ethyl guaiacol. These phenols share a similar structure and differ in their radical groups. B-1 has a higher concentration of cerulignol, while A-1 and Q-1 are more expressive for 4-ethyl guaiacol. In addition, samples A-2, B-2, and Q-2 only had concentrations of 4-ethyl guaiacol, which contributes to beverages with aromatic notes of smoke and spices (Petrozziello et al., 2014).

The other alcohols present (Fig. 6 A) - 2-heptanol, octanol, nonanol, 2-nonanol, decanol, and 1-Hexadecanol - are long-chain structures and were abundant in Q-1, A-1, and B-1, but they have less of an odoriferous impact on the distillates compared to the short-chain and aromatic ring alcohols.

Evaluating the profile of aldehydes and ketones (Fig. 6. B) shows two hierarchical groupings, with the samples from the first use of the chips being related by the greater intensity of the compounds, while those from the second use are grouped by the reduced concentration of these compounds. In the first and second uses, the oak and chestnut samples were similar in terms of the aldehydes and ketones formed.

Isopentanal was the only aldehyde present in greater intensity when the beverages were obtained by reusing the chips. This indicates that in reuse, the oxidation conditions of isobutanol (Fig. 6 B) favor the formation of aldehydes, a behavior that does not occur in first use, where the oxidative reaction of isobutanol favors the formation of esters, such as isobutyl decanoate (Fig. 6 C).

Among the first-use wood samples, Q-1 stands out for the presence of furfural, Carvomenthone, and Damascenone, which carries descriptors associated with herbal, floral, fruity, and honey aromas (Pino & Queris, 2011). A-1 is marked by Diethyl acetal, trans-2-decenal, Acetaldehyde, and Decaldehyde, while B-1 is expressive of Hexyl cinnamic aldehyde and Benzaldehyde. Acetaldehyde is described as contributing positively to the aroma of beverages (Bortoletto & Alcarde, 2013).

The long-chain aldehydes found in the samples of the first use of chips, such as Nonaldehyde and Decaldehyde, can add unpleasant

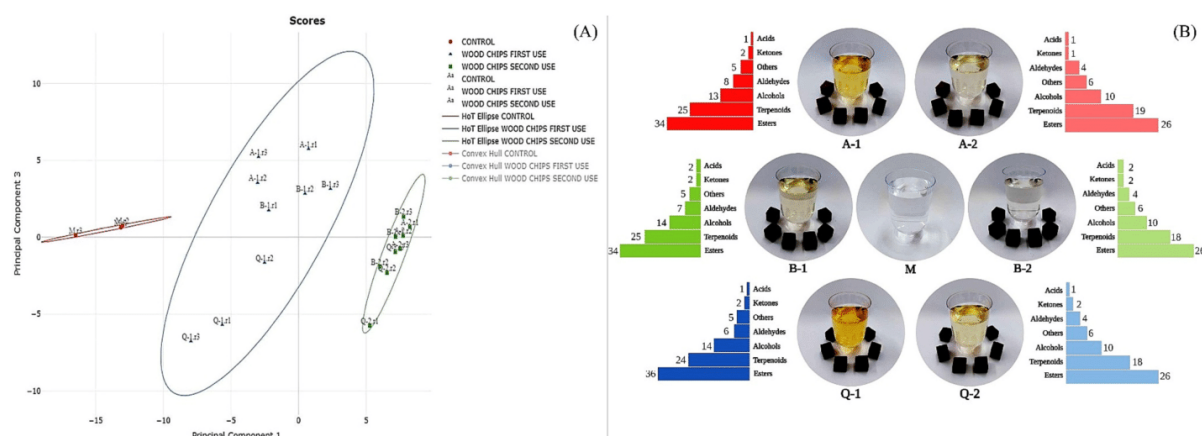


Fig. 5. Use of different wood chips in the volatile profile of sugarcane spirits: (A) Principal component analysis for the volatile compound profile (GC-MS); (B) Classification based on the volatile compounds obtained. Note (s): M: alcoholic matrix (non-aged spirits; control). A-1: sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use. Q-1: sugarcane spirit aged with *Quercus sp.* chips - first use. Q-2: sugarcane spirit aged with *Quercus sp.* chips - second use.

