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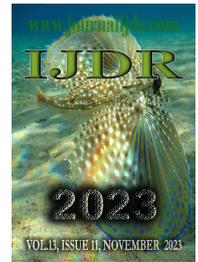
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RESEARCH ARTICLE

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WOOD CHIPS AND CACHAÇA AGING

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ABSTRACT

Recently, Brazilian legislation authorized the storage of cachaça in contact with wood fragments. However, the practice was admitted exclusively for producing an alternative cachaça, different from the aged one, and the differentiation must appear on the label, with words the same size as the brand of the drink. The objective of this work was to evaluate the differentiation between stored cachaças containing wood chips and cachaça aged in wooden barrels. Samples of oak chips were kept in contact with freshly distilled cachaça in glass containers for eight months. Next, chemical analyzes (phenolic content by liquid chromatography) and sensory evaluations were carried out. The results demonstrated that exclusive contact with chips makes it possible to reproduce the typical chromatographic profile of cachaças aged in oak barrels. However, it does not provide the typical reactions of aging, which respond to body and sensory complexity. The authors propose that, instead of creating a new type of cachaça, the chips should be classified as process aids, authorizing their use directly inside the wooden barrels intended for aging. This way, the handling of used barrels would be easier, color adjustment by adding caramel coloring would be unnecessary and producers would be encouraged to advance in the scope of standardization and sensorial excellence.

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INTRODUCTION

Wooden barrels and casks make up the classic setting of cellars intended for the maturation and/or aging of distilled beverages. In Brazil, cachaça producers use barrels made from various native woods, creating a diversity of tones and flavors that, in general understanding, enhance the identity and origin of the drink. In relevant proportions, they also use imported oak barrels that, in general, have already been used in the country of origin. Being barrels for second or third use, producers who acquire them deal with certain problems, both related to structural damage (generating leaks) and the marked evolution of volatile acidity (Maia & Campelo, 2006; Alcarde et al., 2014, Tark et al., 2023). In some cases, sulfur odors still occur, which come from the conditioning applied to already used and empty barrels for maritime transport purposes (Kleinbeck et al., 2011, Stadler & Fisher, 2020; Wanikawa & Sugimoto, 2022). Recently, Brazilian legislation (MAPA, 2022) opened an important possibility to be considered, authorizing the "conditioning of cachaça in contact with wood fragments, with the aim of giving it sensorial characteristics specific to wood". In several countries, this practice had been accepted since the 1960s and, in France, since the 19th century. In 1993, the United States regulated the use of wood fragments in winemaking; the same happened in the European Community, in 2006 (Singleton & Drapper, 1961, Niu et al., 2017; Petrozziello et al., 2021).

In Brazil, oak chips are already used in oenology, with the support of many studies on their preparation, application and sensory effects in fermented drinks (Barros et al., 2019; Návojská et al., 2012, Hamm et al., 2014; Jordão et al., 2016; Vargas, 2017; Tarancón et al., 2018, Dumitriu et al., 2019). There are fewer publications regarding the possible use of chips in distilled beverages (Otsuka & Imai, 1964; Bortoletto & Alcarde, 2015; Gurung, 2019; Krüger et al., 2022). Hundreds of producers who work in small scale and in an agricultural environment (referred to as still cachaça) have made efforts to improve the sensorial excellence of their cachaça. There is a growing movement towards insertion/consolidation of the drink in the international market. In this context, they question whether the authorization for the use of wood chips is intended exclusively for the production of a drink other than cachaça matured and/or aged in oak barrels. In fact, the new regulation establish that:

- The use of wood fragments requires the manufacturer to declare on the front panel of the label in a clear and conspicuous manner, with characters of minimum dimensions equal to those used in the product name, the expression: packaged with (description of the type of wood fragment) from (name of wood).
- The label cannot contain any expression that directly or indirectly associates the product with the aging process or the classification of aged.

The above reservations lead to the understanding that the use of wood chips would be an option applicable exclusively to lower quality and

low-cost cachaças. Furthermore, by describing minimum requirements for the preparation of “fragments”, assuming that they are used raw or roasted, the new regulations lead to the interpretation that producers themselves are able to prepare the wooden fragments to be introduced into the cachaça – the which is not true (Fan *et al.*, 2006, Kanakaki *et al.*, 2015, Petrozziello *et al.*, 2021). In this sense, the English name of origin “chip” seems more appropriate, referring more directly to associations with products obtained from standardized procedures, certifiable and traceable criteria. In fact, there are already chips of various sizes and shapes on the Brazilian market, indicating the origin of the wood and the roasting intensity (low, medium or high). Soon after the publication of the new ordinance (MAPA, 2022), it was decided to carry out this work, in order to contribute to the understanding of the potential of using wood chips in the standardization and sensorial quality of still cachaça.

