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Sensory profile and chemical quality of Merlot aged in barrels of different wood species

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Abstract

The cultivation of *Vitis vinifera* varieties in Brazil was established in 1970. With the opening of the market to imported labels and a greater variety of wines, viticulture became more competitive, and consumers became more demanding. The aging of wine is a system that involves several reactions based on the extraction of molecules from the wood and also on the micro-oxygenation of beverages. The objective of this study was to investigate the sensorial and chemical evolution of wines aged in barrels of different woods: French oak (*Quercus petraea*), Amburana (*Amburana cearensis*), and Jequitibá Rosa (*Cariniana legalis*). The comparison of the sensorial and chemical characteristics of the aged wine with the non-aged one allowed us to know the modifications resulting from the maturation time. Their distinct evolution among the different species was identified through chromatographic analysis of maturation congeners. Perceived sensory characteristics and acceptance of Brazilian woods were measured using the Napping and UltraFlash Profile methodologies. Tropical woods stood out in terms of acceptance by tasters, and the sensory attributes listed by the panel differ between both.

Keywords: tropical woods; napping; sensory analysis; aging congeners; liquid chromatography.

Practical application: The aging potential of tropical woods in wine is an underexplored and considerable subject for the beverage industry.

1 INTRODUCTION

Aging is widely used by the industry due to the sensorial improvement conferred on beverages subjected to this process (Mosedale, 1995). The maturation of the wines, as well as most spirits, takes place in wooden barrels, traditionally oak, where the wine undergoes important transformations and positive evolutions in its aromatic bouquet. These changes result from esterification, polymerization, hydrolysis, and oxidation reactions (Litchev, 1989; Onishi et al., 1977; Schwarz et al., 2011).

The potential of tropical woods to enrich the aromatic bouquet of distilled beverages, such as Cachaça, is already known. It is believed that these woods can also be an option to add positively to the sensory characteristics of red wine. In addition to this fact, they are also a less expensive and simpler alternative when compared with importing European Oaks. Studies related to the aging of wine in tropical wood are still rare when compared with other beverages, mainly distilled ones, thus making it an underexplored and promising subject for the beverage industry. The tropical woods *Amburana cearensis* (Amburana) and *Cariniana legalis* (Jequitibá Rosa) present unique characteristics alike oaks. Amburana adds to the distilled spirit characteristic aromas of phenolic compounds from the vanillin group, such as vanilla and other spices, in addition to an accentuated woody aroma. Jequitibá Rosa has less pronounced aromas. However, it contributes to the sweetness of the alcoholic beverage due to the aging markers vanillin and vanillic acid (Bortoletto & Alcarde, 2013).

The objective of this study was to analyze the chemical transformations resulting from the aging of wine in French Oak (*Quercus petraea*), Amburana (*A. cearensis*), and Jequitibá Rosa (*C. legalis*) woods. Alternatively, the sensory characteristics of the samples were analyzed using the Napping and UltraFlash Profile methodologies to understand the different treatments on the sensory complexity of the wines and their effects.

2 MATERIALS AND METHODS

The wine used in the project was made of Merlot grapes, 2019/2020 harvest, from Serra Gaúcha. The property where the grapes were grown is located under latitude and longitude -29.109006449710023 and -51.32990444961291, respectively, in the city of Flores da Cunha, Rio Grande do Sul, Brazil.

The vines were planted in the espalier system grape vines, allowing a better yield due to the increase in leaf area. After the harvest, the sugar content present in the grape was 22 °Brix; therefore, there was no need for chaptalization. The vinification took place from the separation of the stems and grinding, tumultuous fermentation using CRU-05 yeasts, separation of the skin, and clarification by decantation, with racking every 20 days for 5 months. After being finalized, the wine was delivered for research in 5-L glass bottles sealed with cork stoppers.

The first use barrels of French oak (*Q. petraea*), Amburana (*A. cearensis*), and Jequitibá Rosa (*C. legalis*) were purchased from Tanoaria Mesacaza, located in Monte Belo do Sul (Brazil).

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All barrels had a volume of 10 L and a light roast (approximately 100°C with a roasting duration of 10 min).

During preparation, the barrels were previously washed and filled with water for 24 h, to hydrate the wood and also to stop any leaks. Then, sanitization was carried out with the application of steam at 100°C for 15 min and 36 h in contact with peracetic acid at 0.5% v/v. Finally, the barrels were again filled with water for 24 h. The maturation site was maintained at an average temperature of 15°C and relative humidity between 70 and 80%, protected from vibrations.