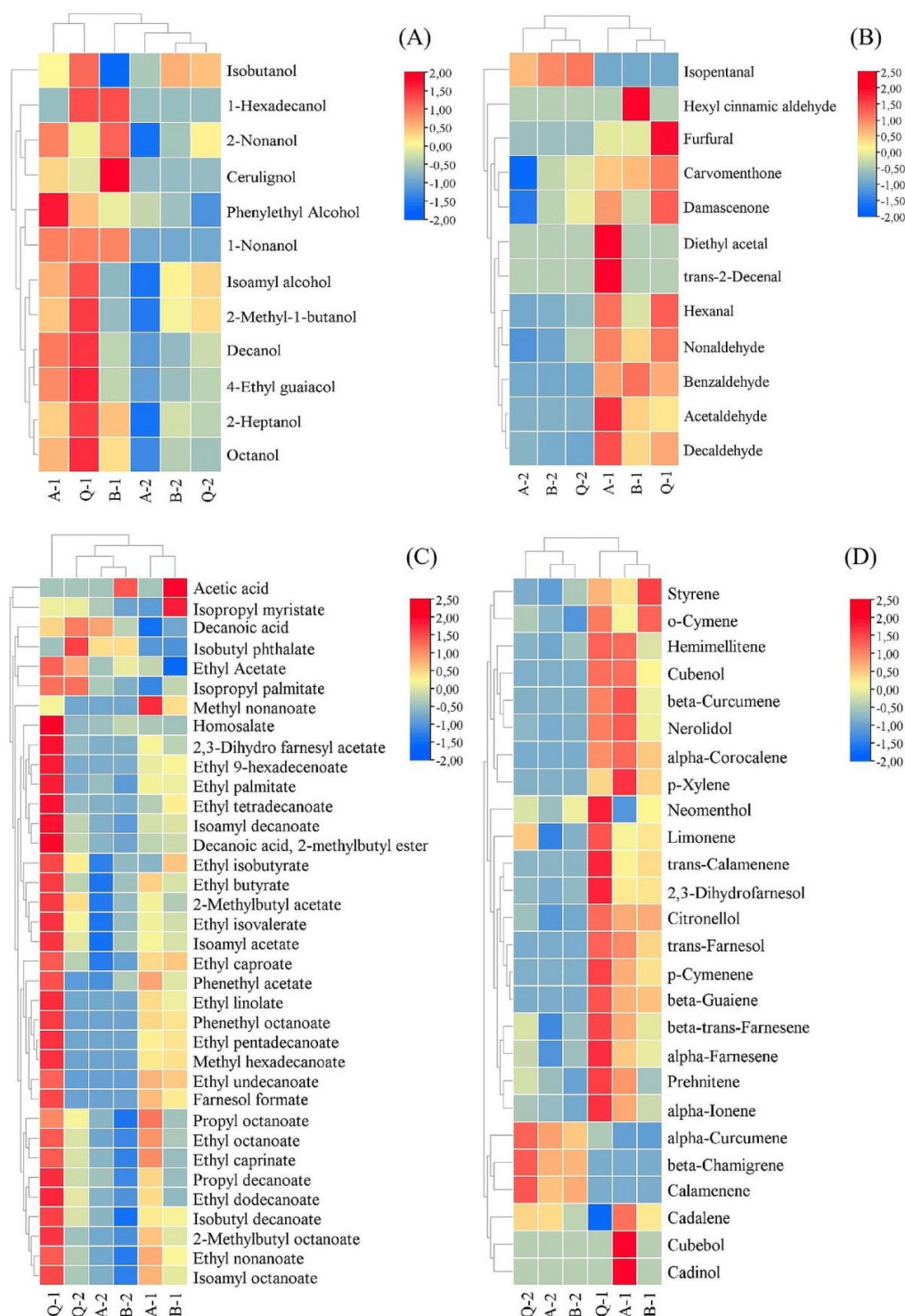


Fig. 6. Heatmap and hierarchical clusters for the profile of: (A) volatile alcohols; (B) volatile aldehydes and ketones; (C) volatile esters; (D) volatile terpenes. Note (s): A-1: sugarcane spirit aged with *Amburana cearensis* chips - first use. A-2: sugarcane spirit aged with *Amburana cearensis* chips - second use. B-1: sugarcane spirit aged with *Bertholletia excelsa* chips - first use. B-2: sugarcane spirit aged with *Bertholletia excelsa* chips - second use. Q-1: sugarcane spirit aged with *Quercus* sp.chips - first use. Q-2: sugarcane spirit aged with *Quercus* sp.chips - second use.

aromas, so the lower intensity of these compounds in spirits matured using the second use of chips may be positive sensorial. As for the aldehydes with an aromatic ring, Hexyl cinnamic aldehyde may have been derived from the oxidation of trans-cinnamic acid (Table 2), while Benzaldehyde is the result of the oxidation of Phenylethyl Alcohol (Fig. 6 A). Therefore, the low concentration of trans-cinnamic acid and Phenylethyl Alcohol for B-1 compared to the other woods in the first use of the chips can be understood by the formation of their derived aldehydes.

As for the profile of esters and acids (Fig. 6 C), we observed the formation of distinct clusters with Q-1 standing out on its own as the sample with the highest expression of esters, while the other clusters were formed depending on the number of times the chips were used. The complex interaction between the components of the wood and the constituents already present in the spirit can result in the formation of new esters or the intensification of compounds already present. The greater presence of esters in spirit Q-1 may be associated with factors related to the composition of the wood, the chemical reactions established between distillate and wood, and the maturation conditions.

Long-chain fatty acid esters - Ethyl tetradecanoate, Ethyl pentadecanoate, Methyl hexadecanoate, Ethyl 9-hexadecenoate, Ethyl palmitate, Isopropyl palmitate, Ethyl linolate, Ethyl Oleate - were abundant compounds in Q-1 and contribute to the flavor and viscosity of the beverage, giving body to the distillate. However, an excess of them can lead to negative perceptions because they are associated with fat (Bortoletto et al., 2018).

Along with spirit Q-1, A-1 also stood out in terms of the presence of esters with an aromatic ring, such as phenethyl acetate, Farnesol formate, 2,3-dihydro farnesyl acetate, and Phenethyl octanoate. These compounds contribute positively to the beverages, resulting in floral, woody, or balsamic aromas. However, all the samples from the second use of the chips showed Isobutyl phthalate as the aromatic ester, which may have come from the esterification of isobutanol (Fig. 6 A). Amburana also stands out for containing medium-chain esters such as Ethyl octanoate, Propyl octanoate, Ethyl nonanoate, and Ethyl capriate, which contribute to the beverage's oiliness, as well as highlighting fruity and floral aromas (Pino & Queris, 2011; Silva et al., 2020).

The chestnut added a greater amount of acetic acid to the distillates obtained (B-1 and B-2). This compound can be formed by the degradation of long-chain esters, and also by the degradation of the carboxylic group of phenolic acids, which would help explain the low intensity of total phenolic compounds in this wood since its degradation is favored by the formation of acids. The reuse of the chips also led to an increase in Decanoic acid, which can be explained by the degradation of esters with chains longer than 10 carbons, mainly Propyl decanoate, Ethyl undecanoate, and Ethyl dodecanoate.

Fig. 6 (D) shows the heat map for the terpene profile. The hierarchical cluster shows grouping according to the number of uses of the wood, with the first cluster comprising Q-2, A-2, and B-2 and the second grouping Q-1, A-1, and B-1. The first cluster is formed mainly by the significant presence of α -Curcumene, β -Chamigrene, and Calamenene in all the distillates matured with reused wood. This indicates that reuse alters the structure of previously migrated compounds, as is the case with the isomers β -Curcumene and trans-Calamenene, which were in greater quantities in distillates A-1, Q-1, and B-1, and converted to α -Curcumene and Calamenene in the second maturation.

Oak is the wood that stands out most from the others in terms of its terpene profile, both in the first and second uses, and has the most expressive compounds: Neomenthol, trans-Calamenene, 2,3-Dihydrofarnesol, p-cymenene, b-guaiene, b-trans-farnesene, a-farnesene, prehnitene, a-lonene. Limonene also appears in the first and second uses of oak. These compounds are associated with citrus (orange, lemon), herbal, raspberry, and cedarwood aromas (Petrozziello et al., 2020).

Amburana is recognized for providing characteristic aromatic profiles in beverages and the use of this wood has highlighted the compounds β -Curcumene, Nerolidol, α -Corocalene, p-Xylene, Cadelene,

Cubebol, and Cadinol. β -Curcumene is associated with woody aromas, while Nerolidol contributes floral and fruity aromas. α -Corocalene and p-Xylene add complexity, offering spicy and sweet scents. Cubebol and Cadinol, exclusive to amburana, are related to woody, spicy, and balsamic descriptors (Silva et al., 2020).