MATERIAL AND METHODS

Oak chips: oak chips were tested from a manufacturer in Chile and another from the United States, samples made available by the respective sales representatives in Brazil. All of them had the information “medium toast” on the packaging. One of them, referred to as flakes (C.1) had smaller and more irregular dimensions, with a thickness of around 2 mm; the others were mini staves, with approximate dimensions of 1.5 cm x 1.0 cm x 0.5 cm (Figure 1).

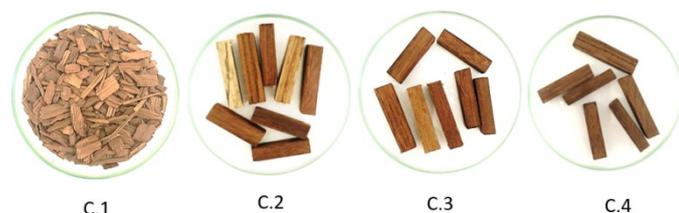


Figure 1. Illustrative photos of the chips used: C.1 = chips, C.2, C.3 and C.4 = mini staves

The samples were weighed and placed in seven clear glass jars, each containing 2 L of the same white cachaça (Table 1).

Table 1. Conditions used in chip testing

Coding		Test conditions			
Supplier	Chip	Test	Chip content(g/L)	T _{init.} cachaça (°C)	Note.
I	C.1	A	2,0 ⁽¹⁾	22 ⁽²⁾	Aeration for 30' after mixing
		B		35 ⁽³⁾	
		C		45 ⁽³⁾	
	C.2	D	2,5 ⁽¹⁾	22 ⁽²⁾	-
		E	5,3		
II	C.3	F	2,0 ⁽¹⁾		
	C.4	G			

⁽¹⁾ As recommended by suppliers ⁽²⁾ Room temp. ⁽³⁾ Adjusted at the time of mixing (4)

White cachaça, distilled at the end of the 2022 harvest and with an alcohol content of 40% vol. (20°C), was made available by a local producer. It was chosen based on sensory evaluation (smooth, slightly alcoholic, without defects) and proof of compliance with the parameters of current legislation (MAPA, 2022). Aeration in pots A, B and C during the initial 30 minutes after mixing was done using a mini aquarium aerator. All jars were covered and kept on a shelf, in a cool environment and protected from light. After eight months, the pots were opened, the chips were removed and each sample was divided into two aliquots, one for chromatographic analysis (HPLC) and the other for sensory evaluation.

HPLC reagents: Glacial acetic acid (Neon), 70% methanol HPLC standard (Supelco), Sigma-Aldrich chromatographic standards: gallic acid, vanillic acid, vanillin, syringic acid, syringaldehyde, sinapaldehyde, coniferaldehyde and ellagic acid. Analysis of phenolic

compounds: adjusted and previously described methodology (Maia *et al.*, 2022), using a Shimadzu SPD-10A chromatograph with UV detector (readings at 274 and 375 nm). Most of the phenolics analyzed showed maximum absorbance and were quantified at 274 nm. For sinapaldehyde and coniferaldehyde, quantification took place at 375 nm.

Sensory evaluation: Five experienced tasters carried out a blind tasting of ten samples, seven of which corresponded to cachaças stored with chips (Tests A to G, Table 1) and three were cachaças aged in oak and recently awarded in a national competition. Each taster completed a structured evaluation form in which they were asked to provide descriptive information regarding the appearance in the glass (oiliness, color, shine and clarity), olfactory evaluation (balance, aromatic associations and presence or absence of characteristics originating from the wood) and taste evaluation. (balance between body, alcohol and acidity, maturity, aromatic complexity and possible flavor associations due to the retronasal odor). At the end of each evaluation, the tasters classified the samples with numerical values, on a scale from 1 (very poor) to 5 (excellent).

RESULTS AND DISCUSSION

Phenolic content: Figure 2 shows results corresponding to samples A, B and C – all containing chip C.1 (2.0 g/L) and subjected to aeration in the first 30 minutes, varying only the initial temperature of the cachaça introduced into the pot. The chromatograms correspond to the analysis at 274 nm, but the quantification of peaks 7 and 8 occurred at 375 nm.