The wine was distributed in barrels of approximately 10 L, two of Amburana, two of French Oak, and two of Jequitibá Rosa. Part of the wine was not aged and was stored horizontally in dark glass bottles sealed with stoppers (as well as the barrels), between 12 and 13°C, with relative humidity between 70% and 80%.

The samples were aged in barrels for 4 months and, after that period, the wine was bottled in glasses and sealed with cork stoppers. The aging room was maintained at an average temperature of 16°C and relative humidity at 60% and protected from vibrations, with the bottles in a horizontal position for 2 months before physical-chemical and sensorial analyses.

2.1 Chromatographic analysis

The wine samples were previously submitted to solid-phase extraction (SPE). Sample preparation was performed by diluting the wine in 0.1 mol L^{-1} HCl (3 mL of wine + 3 mL of HCl). Then, the packaging was carried out in a solid-phase cartridge with the aid of a manifold system, 5 mL P.A. through the extractor cartridge, and then 5 mL of ultrapure water. The sample passed drop by drop through the extraction cartridge, and then the extractor was washed with 5 mL of 0.1 mol L^{-1} HCl and then 5 mL of ultrapure water, completely drying the cartridge. The recovery of phenolic compounds was performed by passing 6 mL of ethanol through the extraction cartridge. The collected sample was evaporated and resuspended in 3 mL of 20% ethanol.

The liquid chromatography analysis with a diode array detector was performed by Shimadzu Nexera XR equipment, with Supelcosil LC-18 column (25 cm × 4 mm × 5 µm), oven temperature at 35°C, and a flow rate of 0.8 mL/minute. The run time was 40 min, and the mobile phase was composed of (A) 99% ultrapure water and 1% formic acid and (B) 99% HPLC grade methanol and 1% formic acid. The gradient schedule was: 6 min 100% phase A, 26 min 100% phase B, 5 min 100% phase B, 3 min 100% phase A, and, finally, 4 min 100% phase A (Rodriguez-Delgado et al., 2001). The aging markers such as gallic acid, vanillic acid, syringic acid, vanillin, p-coumaric acid, 4-vinyl guaiacol, and guaiacol had concentrations defined by retention time (min) and wavelength (nm). The standards used in the analysis were Sigma Aldrich and Merck mobile phase. The wavelengths used are shown in Table 1.

Table 1. Wavelengths used in liquid chromatography analysis with diode array detector.

Aging markers	Wavelengths (λ)
Gallic acid	280
Syringic acid	280
Vanillic acid	290
Vanillin	300
p-Coumaric acid	310
Guaiacol	250
4-Vinyl guaiacol	250

2.2 Sensory analysis

The methodology used was the projective mapping Napping (Pagès, 2005; Pagès et al., 2010) with the UltraFlash Profile descriptive method (Dairou & Sieffermann, 2002), which proved to be a faster alternative compared with quantitative descriptive analysis. Through the UltraFlash Profile, it is possible to obtain information on similarities and differences between the samples, in addition to their description. All statistical analyses were performed in the R program, with the Factor Mine R package (Josse et al., 2008).

The panel of tasters was made up of regular red wine connoisseurs and consumers. There were 52 participants with ages between 18 and 53 years. The analysis project was reviewed and approved by the Human Research Ethics Committee of ESALQ/USP (CAAE 46063921.4.0000.5395).

The tasters did not undergo prior training before the analysis, as the objective of the research involved the use of methodologies with consumers. Therefore, the presentation of references was necessary for a better description of the sensory profile of the samples. The presentation of flavors and aromas was based on a sensorial aroma wheel developed by Noble et al. (1987).

Amburana (*A. cearensis*) is considered a noble wood with medicinal properties. Regarding its sensory characteristics, it is very aromatic, and it promotes smoothness to distilled beverages, such as Cachaça, reducing acidity and the sensation of alcohol content. Regarding the flavor, it has notes of spices such as cloves, cinnamon, and pink pepper, in addition to a pronounced sweetness and woody aroma (Catão et al., 2011). Coumarin, a compound rich in Amburana wood, is responsible for the aroma and flavor of spices, especially vanilla.