Chestnut was the wood that showed the least amount of expressive terpenes, with Styrene and o-Cymene standing out the most. Styrene is known for its slightly sweet and herbal aroma and is naturally occurring in plants. o-Cymene contributes citrus and spicy aromas (Purdy et al., 2021). This shows that the chestnut has a less complex aromatic profile.

4. Conclusions

The process of accelerated maturation using wood chips is a promising technological alternative for aging spirits. The first use of the chips makes it possible, in a short period of interaction, to obtain chemical (volatile and non-volatile) and physical characteristics (such as color) similar to those of aged beverages, without exceeding the legal limits for marketing. This allows producers with limited resources to improve the chemical quality of their beverages, making them competitive in demanding markets. Oak gave more expressive color, phenolics formed, antioxidant activity, and volatile compounds. Amburana showed specific chemical compounds, especially in the phenolic profile, making it the most distinctive among the woods. The chestnut was less intense in terms of the formation of volatiles and phenolics but was qualitatively similar to oak. The reuse of wood chips has a negative impact on the intensity of compounds that characterize aged beverages, limiting the process. Even so, the use of the chips resulted in spirits with a greater intensity and diversity of compounds associated with biological activity. This fact opens up space for new studies to explore the impacts of these beverages on the body, considering that many of these phenolics are already reported to have health benefits.

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CRediT authorship contribution statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodchem.2025.143163>.

Data availability

No data was used for the research described in the article.

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SUPPLEMENTARY DATA

Table S1. Kinetic behavior equations for the formation of phenolic compounds and color intensity in brandies matured with chips.

Wood Chips	N° of Use	Total Phenolic Content (mg EGA/100mL)*	R ²	Color Intensity (Abs 420 nm)*	R ²	Color intensity x Total phenolic Content (mg EGA/100mL)**	R ²
<i>Quercus sp.</i>	First use	$y = 2.8001\ln(x) + 2.4405$	0.9578	$y = 0.1603\ln(x) + 0.04$	0.9952	$y = 37.296x + 1.3629$	0.9994
	Second use	$y = 0.1233x + 1.5742$	0.9928	$y = 0.0214x + 0.0062$	0.9878	$y = 38.686x + 0.5966$	0.9819
<i>Amburana cearensis</i>	First use	$y = 1.4371\ln(x) + 2.5311$	0.9951	$y = 0.1177\ln(x) + 0.0474$	0.9912	$y = 25.161x + 1.6535$	0.9718
	Second use	$y = 0.0831x + 1.3761$	0.9932	$y = 0.0127x + 0.013$	0.9687	$y = 43.542x + 0.332$	0.9819
<i>Bertholletia excelsa</i>	First use	$y = 1.1236\ln(x) + 1.4185$	0.9716	$y = 0.0427\ln(x) + 0.014$	0.9926	$y = 54.93x + 0.8702$	0.9702
	Second use	$y = 0.0831x + 1.3761$	0.9932	$y = 0.0059x + 0.0034$	0.9745	$y = 65.167x + 0.7544$	0.9428

* Independent variable (x) is the interaction time in days; **Correlation equation between the variables TPC and Color intensity, with the dependent variable (x) being the absorbance measured at 420 nm.

Table S2. Volatile Profile of sugar cane spirits aged for 28 days with chips reused from different woods.

N.	CAS	Compound	Concentration (µg/L)						
			M	A-1	B-1	Q-1	A-2	B-2	Q-2
Alcohols									
1	78-83-1	Isobutanol	31009.7 ± 12880.1	101227.3 ± 38676.6	37252.4 ± 2263.3	138150.4 ± 18055.0	79859.7 ± 3310.9	119741.0 ± 29488.8	115504.1 ± 23282.8
2	123-51-3	Isoamyl alcohol	704330.9 ± 7647.0	852586.6 ± 151614.8	681490.0 ± 58278.3	939628.8 ± 147472.0	574072.9 ± 18642.2	791501.2 ± 130024.9	818749.8 ± 164911.9
3	137-32-6	2-Methyl-1-butanol	784644.8 ± 31408.1	328684.8 ± 69677.6	275919.0 ± 19166.0	375483.1 ± 52136.1	236967.2 ± 26899.8	307941.9 ± 46074.1	321411.4 ± 53546.9
4	71-41-0	1-Pentanol	251788.9 ± 12015.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
5	543-49-7	2-Heptanol	13401.2 ± 241.5	7943.3 ± 1436.9	8122.1 ± 2414.8	9980.9 ± 2391.4	4000.0 ± 869.8	6696.4 ± 1237.2	6411.6 ± 1635.0
6	111-70-6	1-Heptanol	2945.8 ± 1398.9	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
7	111-87-5	Octanol	19386.2 ± 3204.1	9336.6 ± 1217.3	8560.3 ± 1094.2	11798.1 ± 4580.9	4732.0 ± 1392.5	6837.3 ± 1147.1	6504.4 ± 517.3
8	628-99-9	2-Nonanol	12413.0 ± 975.7	7170.1 ± 636.1	7644.9 ± 3430.0	5088.8 ± 1523.7	1585.2 ± 541.5	3864.1 ± 2012.8	5377.9 ± 1056.3
9	60-12-8	Phenylethyl Alcohol	41666.1 ± 2735.2	22675.4 ± 9966.9	11216.1 ± 1508.7	14783.5 ± 2891.9	9450.9 ± 3457.9	7654.2 ± 3106.2	3875.1 ± 615.3