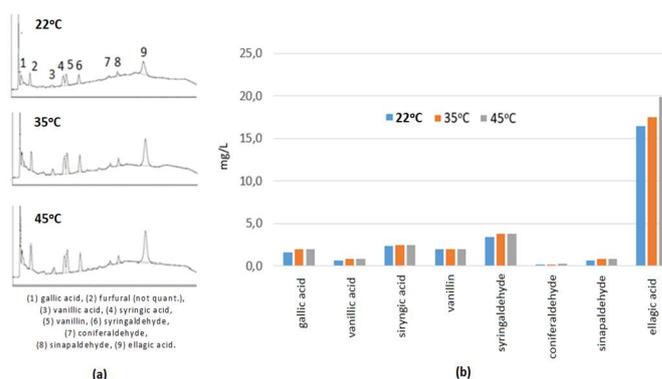


Figure 1. Test results with chips C.1 (22°C, 35°C and 45°C) (a) Chromatographic profiles, (b) Phenolic contents

It is noted that the chromatographic profiles were very similar (Figure 1-a), with a suggestion of a slight increase in phenolic content in cachaças pre-heated at 35°C and 45°C. In fact, the tone developed in the first 30 minutes of contact was slightly different; after six months, however, they were practically identical (see section 3.3). The total phenolic contents were 27.2 mg/L, 29.6 mg/L and 32.0 mg/L for pots A, B and C, respectively. The data referring to chip C.2 (pots D and E) can be found in Figure 2. Due to the masses of chips applied (2.5 g/L and 5.3 g/L), it was expected that the levels of each phenolic would maintain a ratio of around 0.47. However, there were marked divergences, especially in the case of gallic (0.24) and ellagic (0.33) acids. Investigating the possible origin of the deviation, it was noted that, unlike the others, the C.2 chips had been made available individually inside small envelopes, of which a small fraction had been opened. When opening the other envelopes, marked differences were noticed in the tone and porosity of each mini stove, allowing us to infer that each one came from different fractions of the wood, such as heartwood or sapwood (Louzada *et al.*, 2009) or even from different production batches. Having used a small number of mini staves in each pot, possible differences in the origin of the chips determined the divergences observed. Total phenolics were 17.0 mg/L and 67.4 mg/L (pots D and E, respectively).

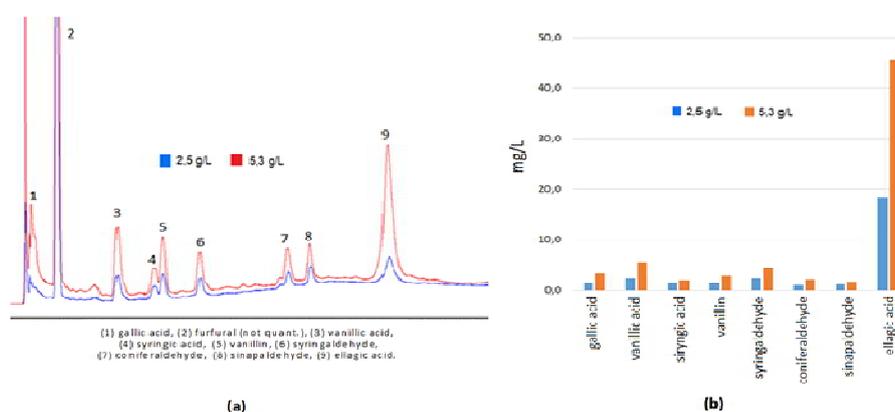


Figure 2. Test results with C.2 chips (2.5 g/L and 5.3 g/L) (a) Chromatographic profiles, (b) Phenolic contents

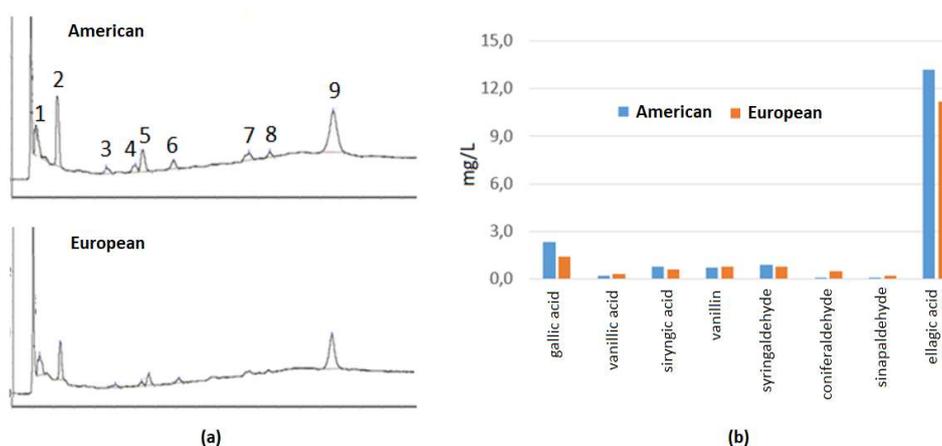


Figure 3. Test results with C.3 (American) and C.4 (European) chips (a) Chromatographic profiles, (b) Phenolic contents

