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**Title:**

# Effect of different closure types and storage temperatures on the color and sensory characteristics development of Argentinian Torrontes Riojano white wines aged in bottles for 18 months.

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**Running title:**

Changes in organoleptic properties of Torrontes Riojano white wines produced by different aging conditions.

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## Abstract

During aging, most bottled white wines lose their distinctive organoleptic characteristics according to storage conditions (closure, temperature, time). However, the combined effect of these factors on organoleptic characteristics of Torrontes Riojano wines (TRw) has not been studied yet. This study aimed to evaluate the influence of closure type and storage temperature on the organoleptic properties development of TRw during 18 months of aging. For this, the wine was bottled with natural (C-TRw) and synthetic (SyC-TRw) corks, and screwcaps (SC-TRw). Bottles were kept for 18 months in thermostatized chambers (15°C; 25°C). At different aging times, consumed oxygen (CO), $SO_2$, total phenols (TP), color, and sensory properties were evaluated. CO, TP, $SO_2$, and browning index evolutions depended on the closure-temperature interaction; whereas CIELab parameters (lightness, chroma, hue) on closure-time and temperature-time interactions. At both storage temperatures, SC-TRw had a lower hue decrease and lower chroma increase than C-TRw and SyC-TRw. The highest temperature prompted that TRw color was more yellow and intense. Thus, TRw were discriminated against throughout the conservation. Considering the aging process as a whole, the aromatic intensity, fresh fruit, yellowish and greenish nuances decreased, while brownish hue, color intensity, linalool, and oxidized character rose as storage time increased. At the end of aging, TRw kept to 15°C were not differenced by closure, and they were characterized by fresh fruit, floral, and high aromatic intensity attributes. At 25°C, SyC-TRw presented higher color intensity and herbaceous characters, while C-TRw and SC-TRw showed a more oxidized character. In conclusion, the interaction closure type-storage temperature is critical for organoleptic properties stability of bottled TRw during aging. Thus, screwcaps and low-temperature storage conditions can preserve the TRw varietal characteristics, significantly increasing their shelf-life.

**Keywords:** Torrontes Riojano white wines, storage conditions, consumed oxygen, sulfur dioxide, wine color, sensory analysis.

## 1. Introduction[·]

Torrontes Riojano (*Vitis vinífera* L.) is an Argentine white variety that originated from a natural cross between Listan Prieto and Moscatel de Alejandría (Torres et al., 2015). This variety, which is noted by its plasticity of implantation in different agro-ecological regions, produces wines with distinctive organoleptic characteristics (Fanzone M., Griguol R., Mastropietro M., Sari S., Pérez D., Catania A., Jofre V., Assof M., 2019). Traditionally, these wines are consumed in the year; although, based on their experience, winemakers state that they have a great potential to be aged. However, there are no scientific criteria to prove which are the optimal storage conditions for Torrontes Riojano wines to keep their typicality during long periods of conservation.

As is well known, the chemical composition and sensory attributes of bottled young wines change during aging, depending mainly on storage conditions (e.g. closure type, temperature, time) (Tarko et al., 2020; Giuffrida-de-Esteban et al., 2019). The closure type has direct implications on the development of the color and aromas of the wines during aging. This is because depending on its air permeability, it allows the entry of differential amounts of oxygen into bottles, facilitating the progress of degradative reactions on compounds related to the organoleptic properties of the wines (Lagorce-Tachon et al., 2016; Ugliano, 2013). Wine post-bottling development is complex and differs between red and white wines. Whereas red wines benefit from a small degree of oxygenation as it contributes to color stabilization, astringency reduction, and aroma improvement (Ćurko et al., 2021); white wines are less resistant to oxygen, leading to oxidative off-flavors and browning that reduce wine quality (Coetzee et al., 2016). However, tight-sealing and lack of oxygen can also lead to negative sensory attributes (Karbowiak et al., 2010). On the other hand, during aging, there are changes in the balance of the $SO_2$ active forms depending on oxygen level entering through the closure and