The Jequitibá Rosa (*C. legalis*) is found in practically all regions of the Brazilian territory. It is usually used to age Cachaças and preserve their original aromas and flavors. However, it adds softness and balanced acidity (Ditchun, 2018). The accentuated presence of vanillin in this wood is responsible for adding vanilla notes to the drink.

According to Catão et al. (2011), although Oak is the predominantly used wood for the aging stage, the chemical composition and physical aspects of woods such as Amburana and Jequitibá Rosa make them legitimate tropical substitutes for Oak.

The grape variety *Vitis vinifera* Merlot is the second most produced in the region of "Serra Gaúcha," in the state of Rio Grande do Sul (Brazil), whose wines stand out for their fruity aroma with notes of red fruits (Rizzon & Miele, 2009).

Despite the asepsis adopted for cleaning the containers, during the months of maturation, some barrels showed excess acidity in the stored wines, resulting from the microorganism *Brettanomyces*, often found in the fermentation and aging of red wines (Wedral et al., 2010). These barrels were the two barrels made of French Oak, in addition to one of the Jequitibá Rosa barrels. The content of these barrels was not used in the sensory stage so that the judgment of the evaluators would not be influenced by the oxidation of the samples. The samples obtained for sensorial analysis from the duplicates of the drinks were Amburana (A1 and A2), Jequitibá Rosa (J1), and non-aged wine.

The three resulting treatments were presented to the tasters according to Williams' Latin-Square Design (Williams, 1949), Each evaluator received a single portion of 20 mL per sample in ISO 3591:1977 cups, which were coded with different numbers of three random digits. In total, four samples were presented: non-aged (451), jequitibá rosa (743), and duplicate samples of Amburana (268 and 896), in order to test consumer repeatability.

Napping (Pagès et al., 2010) was based on the spatial grouping of samples on a white A3 paper. The greater the similarity between the samples, the greater their proximity, and the greater the differences, the more distant they would be in the spatial plane. Then, the UltraFlash Profile (Dairou & Sieffermann, 2002) consisted of guiding the tasters to characterize the samples by assigning terms that encompassed the visual aspects, aroma, flavor, and aftertaste. At this stage, the tasters had the help of the table of attributes adapted from the sensory wheel of wine aromas (Noble et al., 1987), in addition to the olfactory and gustatory personal memory.

2.3 Data analysis

The RStudio IDE (R version 4.1.2) was used with the aid of the analysis libraries FactoMineR (Lê et al., 2008) and SensoMineR (Lê & Husson, 2008), native functions of hypothesis tests, and the graphic library ggplot.

4 RESULTS AND DISCUSSION

4.1 Chromatographic analysis

At the end of 4 months of aging, the chromatographic analysis allowed a detailed study of the chemical profile of the samples and their aging markers, also called maturation congeners, due to the interaction between the wine and the Amburana, Jequitibá, and French Oak woods. The main maturation congeners are free phenolic acids, such as gallic, vanillic, ellagic, ferulic, and syringic acids, as well as phenolic aldehydes of the hydroxybenzoic series: vanillin and syringaldehyde and hydroxycinnamic series: conifer aldehyde and synapaldehyde (Cernîsev, 2016; Chatonnet & Dubourdieu, 1998).

Gallic acid, which originated from hydrolyzable tannins in the wood, obtained higher concentrations in oak wood, with an average of 93.93 mg L^{-1}. Wine aged in Amburana had 77.47 mg L^{-1}, which is the second most pronounced concentration among the three treatments. Gallic acid does not directly participate in the aromatic bouquet of the drink, as it is not a volatile compound. However, it contributes to the increase in quality related to visual and texture attributes (Cernîsev, 2016).

According to Cerîncev (2016), the sum of vanillic and syringic acids can be considered a parameter to characterize the degree of aging of the beverage. In this research, the wines obtained total vanillic acid concentrations of 12.43 and 5.18 mg L^{-1} in Amburana and French Oak, respectively. In the Jequitibá species, the acid concentration was below the limit of quantification. Regarding the concentration of syringic acid, Oak stands out with a concentration greater than 12 mg L^{-1}, followed by Jequitibá Rosa wood (10.9 mg L^{-1}) and Amburana (9.56 mg L^{-1}).

In the presence of ethanol, both acids can be esterified, giving rise to the compounds ethyl syringate and ethyl vanillate. Such compounds are sensorially associated with "spices," "peppers," "vanilla," "almonds," "floral," and "cherry" (Cernîsev, 2016; Conner et al., 2003).