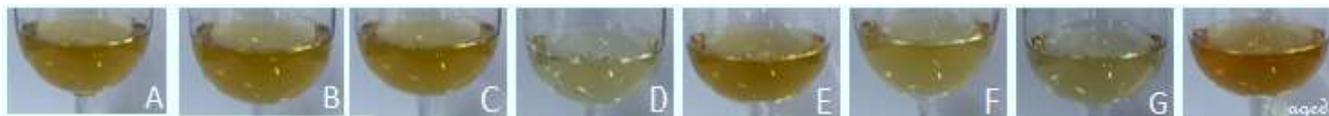


Figure 4. Photos of cachaça stored in contact with chips (A to G) and of a commercial cachaça aged in oak and awarded in a national blind tasting competition

In Figure 3 are the chromatographic profiles of samples subjected to the same standard treatment, both coming from the same supplier, differentiated according to the oak variety, according to the information contained on the packaging: one corresponding to American oak (*Quercus alba*) and the other to European oak (*Q. petraea*). Being differentiated in terms of oak variety and geographical origin, it would be natural to find greater variation between the levels of each phenolic. However, the chromatograms were very similar, even allowing us to assume that they actually belonged to the same batch. The total phenolic contents were 36.7 mg/L and 32.0 mg/L, that is, they were in the same order of magnitude as the pots stored with C.1 chips. In previous work, Maia *et al.* (2022) analyzed the phenolic contents in 17 samples of distillates aged in oak, including cachaças and whiskeys. The results showed, in all cases, the predominance of ellagic acid (7.9 to 71.1 mg/L), followed by gallic acid (2.4 to 30.6 mg/L) and syringaldehyde (1.4 to 27.2 mg/L). Total phenolics ranged between 20.6 and 132.1 mg/L. Therefore, it can be concluded that, after eight months of storage, all chips provided the chromatographic profiles and phenolic levels consistent with oak wood of the beverage storage barrels.

Sensory tests: All tests with chips (samples A to G) resulted in clear and shiny cachaças (Figure 4), having developed the typical oiliness on the internal surface of the glasses. The three samples added with C.1 chips, all at a rate of 2 g/L (A, B, C) provided a much more pronounced tone than that of C.2 chips, when tested at a content of 2.5 g/L (D) and similar to C.2 chips (E) when tested at 5.3 g/L. The samples corresponding to the C.3 and C.4 chips (F,G) developed a

lower tone than the C.1 chips and differed from each other, with the American chips (F) being slightly more accentuated than the French chips (G). In comparison with cachaça aged in oak barrels (aged), C.1 chips corresponded to the most similar tone. It is worth noting that, among 17 samples of cachaça and whiskey previously analyzed, the tone was more uniform, although the phenolic content varied over a wider range (Maia *et al.*, 2022). This observation suggests the practice of adjusting the tone through the addition of caramel coloring which, in our opinion, should be discouraged: the phenolics in oak (as well as in several other woods used in cooperage) are agents widely valued for their nutraceutical properties (Caleja *et al.*, 2017; Rio *et al.*, 2013; Soto-Vaca *et al.*, 2012). In terms of odor, they were all considered smooth and pleasant, devoid of defects, but with little association with oak. In terms of flavor (including retronasal odor), they were evaluated as lacking body and maturity. On a scale of 1 to 5, scores ranged between 3.2 and 3.6. The highest score was given to sample E (with 5.3 g/L of chips), due to the odor most associated with oak; however, the flavor was evaluated as slightly peppery and spicy, which may be associated with the high ellagic acid content. The three samples of cachaças aged in oak, tasted blindly and interspersed with the others, were described as harmonious, odor clearly reminiscent of oak, velvety palatability, with buttery touches, slightly smoked, floral, fruity (banana, plum, coconut), in addition to associations with vanilla, chestnut, chocolate and honey, varying depending on the brand evaluated. Thus, it became clear that, despite the delicate evolution of odor and flavor, the cachaça samples stored exclusively with chips proved to be devoid of sensorial complexity and could be referred to as “wood extracts”. These results corroborate the