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[·] **Abbreviations:**

**TRw:** Torrontes Riojano wines. **C:** natural cork. **SyC:** coextruded-synthetic stopper. **SC:** screwcap. **C-TRw:** TRw sealed with natural corks. **SyC-TRw**: TRw sealed with synthetic stoppers. **SC-TRw:** TRw sealed with screwcaps. **mx-CO:** maximum consumed oxygen. **HSP:** headspace pressure. **C-TRw-15, SyC-TRw-15, SC-TRw-15:** TRw with different stoppers aged at 15°C. **C-TRw-25, SyC-TRw-25, SC-TRw-25:** TRw with different stoppers aged at 25°C. **GH:** greenish hue. **YH:** yellowish hue. **FF:** fresh fruits. **LL:** linalool. **OX:** oxidized character. **BT:** bitterness. **AC:** acidic taste. **AI:** aromatic intensity. **OB:** orange blossom. **CA:** chemical aromas. **CI:** color intensity. **SW:** sweetish taste. **FL:** floral. **HB:** herbaceous. **BH:** brownish hue.

storage temperatures, among others (Karbowiak et al., 2019; Arapitsas et al., 2014). The changes of these factors provoke a decrease of free $SO_2$, causing the loss of its antioxidant capacity on the reactive oxygen species and favoring the development of oxidative reactions (J. C. Danilewicz, 2011; Elias & Waterhouse, 2010).

During the oxidation process, o-diphenols of wines are oxidized to o-quinones and semiquinone radicals, whereas the oxygen is reduced to $H_2O_2$. These radical species may undergo further reactions, e.g. condensation reactions, which lead to the formation of high molecular weight colored products (Waterhouse & Laurie, 2006; J. Danilewicz, 2003). Furthermore, as quinones are electrophilic compounds quickly react with some phenols, producing dimers or polymers that may rearrange their structure to form new o-diphenols. These regenerated o-diphenols will be newly oxidized and, consequently, accelerate the polymerization reactions of phenolic compounds (Li et al., 2008). On the other hand, the $H_2O_2$ formed during the phenols oxidation process, in combination with $Fe^{+2}$ through the Fenton reaction, generates hydroxyl radicals (Elias & Waterhouse, 2010). This last radical species are not selective and react with ethanol and tartaric acid to form acetaldehyde and glyoxylic acid, respectively. These carbonylic compounds are good nucleophiles and can intervene in ulterior reactions associated with wine color development. For instance, glyoxylic acid reacts with catechin to produce a (+)-catechin/glyoxylic acid adduct, which reacts with a further (+)-catechin to form a carboxymethine linked (+)-catechin dimer. The dehydration of these dimers forms xanthenes, which can undergo oxidation generates yellow xanthylium salts that have a maximum absorption around 440 and 460 nm (Tarko et al., 2020; Bührle et al., 2017; Laurie & Waterhouse, 2006). Also, during storage, volatile substances are modified due to the different reactions that occur, including hydrolysis, esterification, and oxidation. During conservation, the freshness and fruity aromas of young wines are mainly lost through ester hydrolysis (Coetzee et al., 2016; Coetzee, 2014; Oliveira et al., 2011); and these reactions are accelerated by high storage temperatures (Cejudo-Bastante et al., 2013; Hopfer et al., 2012). On the other hand, the oxidation of white wines is characterized by the loss of characteristic varietal and secondary aromas and the formation of atypical aromas associated with the wine deterioration, such as honey-like, cooked vegetable, farm food, among others (Coetzee et al., 2016; Karbowiak et al., 2010).

Therefore, the aging process and its suitable management are crucial to preserve the varietal characteristics and to obtain wines with the style and quality wanted. To the best of our knowledge, there is no published information on the interactive effect of different aging factors on the organoleptic properties evolution of

Torrontes Riojano wines. Thus, the aim of this work was, on the one hand, to study the joint effect of the type of closure and the storage temperature on the evolution of oxygen, sulfur dioxide, and phenolic compounds; and on the other hand, to evaluate the development of the color and the sensory characteristics of Torrontes Riojano wines (Mendoza, Argentina) throughout 18 months of bottle aging.