Among the analyzed woods, it can be observed that the species that presented the greatest chemical complexity about the maturation congeners was the French oak (*Q. petraea*). Amburana (*A. cearensis*) also stood out in the total sum of maturation congeners, having a profile more similar to that of oak. Jequitibá (*C. legalis*), despite presenting a smaller contribution compared with aging congeners, mainly related to vanillic acid, also proved to be complex in the other markers.

4.2 Sensory analysis

The partial individual chart (Figure 1) relates the samples to the types of attributes listed by the tasters. The difference between non-aged wine and those that have undergone aging in wood is clear.

Regarding non-aged wine, the aroma group stands out, compared with aftertaste and flavor. When analyzing the samples aged in Amburana, it is possible to highlight the very similar behavior of the tasters who, majorly, identified that it was the same wood. Finally, the sample aged in jequitibá had most of its description explained by the aftertaste, followed by the color group, these two groups being essential in the acceptance and differentiation of this wine (Figure 1).

Regarding the flavor group, we again observed different behaviors between aged and non-aged wines. The variety of flavors among the aged wines is evident, with emphasis on the "spice," "smoked," "woody," and "caramelized" flavors, especially in the Amburana samples. The "vanilla" flavor appears in transition between wines aged in Amburana and Jequitibá (Figure 2).

Regarding the aromas (Figure 3), there is also a segregation in the behavior of aged and non-aged samples. The non-aged wines stood out in terms of "red fruit" and "floral" aromas. The tasters noticed a good distinction between Amburana and Jequitibá woods, which obtained a more complex sensorial bouquet.

The aftertaste was more similar among all analyzed samples (Figure 4). However, the "floral" had a greater relation with Jequitibá. Again, Amburana samples showed proximity behavior, where the highlighted aftertastes were "smoked," "woody," "caramelized," "vanilla," and "spices."

Pearson correlation matrix plot demonstrates trend correlation between variables, ranging from -1 (red) to 1 (blue). Through this graph, it is clear that we understand the behavior of a variable as a function of another (Figure 5).