consolidated understanding that there is a distinction between the reduced number of structures (predominantly phenolic) that are extracted from wood and the diversity of structures that characterize sensory evolution during aging (Conner *et al.*, 2023, Liu *et al.*, 2014, Zhang *et al.*, 2013, Kelly *et al.*, 2023, Kew *et al.*, 2016., Namara *et al.*, 2001). Hundreds of compounds are formed from interactions of ethanol with secondary compounds arising from fermentation (aliphatic) and wood (phenolics), in addition to interactions of secondary compounds with each other. The levels are very low, requiring special pre-extraction techniques (liquid-liquid or solid-liquid) so that they can be quantified. Therefore, although they come from compounds that can be routinely analyzed in quality control, they have little effect on the results of these analyzes over time (Robles, 2018): there is no loss in the nutraceutical functions of the phenolics in each wood. In particular, they involve oxidation reactions of fermentation and wood aldehydes to their respective acids, followed by esterification between alcohols and acids (aliphatic and phenolic), generating hundreds of aging marker compounds. Esters are formed (aliphatic, phenolic and mixed) that incorporate most of the aroma's associations with flowers, fruits, herbs and spices – which are valued as references of maturity and nobility. In fact, corroborating this statement, it is worth noting that the presence of 0.001 micrograms/L (10^{-9} g/L) of a compound with a molecular weight of 300 g corresponds to 2×10^{12} molecules/L – an order of magnitude compatible with certain mechanisms of our olfactory capacity (Zulfall & Zulfall, 2000). Finally, in addition to the low sensorial complexity, the evaluation described as “absence of body” in cachaça stored with chips also denounces the lack of the final stage of maturation, which is the formation of molecular aggregates (clusters) that give the drink the effect associated with viscosity, consistency and sensory balance: body (Ferreira *et al.*, 2006; Morishima *et al.*, 2018). Using light scattering (DLS) and X-ray (SAXS) methods, Morishima *et al.* (2018) assessed that the maturation and progressive nobility of whiskeys came from the progressive increase in the number of clusters for up to ten years or more of storage. Here, the term applies to specific three-dimensional arrangements involving water and ethanol molecules around phenolic acids. Clusters can have different conformations and sensory effects, depending on the degree of variety of phenolic structures and their contents (Nose & Hojo, 2006; Morishima *et al.*, 2018).

CONCLUSION

By inserting oak chips (in the range of 2.0 to 5.3 g/L) into white cachaça kept in glass jars, it was possible to detect the phenolic compounds typical of oak after eight months of storage, in proportions compatible to those found in cachaças aged in oak barrels. However, in the sensory evaluation, the cachaças stored with chips were considered to be devoid of body and sensory complexity. Therefore, the addition of chips to cachaça is not enough to make it equivalent to cachaça aged in wooden barrels. This finding leads us to question the wording of the recently approved law, which defines separately cachaça aged in wooden barrels and cachaça stored in contact with wood chips. It is known that, after the first use, there is a marked loss in the phenolic content extractable from the wood of the barrels. Third-use barrels practically do not color the cachaça, forcing the producer to dismantle each barrel in order to roast the staves again (a laborious practice that requires knowledge and expertise). The other option is to correct the shade of the drink by adding caramel coloring. Provided by legislation, this practice makes it possible to standardize the color of the final drink. But it does not correct the fact that the weakened color at the end of aging reveals a phenolic content below the standard of drinks aged in first-use barrels. In this context, instead of creating a lower quality cachaça (with chips added), it would be wiser to authorize the introduction of chips into traditional wooden barrels and/or barrels, as supporting elements in the process and without the need to highlight this practice on the labeling. This measure could enable refill of the barrels successively (without the need of dismantling for revitalization) by exchanging and increasing the chip content progressively at each beverage recharge in an estimated range between 0.5 g/L and 5 g/L. If a 300 L barrel has a

mass around 40 kg, the wood mass corresponds to about 133 grams per liter of cachaça stored. Therefore, even applied in the order of 5 g/L chips would still correspond to less than 5% of the barrel mass. But adding immense potential in terms of sensorial evolution and effective standardization, eliminating the need to adjust the color with caramel coloring and increasing safety and effectiveness in quality control. The next step, no less important, consists of establishing standards and criteria for the production and certification of wood chips, avoiding attempts at fraud and adulteration.

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