10	143-08-8	1-Nonanol	0.0 ± 0.0	34318.3 ± 9158.0	33542.5 ± 12860.7	33816.4 ± 8469.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
11	112-30-1	Decanol	31703.7 ± 1679.6	20938.7 ± 2658.3	12646.9 ± 1293.5	24000.7 ± 7287.8	8307.1 ± 1947.2	10739.9 ± 3064.5	13193.8 ± 1394.1
12	2785-89-9	4-Ethyl guaiacol	94650.6 ± 13320.4	56695.4 ± 9936.6	31865.2 ± 4858.5	71110.2 ± 15351.1	18879.5 ± 3872.4	26579.3 ± 8916.7	31347.4 ± 5742.8
13	2785-87-7	Cerulignol	0.0 ± 0.0	3933.9 ± 1989.1	9875.0 ± 1634.6	2066.6 ± 1049.1	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
14	112-53-8	1-Dodecanol	7095.1 ± 202.8	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
15	36653-82-4	1-Hexadecanol	4840.6 ± 2006.8	0.0 ± 0.0	5399.9 ± 529.2	5417.1 ± 817.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
<i>Acids</i>									
16	64-19-7	Acetic acid	0.0 ± 0.0	0.0 ± 0.0	31932.7 ± 1676.8	0.0 ± 0.0	0.0 ± 0.0	23271.9 ± 2768.6	0.0 ± 0.0
17	334-48-5	Decanoic acid	14892.1 ± 3462.1	5417.6 ± 453.5	12225.2 ± 2504.6	24827.3 ± 11285.0	28331.7 ± 2604.6	17535.2 ± 4172.9	31144.7 ± 13279.3
<i>Esters</i>									
18	141-78-6	Ethyl Acetate	46998.5 ± 28655.8	180481.8 ± 66183.9	66393.7 ± 8920.2	305450.6 ± 52653.1	161806.8 ± 5412.0	203932.8 ± 64691.7	260878.0 ± 62041.1
19	97-62-1	Ethyl isobutyrate	6053.6 ± 2574.2	3106.6 ± 1000.1	4069.4 ± 1608.4	4793.4 ± 591.3	2580.6 ± 216.6	3134.6 ± 833.8	3829.3 ± 967.1
20	105-54-4	Ethyl butyrate	6804.4 ± 1745.2	2426.0 ± 513.9	2113.3 ± 232.5	3029.5 ± 717.6	1365.9 ± 152.1	1882.6 ± 227.4	2012.0 ± 355.2

21	97-64-3	Ethyl lactate	24435.0 ± 1504.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
22	108-64-5	Ethyl isovalerate	9039.8 ± 4469.0	4212.8 ± 1004.5	3985.3 ± 713.1	5627.3 ± 1268.9	2769.4 ± 325.7	3589.2 ± 450.8	4247.2 ± 1058.1
23	123-92-2	Isoamyl acetate	40672.7 ± 20307.2	18022.0 ± 2949.3	17113.7 ± 3107.1	23707.3 ± 5104.6	11671.8 ± 1533.0	15726.0 ± 2263.5	17465.2 ± 4281.9
24	624-41-9	2-Methylbutyl acetate	6403.3 ± 96.9	4761.9 ± 819.9	4309.3 ± 765.3	6097.5 ± 1850.3	3365.2 ± 374.8	4117.9 ± 577.5	4978.9 ± 1138.2
25	123-66-0	Ethyl caproate	219642.0 ± 111484.6	133602.9 ± 24435.8	137516.7 ± 6320.6	162963.9 ± 35527.4	77823.3 ± 7734.2	91702.7 ± 12690.8	111161.9 ± 31131.0
26	10348-47-7	Pentanoic acid, 2-hydroxy-4-methyl-, ethyl ester	5034.2 ± 2112.1	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
27	106-30-9	Ethyl heptoate	12257.6 ± 549.7	7426.6 ± 766.6	8112.1 ± 2734.7	9550.6 ± 3035.9	1657.9 ± 269.7	3020.1 ± 1405.8	4311.6 ± 1012.8
28	123-25-1	Diethyl succinate	28592.5 ± 137.6	12266.8 ± 3166.0	11002.8 ± 355.3	14158.1 ± 4476.5	4755.6 ± 811.3	7583.4 ± 2529.6	7549.3 ± 1304.7
29	119-36-8	Methyl salicylate	1747.5 ± 582.5	3337.8 ± 1982.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
30	106-32-1	Ethyl octanoate	4057634.3 ± 1426741.0	2557716.0 ± 520775.2	1872655.2 ± 351289.7	2785348.8 ± 395683.6	1647078.1 ± 57446.3	1456530.6 ± 256694.2	2046827.8 ± 594465.1
31	1731-84-6	Methyl nonanoate	0.0 ± 0.0	6254.7 ± 3175.1	2976.3 ± 1002.9	2390.1 ± 97.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0

32	103-45-7	Phenethyl acetate	0.0 ± 0.0	10067.4 ± 1733.7	7743.5 ± 363.4	11844.9 ± 1376.6	4669.8 ± 852.7	6789.5 ± 2510.6	5018.0 ± 2091.4
33	624-13-5	Propyl octanoate	0.0 ± 0.0	5518.5 ± 1983.1	2695.7 ± 304.6	5282.2 ± 1996.1	2380.0 ± 534.8	1023.2 ± 344.1	3745.8 ± 1043.6
34	123-29-5	Ethyl nonanoate	130615.3 ± 1168.1	90530.9 ± 20150.0	75332.4 ± 32304.8	105556.1 ± 15718.1	49804.7 ± 786.9	35375.6 ± 9157.4	61598.1 ± 22106.3
35	-	Ethyl (4E)-4-decenoate	6197.6 ± 1000.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
36	110-38-3	Ethyl caprylate	8544096.3 ± 29612.1	7114875.3 ± 1363807.3	4836263.8 ± 863034.4	7660116.0 ± 1009590.8	4666641.2 ± 63577.2	3711194.6 ± 858111.8	5503372.0 ± 1549658.5
37	2035-99-6	Isoamyl octanoate	148489.6 ± 3369.9	102810.2 ± 16887.4	82355.5 ± 37047.3	127051.9 ± 21120.2	59434.7 ± 1373.3	41039.7 ± 11933.2	68976.9 ± 21756.3
38	67121-39-5	2-Methylbutyl octanoate	47856.8 ± 625.7	32738.7 ± 5226.9	26909.5 ± 11770.1	42925.0 ± 6515.0	19283.5 ± 96.9	16253.1 ± 4595.0	22428.5 ± 5944.0
39	30673-60-0	Propyl decanoate	15669.8 ± 288.1	6818.1 ± 1207.6	4503.8 ± 1251.0	9734.5 ± 792.4	4674.7 ± 681.4	2924.5 ± 475.7	5396.2 ± 1244.0
40	627-90-7	Ethyl undecanoate	59087.4 ± 1026.0	42805.0 ± 5952.9	38945.2 ± 17871.1	58719.3 ± 6406.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
41	30673-38-2	Isobutyl decanoate	76152.8 ± 4949.8	46266.3 ± 8248.1	43855.6 ± 18157.4	65540.0 ± 10319.1	31752.0 ± 4586.1	18279.7 ± 886.1	40455.9 ± 10901.4