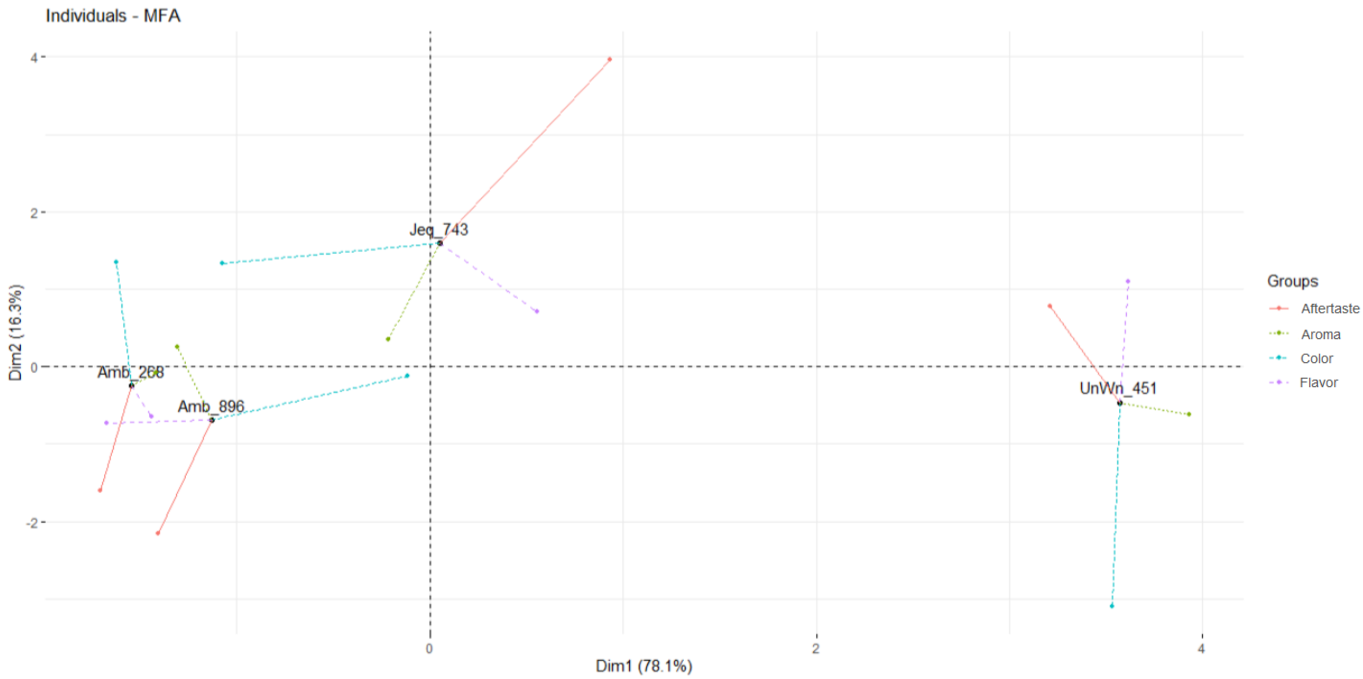

Figure 1. Representation of aged and non-aged wines in Dimensions 1 and 2 in relation to the types of attributes raised in the sensory analysis.

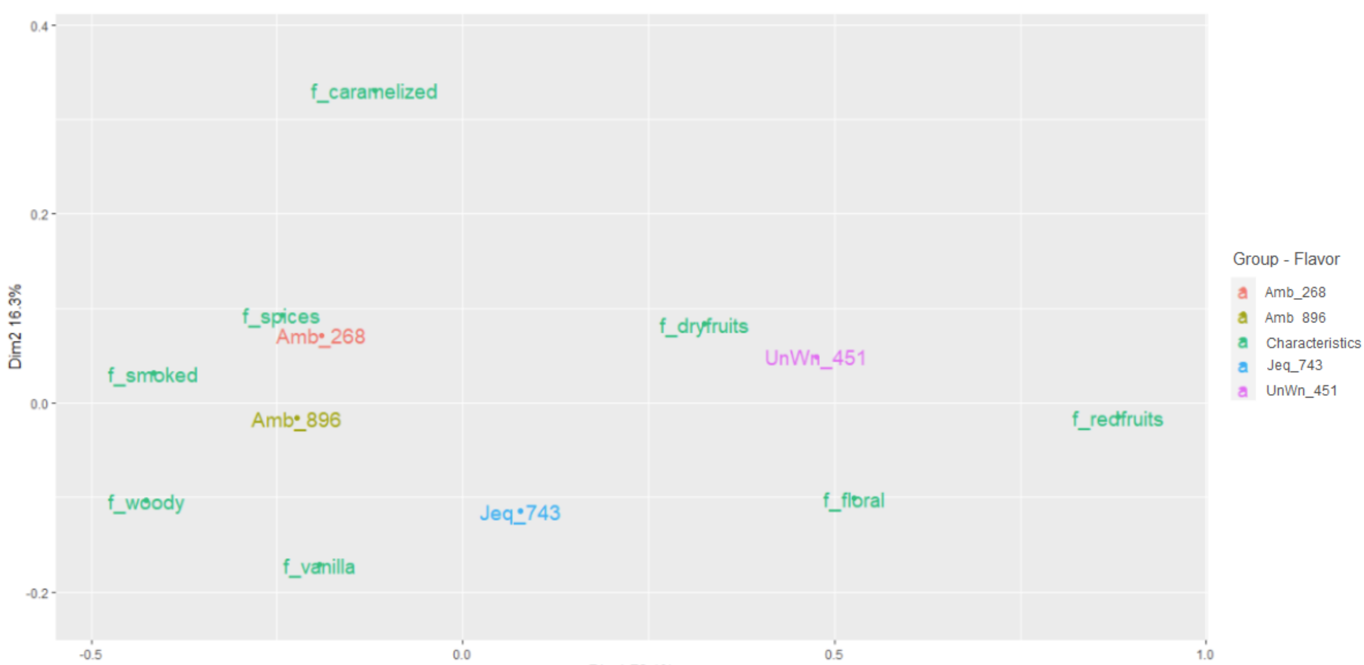

Figure 2. Representation of aged and non-aged wines in Dimensions 1 and 2 about the flavor attributes raised in the sensory analysis.

The blue regions correspond to positive correlations, for example, between woody aroma and vanilla flavor; therefore, there is a strong tendency to find both variables together in the same aged wine. The same occurs with the smoked aroma in samples that have a caramelized aftertaste and for samples with a pronounced aroma of red fruits and a straw purple color.

In turn, the red regions have negative correlations, that is, they are unlikely to be found together in the same wine. One can cite the aftertaste of red fruits that will hardly be perceived in wines that have woody aromas. The same occurs with the samples that present the flavor of red fruits and the aroma of spices.