## 2. Materials and Methods

### 2.1. Wine samples and experimental design

Torrontes Riojano grapes (22.2°Brix, season 2016, Ugarteche, Mendoza, Argentina) were vinified in Fincas Patagonicas winery using standard protocols. The alcoholic fermentation was conducted by Uvaferm CGC62 (20 $g.hL^{-1}$, Lallemand-Inc., Canada) into a 150 hL stainless-steel tank. After stabilization treatments, molecular sulfur dioxide was adjusted to 0.8 $mg.L^{-1}$. The Torrontes Riojano wine (TRw) was fractionated (15±2°C) in 750-mL transparent bottles (300 units). One hundred bottles were closed with natural cork (C, 24x45mm, Portocork, USA), 100 with coextruded-synthetic stopper (SyC, 24x45mm, SelectSeriesTM, Nomacorc, USA), and 100 with screwcap (SC, 30×60mm, tin-foil-Saran/Tin, Arpex-Internacional, Argentina). Bottle headspace volumes were 6 $cm^3$ for C and SyC, and 16.6 $cm^3$ for SC. Bottling manipulations (filling, purging, etc.) were performed using high-purity nitrogen (Praxair-TechInc., Argentina). Bottles were divided into 2 sets, each consisting of 50-C, 50-SC, and 50-SyC, which were placed vertically in cardboard boxes and kept in thermostatized chambers (T15:15±2.4°C, T25:25±1.9°C) for 18 months. Sampling (bottles in triplicate) was at 0, 2, 3, 4, 6, 9, 12, 15, and 18 months. The microbiological stability was evaluated by OIV protocols (OIV, 2010).

### 2.2. Oxygen measurements

Twenty-four bottles were adapted for oxygen measurements (4 per treatment). In each one, using food-grade silicone, 2 sensors (Planar-Oxygen-Sensitive-Spot-PSt3, PreSens, Germany) were glued: one in the middle of the bottleneck, and one in the middle of the bottle-body. Dissolved (DO) and headspace oxygen (HSO) concentrations were measured by luminescence non-destructive technology Fibox3-Trace fiber-optic oxygen-meter (PreSens, Germany). The readings were taken without shaking bottles. Initial measurements (zero time) were considered at 20 hours after bottling when the percentual relative standard deviations (%RSD) of measurements were below 20%. Subsequent measurements were every 3 days during the first 6 mos, 1 time/week during the second semester, and 1 time/15-day from 12 to 18 months. Consumed-oxygen (CO) was estimated by Vidal, Toussaint, & Salmon (2014). Oxygen transmission rate values (OTR, provided by cap-suppliers) were 4.56, 4.07, and 0.61 mg.L$^{-1}$/year for C, SyC, and SC, respectively.

### 2.3. Chemical and physical parameters measurements

Standard enological parameters were determined by a platinum-diamond ATR single-reflection sampling module-cell coupled to a Bruker-Alpha instrument-OPUS software (Bruker-Optics, Germany). Free ($fSO_2$) and total ($tSO_2$) sulfur dioxides were measured with a FIAstar™5000 analyzer (Foss Analytical, Denmark). The UV-vis-spectrophotometer (Lambda25, PerkinElmer, USA) was used for absorptiometric measurements. Total phenols (TP) were determined by the Folin-Ciocalteau assay (Singleton et al. 1999). CIELab parameters and color differences were determined by Giuffrida-de-Esteban et al. (2019). Table 1 shows the chemical and physical parameters of the initial wine.

**Table 1**

### 2.4. Sensorial analyses

A judging panel (9 women and 4 men, aged 30 to 50) was constituted to carry out sensory studies. In each session, 30-40 mL of wine (8°C-10°C) were dispensed in tasting glasses (IRAM, 1999). Each panelist worked in an isolated booth (lit by full-spectrum 6500 K Candil-lamps). Panel training was performed over 14 sessions. In the first, using commercial TRw wines, standardized sensory evaluating criteria. In the second and third sessions, pattern blind identifications (acidity, bitterness, aromas, Supplementary Table 1) were done. In the 4th, following Cadena, Vidal, Ares, & Varela (2014), the visual, taste, and olfactory attributes of the young-TRw were evaluated and selected. In the fifth, Triangular tests were done with 3-month-old TRw. For this, consecutive sessions corresponding to each treatment were carried out. In those sessions, three glasses with different combinations of treatments were assessed (e.g., C15-C25-C15; C15-SC15-C15), and the tasters had to select the odd sample. The judges rested five minutes between sets to minimize the tiredness of sense-organs. In these studies, it was considered that the samples analyzed were significantly different from the control, at a 95% confidence level, when 10/13 judges made the correct choice over the whole set of wines (Roessler, R. M. Pangborn, 1978). In the remaining sessions, before descriptive sensorial analyses (6, 12, and 18 months), judges agreed on descriptors used to characterize treatments. To evaluate each treatment, three sessions were conducted for each one, where judges were only informed about wines´ variety.