42	106-33-2	Ethyl dodecanoate	5227196.9 ± 2292944.8	2768006.9 ± 523126.1	2176350.1 ± 226519.1	3554447.4 ± 348191.3	2122348.5 ± 94425.6	1889049.1 ± 457322.3	2530785.4 ± 560625.0
43	2306-91-4	Isoamyl decanoate	231309.1 ± 7070.2	130115.6 ± 23490.2	132745.4 ± 48423.2	217212.2 ± 22664.7	103803.6 ± 9372.1	92921.4 ± 26960.9	123558.5 ± 28787.5
44	68067-33-4	Decanoic acid, 2-methylbutyl ester	64119.5 ± 858.8	36842.4 ± 13337.1	37643.7 ± 10621.4	55833.2 ± 2264.7	33341.9 ± 2206.8	31836.0 ± 8449.6	37107.5 ± 7219.3
45	917105-98-7	Farnesol formate	19639.4 ± 1772.7	9134.4 ± 1260.8	6976.6 ± 58.9	14204.4 ± 3055.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
46	124-06-1	Ethyl tetradecanoate	148472.1 ± 2860.8	64683.0 ± 12898.1	86784.8 ± 42703.3	145774.4 ± 15340.4	51928.5 ± 11147.3	48960.9 ± 21032.0	57178.3 ± 16514.9
47	58130-58-8	2,3-Dihydro farnesyl acetate	20719.2 ± 1372.7	9971.2 ± 4276.5	7412.8 ± 391.4	20690.6 ± 5057.1	4685.3 ± 1006.2	4771.5 ± 1818.7	5628.3 ± 1516.7
48	110-27-0	Isopropyl myristate	8069.6 ± 5915.2	0.0 ± 0.0	10973.9 ± 1973.9	4137.2 ± 1562.1	2355.2 ± 96.4	475.5 ± 228.1	4069.9 ± 1007.3
49	4128-17-0	Farnesyl acetate, (E,E)-	40418.0 ± 20106.0	7426.2 ± 2205.6	9286.1 ± 4380.7	13827.7 ± 3285.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
50	6309-51-9	Isoamyl laurate	8592.9 ± 4072.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
51	5457-70-5	Phenethyl octanoate	14638.5 ± 880.6	6063.4 ± 2478.3	5714.8 ± 316.9	11501.4 ± 2039.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
52	84-69-5	Isobutyl phthalate	14033.7 ± 2323.9	8065.4 ± 1596.3	7627.0 ± 3048.1	11711.6 ± 3689.1	17770.4 ± 5531.2	17934.3 ± 596.3	25758.7 ± 3814.0

53	41114-00-5	Ethyl pentadecanoate	11429.1 ± 378.0	3937.1 ± 1524.3	4245.0 ± 116.2	8940.2 ± 1794.5	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
54	118-56-9	Homosalate	2446.2 ± 452.7	2407.9 ± 383.5	2293.7 ± 396.1	5991.0 ± 1901.2	2241.2 ± 43.5	2640.3 ± 387.9	2089.4 ± 1055.5
55	112-39-0	Methyl hexadecanoate	8774.4 ± 1559.6	2029.0 ± 945.6	2128.7 ± 392.4	4387.2 ± 1401.3	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
56	-	E-11-Hexadecenoic acid, ethyl ester	1384.0 ± 779.1	9684.5 ± 1172.0	6409.8 ± 602.7	15764.3 ± 1114.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
57	54546-22-4	Ethyl 9-hexadecenoate	6645.1 ± 160.2	1909.1 ± 321.4	2209.6 ± 181.6	6192.1 ± 1631.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
58	628-97-7	Ethyl palmitate	208530.6 ± 24250.8	90139.5 ± 35931.0	87362.3 ± 12448.0	180237.2 ± 18892.8	54908.5 ± 17270.6	31203.3 ± 5077.7	44700.5 ± 18506.7
59	142-91-6	Isopropyl palmitate	1065.4 ± 363.7	0.0 ± 0.0	2513.7 ± 1401.5	6067.8 ± 2966.5	2142.8 ± 841.5	1351.3 ± 810.1	6036.9 ± 1802.6
60	544-35-4	Ethyl linolate	13962.9 ± 3294.6	6564.2 ± 3065.9	4791.4 ± 1380.9	13484.7 ± 3550.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
61	111-62-6	Ethyl Oleate	4060.8 ± 328.4	0.0 ± 0.0	2756.3 ± 506.1	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
<i>Aldehydes</i>									
62	75-07-0	Acetaldehyde	0.0 ± 0.0	22462.1 ± 6886.3	11169.1 ± 2741.7	9716.2 ± 1716.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
63	590-86-3	Isopentanal	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	873.3 ± 155.3	1092.7 ± 140.7	1161.3 ± 350.2
64	105-57-7	Diethyl acetal	0.0 ± 0.0	7160.5 ± 997.8	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
65	66-25-1	Hexanal	2493.9 ± 860.8	1344.2 ± 81.4	828.8 ± 212.0	1400.9 ± 268.3	536.3 ± 68.5	564.4 ± 60.8	637.3 ± 121.8

77	527-84-4	o-Cymene	0.0 ± 0.0	1921.3 ± 458.0	2961.6 ± 355.5	2819.6 ± 982.4	1155.8 ± 107.0	812.7 ± 204.5	1435.8 ± 477.1
78	535-77-3	m-Cymene	5000.2 ± 2436.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
79	1461-27-4	Sylvestrene	2457.8 ± 218.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
80	138-86-3	Limonene	0.0 ± 0.0	2029.4 ± 334.0	2105.9 ± 929.5	2553.5 ± 811.7	1449.1 ± 223.9	1679.9 ± 267.1	2203.9 ± 684.1
81	13877-91-3	β -Ocimene	3252.4 ± 1453.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
82	1195-32-0	p-Cymenene	8909.1 ± 319.9	2287.7 ± 145.0	1708.4 ± 697.0	3560.2 ± 1186.8	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
83	488-23-3	Prehnitene	8048.7 ± 1602.3	3540.1 ± 1156.8	1533.6 ± 578.6	4473.8 ± 2230.9	1431.7 ± 265.7	904.2 ± 225.7	2017.2 ± 932.7
84	491-01-0	Neomenthol	0.0 ± 0.0	0.0 ± 0.0	1017.5 ± 215.3	2286.3 ± 1158.1	413.2 ± 168.6	912.7 ± 145.9	787.5 ± 219.7
85	33081-34-4	Lilac alcohol A	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	7343.6 ± 1096.2	0.0 ± 0.0	0.0 ± 0.0
86	106-22-9	Citronellol	26696.9 ± 1805.2	20879.5 ± 2979.0	20964.6 ± 8728.0	24020.7 ± 6161.6	9307.4 ± 1078.9	10694.1 ± 2897.1	12637.6 ± 1946.2
87	475-03-6	α -Ionene	0.0 ± 0.0	40735.0 ± 3263.9	19985.5 ± 8968.2	59734.3 ± 14667.5	11566.8 ± 1268.6	6827.9 ± 1839.0	14822.1 ± 6032.2
88	942-43-8	2,3-Dihydro-1,1,5,6-tetramethyl-1H-indene	0.0 ± 0.0	30724.0 ± 6113.6	19382.0 ± 9614.1	29849.1 ± 6613.3	47100.8 ± 5673.9	25040.4 ± 6349.0	52660.1 ± 20364.3
89	1078-04-2	1,1,4,7-Tetramethylindan	2593.9 ± 609.6	6190.4 ± 201.1	3302.0 ± 1081.4	9258.9 ± 2612.5	7133.5 ± 345.9	4455.5 ± 1716.4	9425.5 ± 4184.6