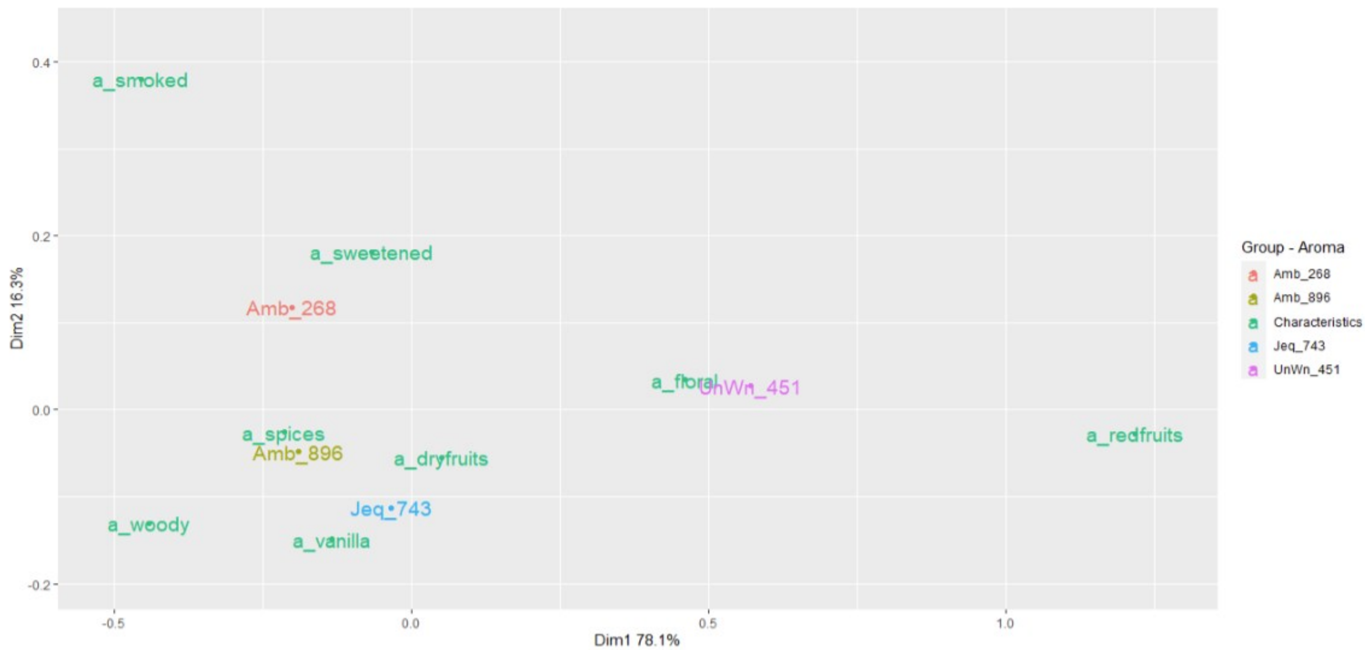

Figure 3. Representation of aged and non-aged wines in Dimensions 1 and 2 in relation to the aroma attributes raised in the sensory analysis.