### 2.5. Statistical analyses

Data were analyzed by multifactorial analysis of variance (Multifactorial ANOVA). Tuckey-HSD-test, Levene-test, and Pearson-test ($\alpha$=0.05) were used to analyze differences among treatments, homogeneity of variances, and the correlation between variables, respectively. Principal Component (PCA) and Linear Discriminant (LDA) Analyses were performed on sensory analyses. The sensory data were collected with Sodelsa's free software. Statgraphics Centurion-XVI (StatPoint-Technologies-Inc., USA), GraphPad Prism-7 (GhaphPad-Software-Inc., USA), and the free R-SensoMineR-package were employed for statistical analyses.

## 3. Results and discussion

### 3.1. Wine composition

At bottling, the TRw presented pH 3.20, 13.40 %V/V ethanol, 0.30 g.L$^{-1}$ volatile acidity, and 6.00 g.L$^{-1}$ total acidity. During wines´ conservation, these parameters were not significantly affected by closure types, nor storage temperatures (Supplementary Table 2). For all treatments, at 18 mos of aging, ethanol contents and pH values did not show differences from the initial wine. Throughout the conservation, total acidity decreased to 5.08 ±0.62 g.L$^{-1}$ and volatile acidity increased to 0.58±0.03 g.L$^{-1}$. These results are in agreement with other studies focused on the conservation of the white wine from different varieties (Ricci, Parpinello, & Versari, 2017; Liu et al., 2015; Hopfer, Ebeler, & Heymann, 2012; Lopes et al., 2009). This increase in volatile acidity, which remained below the acceptable level of 0.7–0.9 g.L$^{-1}$ (Goode J. and Harrop S., 2011), could be linked to chemical oxidative processes (Bakker & Clarke, 2011) and no to microbiological spoilage (Supplementary Table 3).

On the other hand, TP content declined as the aging time rose. Over the first year, TP evolution was conditioned by the closure type-storage temperature interaction (p-value 0.0388); however, from months 12 to 18, there weren't any statistical differences among treatments (Supplementary Table 2). During the conservation at 15°C, Torrontes Riojano wines sealed with screwcap (SC-TRw) showed higher TP levels compared with those closed with corks (C-TRw) and synthetic stopper (SyC-TRw); but at 25°C the wines were no differentiated by closure types. The TP drops observed during aging could be associated with chemical reactions (polymerization, complexation, condensation) where wine phenolic compounds are involved (Pati, Crupi, Savastano, Benucci, & Esti, 2020; Kallithraka, Salacha, & Tzourou, 2009). Although phenolic constituents are slightly dissociated (pKa ~ 9 to 10) at wine´s pH and do not react directly with oxygen, they are the key substrate for non-enzymatic oxidations during conservation (Oliveira, Ferreira, De Freitas, & Silva, 2011; Waterhouse & Laurie, 2006; J. Danilewicz, 2003). Some authors proposed that oxidation, based on the Fenton reaction, would result in the formation of semiquinone radicals, which later oxidize to quinones (Elias & Waterhouse, 2010; J. Danilewicz, 2003). And these reactions, which facilitate further condensations leading to the formation of phenolic polymers (Oliveira et al., 2011), can be favored by high storage temperatures (Recamales, Sayago, González-Miret, & Hernanz, 2006).

### 3.2. Oxygen and sulfur dioxide

From bottling to the fourth mos of conservation, the evolution of oxygen was dependent on the interaction closure-storage temperature (p-value<0.0001). From 120 days of conservation, until the end of the aging process, the oxygen evolution was changing by the effect of closure type and was independent of storage temperature (Figure 1). Also, throughout the first four months of aging, the CO was the highest in the entire aging process, probably due at the beginning of the conservation the species reactive to oxygen in wines are found in high concentrations (Ugliano, 2013). Besides, there was a maximum decrease in DO, falling 70 times below its initial value (Supplementary Figure 1).