90	941-60-6	Indan, 1,1,4,6-tetramethyl-	7724.6 ± 1501.8	4657.3 ± 1471.6	2417.3 ± 776.1	4334.4 ± 1259.6	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
91	4506-36-9	1,2-Dihydro-1,5,8-trimethylnaphthalene	63550.5 ± 4339.2	48987.6 ± 21679.5	22087.4 ± 7424.2	42704.2 ± 10880.1	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
92	55682-80-9	1,2-Dihydro-1,4,6-trimethylnaphthalene	0.0 ± 0.0	72556.4 ± 15632.1	35007.3 ± 13216.5	80246.8 ± 13802.3	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
93	18794-84-8	β-trans-Farnesene	90264.3 ± 3146.6	81545.8 ± 11910.0	67874.4 ± 25638.7	96085.2 ± 12938.9	45291.2 ± 4314.1	57136.9 ± 16737.9	66059.6 ± 8878.5
94	18431-82-8	β-Chamigrene	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	8554.1 ± 2260.3	8277.4 ± 2722.9	12289.4 ± 2056.6
95	644-30-4	α-Curcumene	12460.5 ± 1124.5	6884.7 ± 2788.6	6737.7 ± 124.6	8852.6 ± 847.3	13417.0 ± 1726.5	12376.9 ± 3035.3	15077.7 ± 2079.9
96	88-84-6	β-Guaiene	0.0 ± 0.0	10510.8 ± 3193.1	9709.3 ± 3842.5	15625.0 ± 2773.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
97	26560-14-5	α-(Z,E)-Farnesene	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	26386.0 ± 3750.9	26184.9 ± 7462.1	35059.0 ± 6231.4
98	502-61-4	α-Farnesene	49351.0 ± 8.6	49904.7 ± 6153.5	43543.0 ± 16700.6	64412.3 ± 9470.8	29405.3 ± 5844.8	36272.8 ± 12347.8	40233.4 ± 4536.8
99	28976-67-2	β-Curcumene	695.3 ± 100.0	2778.7 ± 487.2	1029.2 ± 505.4	2346.0 ± 465.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
100	23445-02-5	Cubebol	0.0 ± 0.0	2074.1 ± 528.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
101	483-77-2	Calamenene	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	2654.6 ± 212.8	2985.9 ± 810.1	4180.3 ± 770.6

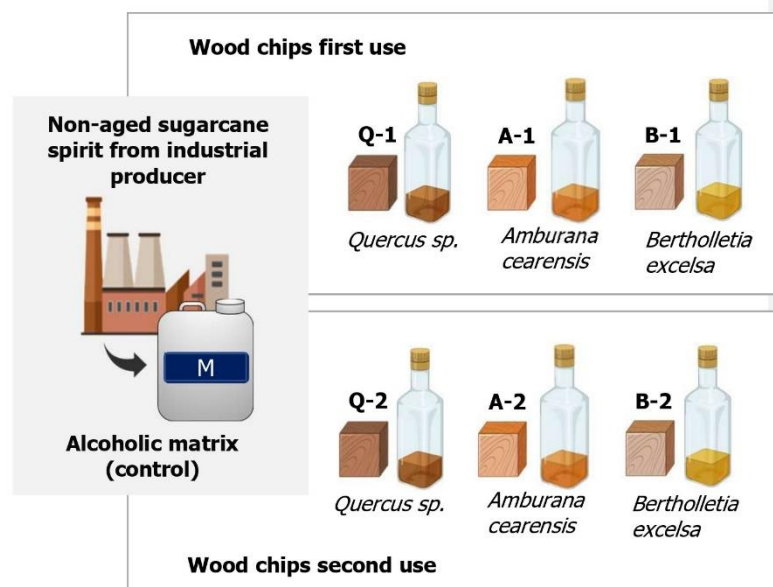
102	73209-42-4	trans-Calamenene	0.0 ± 0.0	2452.8 ± 1230.7	2921.5 ± 26.8	6591.3 ± 2493.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
103	829-26-5	2,3,6-Trimethylnaphthalene	56205.1 ± 996.4	51315.8 ± 23350.4	22087.4 ± 7623.9	37308.2 ± 9426.5	34202.7 ± 6146.7	11475.5 ± 501.8	30255.4 ± 12381.4
104	7212-44-4	Nerolidol	164125.7 ± 29150.0	161265.2 ± 65715.8	81407.0 ± 4951.5	144412.8 ± 18770.4	26226.7 ± 2323.0	30743.3 ± 10474.6	35665.3 ± 6803.9
105	2245-38-7	1,6,7-Trimethylnaphthalene	0.0 ± 0.0	0.0 ± 0.0	603.7 ± 98.4	1547.0 ± 397.3	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
106	124753-76-0	β -Copaen-4 α -ol	9052.3 ± 1876.8	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
107	20129-39-9	α -Corocalene	2674.2 ± 314.9	3326.8 ± 1645.8	2177.1 ± 995.8	2858.0 ± 1050.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
108	21284-22-0	Cubenol	0.0 ± 0.0	10453.6 ± 5216.9	5091.8 ± 828.7	10538.7 ± 625.5	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
109	19435-97-3	δ -Cadinol	0.0 ± 0.0	415.4 ± 61.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
110	483-78-3	Cadalene	0.0 ± 0.0	4061.7 ± 786.9	2799.0 ± 29.6	0.0 ± 0.0	2948.8 ± 521.1	1962.1 ± 401.5	3006.5 ± 1234.2
111	515-69-5	α -Bisabolol	3219.9 ± 382.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
112	526-73-8	Hemimellitene	0.0 ± 0.0	1564.4 ± 1029.7	918.3 ± 300.4	1597.5 ± 715.6	532.5 ± 208.5	699.7 ± 264.4	612.2 ± 126.4
113	106-42-3	p-Xylene	0.0 ± 0.0	2438.8 ± 1278.1	1226.5 ± 488.0	1202.6 ± 224.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
114	51411-24-6	2,3-Dihydrofarnesol	69130.7 ± 14804.3	44520.7 ± 10632.9	46022.7 ± 1545.1	85213.8 ± 18765.1	15473.9 ± 3198.2	19276.3 ± 9471.4	19901.8 ± 4371.3