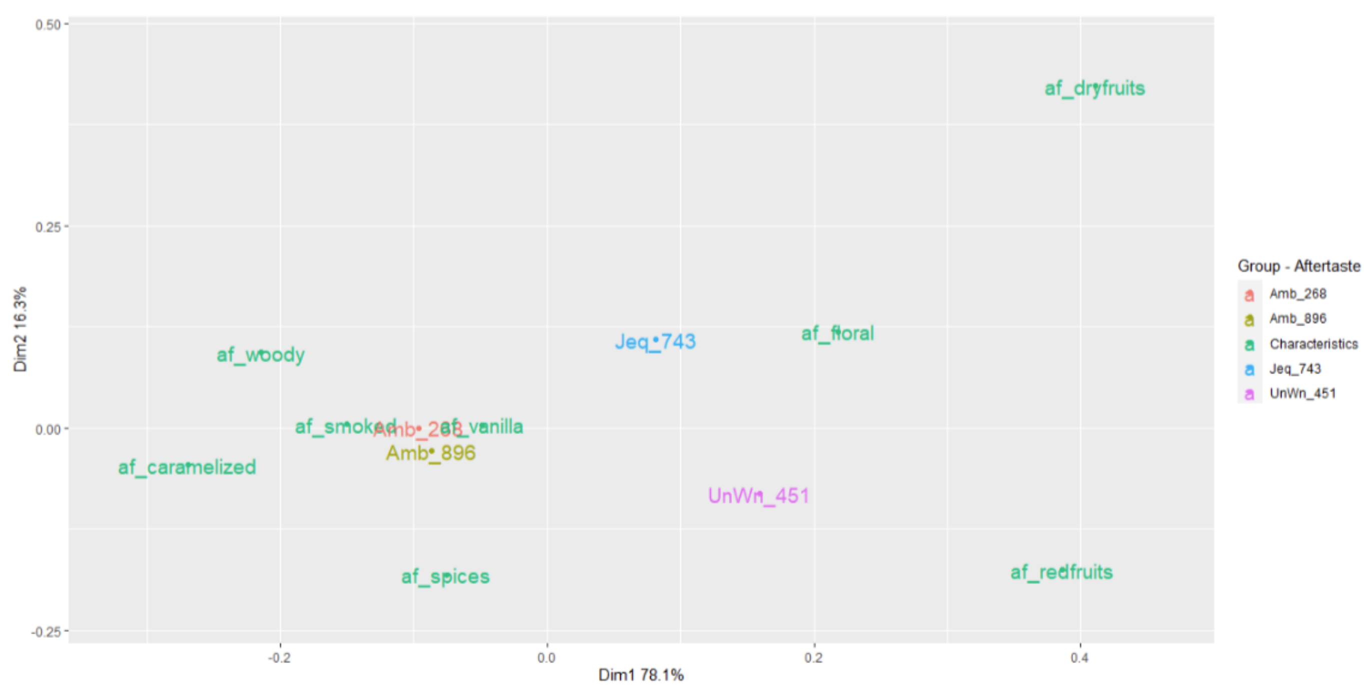

Figure 4. Representation of aged and non-aged wines in Dimensions 1 and 2 considering the aftertaste attributes raised in the sensory analysis.

5 CONCLUSION

Tropical woods used in this study, Jequitibá Rosa and Amburana, showed good potential for aging wines, presenting aging markers in some cases superior to French oak, such as vanillic acid, or closely, syringic acid. During the hedonic test, when compared with non-aged wine, they showed greater acceptance by the panel of tasters, receiving evaluation scores, on average, 23% higher.

The sensory attributes provided by these species, listed by the panel in the UltraFlash Profile methodology, can play an important role in the formulation of new products and give originality to aged wines. In addition, with the correlation between the different sensory attributes provided by Pearson's matrix, it is possible to predict sensory characteristics that are highly related to each other or that are antagonistic, for choosing the species to be used and the expected sensory profile.

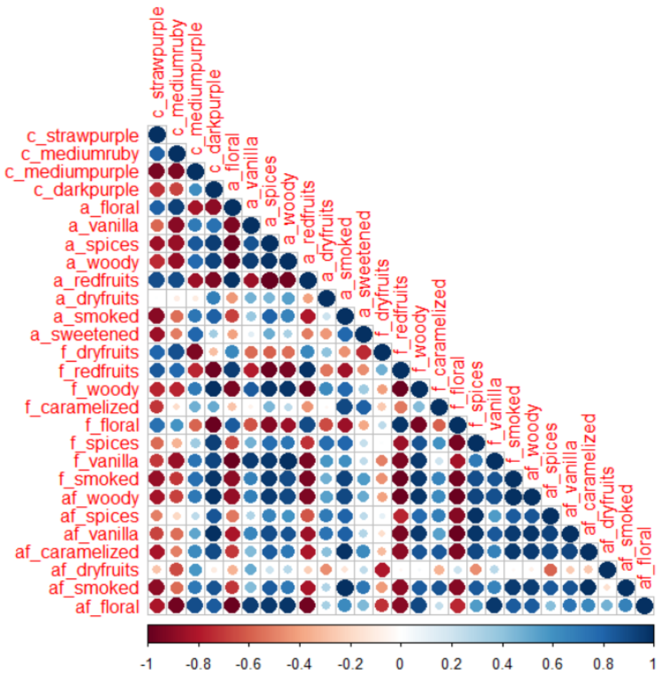

Figure 5. Pearson correlation graph demonstrating the behavior of the variables among themselves.

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