**Figure 1**

The C-TRw conserved at 25°C showed a fast increase of CO, reaching its maximum value (mx-CO, 1.008±0.006 mg/bot) between days zero and 15, but DO was slowly decreasing during that time. At the beginning of the trial, the system can be considered to be in a pseudo stationary state, since in that period the HSO remains high and can recompose the DO lowering, which is being consumed in different chemical processes where it is involved (Perez-Benito, 2017; Ugliano, 2013; Navarro-Laboulais J. et al, 2012). The same behavior was observed in SyC-TRw (mx-CO, 0.939±0.111 mg/bot) and SC-TRw (mx-CO, 0.648±0.1373 mg/bot).

On the other hand, TRw conserved at 15°C showed mx-CO between days 30 and 50 of aging, while DO had a significant fall (Supplementary Figure 1). For C-TRw and SyC-TRw, the mx-CO was, on average, 1.194±0.213 mg/bot and 1.029±0.295 mg/bot, respectively. Into the same period, DO went from 1.750±0.210 mg/bot to 0.018±0.007 mg/bot for C-TRw, and from 1.128±0.028 mg/bot to 0.024±0.006mg/bot for SyC-TRw. This could be cos the system, outside of the stationary state, would not be able to compensate for the DO loss neither by the concentration of oxygen into headspace nor by the oxygen that entering through the closure (Navarro-Laboulais J.; B. Cuartas-Uribe; E. Ortega-Navarro; P., 2012). In turn, SC-TRw presented an mx-CO of 0.709 ±0.059 mg/bot, while DO did not vary significantly. Likewise, during this aging time at both storage temperatures, C-TRw and SyC-TRw showed about 1.4 times more mx-CO than SC-TRw. This would be associated with the differences in headspace pressure (HSP) they had. At the onset of the experiment, C-TRw, and SyC-TRw presented HSP values twofold superior to SC-TRw (data not shown). Consequently, at a constant temperature, and according to Henry's law, C-TRw, and SyC-TRw could have had higher DO, which would be more available to react with wines´reducing compounds (Peters, 2017; Dimkou et al., 2011). Additionally, when bottles are sealed with cylindrical caps, an additional amount of oxygen can enter into the system during the first weeks of aging. This oxygen ingress could be associated with the compression generated in the bottleneck at bottling or with the gas transfer through the glass/stopper interface (Chanut et al., 2021; Lagorce-Tachon et al., 2016; Ugliano et al., 2011). This fact would be leading to an increase in partial pressure into headspace, which could promote dissolution and consumption of oxygen.

Besides, the period of maximum oxygen consumption depended on storage temperatures. During the first four-monthly of aging, TRw stored at 15°C showed a delay (15-20 days) in reaching mx-CO compared to wines kept at 25°C. This might be because the higher storage temperature could have facilitated the triplet oxygen (oxygen molecular form) conversion into radical species (oxidizing agents), increasing the oxygen consumption rate (Ugliano, 2013; Oliveira, Ferreira, De Freitas, & Silva, 2011; Karbowiak et al., 2010; Danilewicz, 2003). After the period described above, the differences found in oxygen consumption were related to the closure type (p-value<0.05), but not to storage temperatures. From the 4th to 18th month, the CO declined in all treatments (Fig. 1), although for SC-TRw it was 21% higher than for C-TRw and SyC-TRw. These results were similar to those shown by Dimkou et al. (2011) in their studies on CO evolution for Riesling wines closed with screwcaps and coextruded cork aged for 2 years. The CO differences observed in this trial could be associated with the stoppers´ physical structures. While corks and screwcaps are practically impermeable to air, the trapped air within the cork structure could be slowly transferred into wines throughout aging (Lopes et al., 2007). Also, as OTRs are linked to the differential permeability of stoppers to the surrounding air, this would lead to different amounts of oxygen (SC<SyC<C) being diffused through the caps into wines during storage (Lagorce-Tachon et al., 2016).