115	106-28-5	trans-Farnesol	9608.7 ± 2889.2	7688.5 ± 1173.7	5305.0 ± 387.6	8932.9 ± 3002.8	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
<i>Others</i>									
116	6126-49-4	Tetrahydro-5-methyl-2-furanmethanol	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	3940.5 ± 707.6	5175.3 ± 953.7	6276.9 ± 1733.5
117	556-52-5	Glycidol	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	6217.5 ± 61.2	1269.3 ± 197.3	2160.5 ± 798.2
118									
119	637-69-4	p-Vinylanisole	40437.3 ± 89.7	20230.7 ± 1304.2	8438.7 ± 411.1	20398.6 ± 7150.5	6564.9 ± 1068.0	5336.2 ± 1615.4	7510.4 ± 1740.0
120	16204-57-2	1,1,4,5-Tetramethylindan	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	5628.4 ± 983.8	4047.2 ± 875.5	7670.2 ± 2783.5
121	629-82-3	Octyl ether	9234.4 ± 2537.7	8989.7 ± 4442.7	5204.0 ± 1078.0	12011.1 ± 2958.7	2250.9 ± 720.7	3089.8 ± 1287.8	3042.0 ± 1480.9
122	1222-05-5	Galaxolide	5384.2 ± 2000.2	3382.1 ± 323.2	4782.6 ± 2013.5	5474.9 ± 2520.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0

Note (s): M: alcoholic matrix (non-aged spirits; control). A-1: sugarcane spirit aged with Amburana cearensis chips - first use. A-2: sugarcane spirit aged with Amburana cearensis chips - second use. B-1: sugarcane spirit aged with Bertholletia excelsa chips - first use. B-2: sugarcane spirit aged with Bertholletia excelsa chips - second use. Q-1: sugarcane spirit aged with Quercus sp.chips - first use. Q-2: sugarcane spirit aged with Quercus sp.chips - second use.

Graphical Abstract

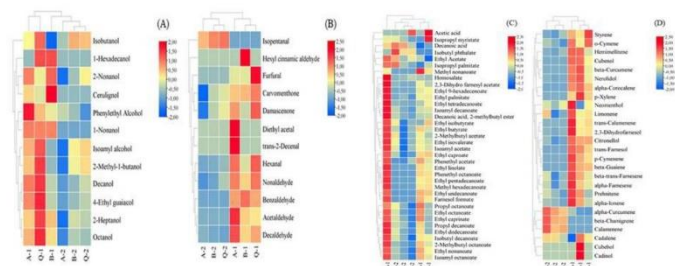
Effects of accelerated ageing of sugarcane spirit (28 days) using reusable wood chips



Distillates legally fit for consumption

PARAMETROS DE IDENTIDADE E QUALIDADE	Valor permitido
Densidade (g/mL)	-
Oxidação alcoólica % (V/V)	15
Ácidos voláteis (mg/100 mL de álcool anidro)	150
Ésteres totais (mg/100 mL de álcool anidro)	200
Aldeídos totais (mg/100 mL de álcool anidro)	30
Soma de furfural e Hidroximetilfurfural	5
Soma de álcoois isobutílicos (2-metil-1-propanol), isobutílicos (2-metil-1-butanol + 3-metil-1-butanol) e n-propílico (1-propanol)	300
Fenólicos totais (cachaça envelhecida)	-
Álcool metílico (mg/100 mL de álcool anidro)	20
Carbonato de sódio (μg/L)	210
Acetaldeído (2-propanol)	5
Álcool sec-butílico (2-butanol)	10
Álcool n-butílico (1-butanol)	3
Colete (mg/L)	5

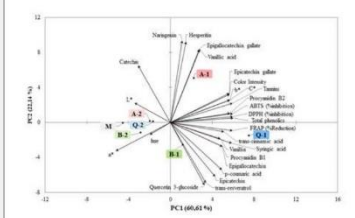
Aromatic profile varies between woods and number of uses



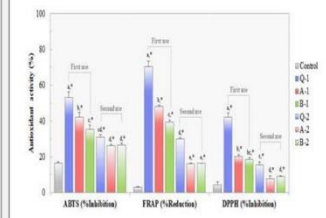
Color intensification



Obtaining phenolic compounds that are markers of ageing



Increase in bioactivity



6. CONSIDERAÇÕES FINAIS

O presente estudo investigou o impacto do uso e reutilização de chips de madeiras (carvalho, amburana e castanheira) no envelhecimento acelerado de aguardente de cana-de-açúcar, considerando os efeitos nas características químicas e bioativas das bebidas. Os resultados revelaram que o uso inicial dos chips promoveu maior extração de compostos fenólicos e voláteis, conferindo maior complexidade química às aguardentes.

Quanto ao perfil químico, o tipo de madeira que mais se destacou foi o carvalho, associado a um conteúdo fenólico superior e maior atividade antioxidante, enquanto a amburana e a castanheira apresentaram perfis únicos, contribuindo para a diversificação das características das bebidas.

A reutilização dos chips, por sua vez, apresentou uma limitação prática de uso, visto que resultou em redução significativa na concentração de compostos bioativos, fenólicos e voláteis. Esse efeito comprometeu a transferência de características químicas desejáveis para a bebida, aproximando as amostras da matriz alcoólica original.

Dessa forma, a técnica de utilização de chips demonstrou ser uma alternativa promissora para otimizar processos de envelhecimento, reduzindo custos devido a manutenção do teor alcoólico das bebidas e utilização de pequenas quantidades em peso do produto, assim como promoveu redução de tempo de maturação.

Além dos avanços obtidos, o estudo sinalizou lacunas que podem direcionar futuras pesquisas, destacando-se:

1. Abordagem por métodos sensoriais para descrever e caracterizar as bebidas, associando respostas de aceitabilidade de consumidores para bebidas produzidas por maturação acelerada.
2. Avaliação de outras espécies de madeiras tropicais brasileiras para identificar alternativas economicamente viáveis e com alto potencial químico e sensorial.
3. Desenvolvimento de métodos híbridos que combinem chips de madeira com extratos vegetais ou em combinações com barris para maximizar o desempenho dos constituintes vegetais durante a reutilização.
4. Investigações aprofundadas sobre os mecanismos de interação entre os constituintes químicos das madeiras e os componentes das bebidas, com foco na formação de compostos marcadores sensoriais e bioativos.

5. Uso de modelagem preditiva e simulações para otimizar as condições de envelhecimento e prever impactos sensoriais e químicos em diferentes pontos de maturação.

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PRINCIPAIS CONTRIBUIÇÕES ACADÊMICAS E SOCIAIS

Ao longo do período de realização do mestrado, diversas atividades acadêmicas foram realizadas, impactando significativamente a produção de conhecimento científico, a formação de novos pesquisadores e a interação entre academia, setor produtivo e sociedade. A seguir, são destacadas as principais contribuições acadêmicas e sociais desenvolvidas além do artigo científico publicado:

1. PUBLICAÇÕES E APRESENTAÇÕES DE TRABALHOS CIENTÍFICOS

1.1. APRESENTAÇÃO DE RESUMO NO 15º SLACAN

O trabalho intitulado "Influência de Diferentes Tipos de Madeiras no Perfil Volátil de Cachaça" foi apresentado durante o 15º Simpósio Latino-Americano de Ciência de Alimentos e Nutrição (SLACAN). Esse estudo teve como objetivo analisar o perfil volátil de cinco cachaças comerciais armazenadas por um ano em barris de diferentes madeiras (Freijó, Umburana, Carvalho, Bálsamo e Jequitibá), contribuindo para a compreensão do impacto da madeira no aroma da bebida. O resumo foi publicado nos anais do evento e pode ser acessado pelo link: [SLACAN 2023](#).

1.2. DESENVOLVIMENTO DE PROJETO DE INICIAÇÃO CIENTÍFICA

Foram desenvolvidos projetos de Iniciação Científica, gerando novos dados para o campo da ciência de alimentos e contribuindo para a formação de novos pesquisadores. Um dos trabalhos apresentados resultou no resumo "Maturação Acelerada de Cachaça com Diferentes 'Chips' de Madeiras e seu Efeito na Formação dos Marcadores de Envelhecimento", exposto no IX Congresso de Inovação em Tecnologia Agroalimentar (IX CITAG). O trabalho elucidou o processo de envelhecimento acelerado com diferentes tipos de madeira, que não foram utilizadas na dissertação, complementando os achados da presente pesquisa.

2. INTERCÂMBIO ACADÊMICO – MESTRADO SANDUÍCHE – COM DESENVOLVIMENTO DE MATERIAL EDUCATIVO

Foi realizado um mestrado sanduíche na Universidad de Extremadura (Espanha) por meio do programa de mobilidade Paraíba Sem Fronteiras (Edital Nº 11/2024), financiado pela Fundação de Apoio à Pesquisa do Estado da Paraíba (FAPESQ-PB). Durante esse período, os estudos realizados na dissertação foram ampliados, incorporando uma abordagem sensorial inovadora que resultará em novas publicações científicas.

Como produto educacional para a FAPESQ-PB, foi desenvolvida uma cartilha de repasse rápido com informações direcionadas aos produtores sobre o processo de maturação com chips de madeira, junto a uma série de vídeos que estão descritos na seção seguinte. Esse material inclui as principais contribuições sensoriais dos produtos, com uma abordagem intercultural. O acesso aos produtos pode ser visualizado por meio do link: [Produto Educacional](#)

3. DIVULGAÇÃO CIENTÍFICA E POPULARIZAÇÃO DA CACHAÇA

3.1. DESENVOLVIMENTO DE MATERIAL AUDIOVISUAL

Com o objetivo de ampliar o acesso às informações científicas, foi produzida uma série de quatro vídeos educativos publicados no YouTube, abordando diferentes aspectos da cachaça:

- [O que é cachaça?](#) – Definição, processo produtivo e contexto de consumo.
- [Envelhecimento da cachaça](#) – Aspectos químicos e sensoriais das alterações na bebida.
- [Maturação com chips de madeira](#) – Efeito sobre os constituintes voláteis e não voláteis.
- [Análise sensorial](#) – Descrição sensorial e impacto emocional da cachaça.

3.2. APRESENTAÇÃO DE PESQUISAS EM EVENTOS E INTERAÇÃO COM A INDÚSTRIA

- Evento Brasil Cachaças: Estudos sobre o perfil aromático de cachaças paraibanas armazenadas em diferentes barris foram apresentados, promovendo a interação entre academia, produtores e consumidores.

- Evento "Conexão Cachaça – Universidade em Parceria com a Indústria": Foram apresentados resultados sobre identidade e qualidade da cachaça a produtores locais, com fornecimento de laudos técnicos de análise de 75 amostras (28 brancas e 47 armazenadas em barris de madeira). Também foram apresentadas as seguintes pesquisas: Perfil Aromático das Cachaças Paraibanas; Compostos Fenólicos e Atividade Antioxidante de Cachaças Maturadas

3.3. PARTICIPAÇÃO EM PODCAST

Foi concedida uma entrevista para um podcast sobre cachaça, abordando sua produção, contexto local e os resultados das pesquisas desenvolvidas. O episódio pode ser acessado em:

[Podcast sobre Cachaça](#)

4. IMPACTO SOCIAL DO PROJETO

As pesquisas e iniciativas desenvolvidas contribuem diretamente para a valorização e qualificação da cachaça, um produto de grande relevância econômica e cultural no Brasil. O fornecimento de dados científicos, a capacitação de produtores e a divulgação das informações para o público geral têm impacto direto na melhoria da qualidade da bebida e no fortalecimento da sua identidade no mercado.

A interação entre universidade e setor produtivo permite a implementação de práticas inovadoras, enquanto a produção de conteúdo educativo democratiza o acesso ao conhecimento, beneficiando tanto pesquisadores quanto consumidores. Dessa forma, o projeto não apenas gera avanços acadêmicos, mas também promove desenvolvimento econômico e cultural, consolidando a cachaça como um produto de alta qualidade e valor agregado.