Related to sulfur dioxide evolution throughout the trial, Torrontes Riojano wines showed a reduction in $fSO_2$ and $tSO_2$ concentrations (Supplementary Table 2). During the first four months of conservation, regardless of treatment, the $fSO_2$ fell near to 50% of their initial value; and from then until the end of aging, it reached values below 5 $mg.L^{-1}$. The $fSO_2$ diminution could be related to different chemical processes where it participates during aging. As mentioned previously, to react with wine components, the dissolved oxygen must be converted into $^{\cdot}OOH$. This radical specie, when in the medium there are $Fe^{+2}$ and/or $Cu^{+}$, reacts with phenolic compounds generating hydrogen peroxide ($H_2O_2$). But this process is controlled by $fSO_2$ which competes with those transition metal ions by $H_2O_2$. Thus, $fSO_2$ prevents the unspecific oxidation of $H_2O_2$ on organic compounds of wines, favoring the chemical and organoleptic stabilities of these beverages over time (J. C. Danilewicz, 2011; Elias & Waterhouse, 2010). Also, $fSO_2$ may decrease due to its participation in the reduction of quinones, which are formed by oxidation of phenolic compounds (Oliveira et al., 2011). With regard, a strong correlation between $fSO_2$ decrease and TP diminution was observed ($r_{15°C}=0.906$; $r_{25°C}=0.934$). Besides, throughout the aging process, the $fSO_2$ fall at 25°C was almost 6% more than at 15°C. It is well known that higher storage temperatures enhance $H_2O_2$ production and increase oxidative reaction rates (Karbowiak et al., 2010). This would lead to a greater loss of $fSO_2$ as its consumption could be increased to reduce the harmful effects of radical species on easily oxidizable compounds in wines. On the other way, elevated storage temperatures would also facilitate the tannins-bisulfite reaction to generate monosulfonated flavanols, thereby resulting in lower $fSO_2$ concentrations during wine aging (Arapitsas et al., 2014).

On the other hand, a decline in $tSO_2$ concentration depended on closure type-storage temperature interaction ($p\text{-value}<0.05$). At the end of the aging, C-TRw-15, SyC-TRw-15, C-TRw-25, and SyC-TRw-25 showed, on average, 1.3% $tSO_2$ lesser than SC-TRw-15 and SC-TRw-25. Moreover, wines stored at 15°C had nearly 2 times more $tSO_2$ than those aged at 25°C. These results might indicate that when $fSO_2$ decrease, the bound $SO_2$ forms ($HSO_3^-$/carbony-compounds hydrolyzable adducts) could begin to dissociate to restore the broken $fSO_2/tSO_2$ equilibrium, with a consequent drop in $tSO_2$ concentration (Sacks, Howe, Standing, & Danilewicz, 2020; Waterhouse et al., 2016; J. C. Danilewicz, 2016). Furthermore, at both storage temperatures, the differences in $tSO_2$ found between wines with porous stoppers and those sealed with screwcaps could be related to the amount of oxygen entering the bottle during aging. When the oxygen input is moderate, as in SC-TRw, $fSO_2$ is preferentially consumed; but when the high $O_2$ input, as in C-TRw, and SyC-TRw, not only the free forms are mobilized, but also the $SO_2$ reversibly bound forms (Karbowiak et al., 2019). Therefore, the OTR high values of porous stoppers compared to screwcaps would favor the $tSO_2$ loss, as has been observed in this study (Supplementary Table 2).

### 3.3. Wine color

The non-enzymatic browning during the wine conservation, among other mechanisms, would be associated with the oxidation of phenolic compounds and the later polymerization of their oxidized products, or with the polymerization reactions between phenols and other compounds such as acetaldehyde or glyoxylic acid (Li et al., 2008). The phenolic compounds of white wines as the o-diphenols (gallic and caffeic acids and its esters, catechin, epicatechin, and their derivatives) are considered the most susceptible compounds to non-enzymatic oxidation. Also, the flavan-3-ols had shown significant correlations with the browning degree of white wines (Scrimgeour, Nordestgaard, Lloyd, & Wilkes, 2015; Motta, 2013; Waterhouse & Laurie, 2006; J. Danilewicz, 2003; Fernández-Zurbano, Ferreira, Escudero, & Cacho, 1998). During aging, absorbance at 420 nm (A420) has been employed as a useful index to evaluate the browning degree of white wines due to non-enzymatic oxidation (Pati et al., 2020). The A420 evolution depended on closure type-aging time and storage temperature-aging time interactions (Table 2). The wines' pale yellow color was changing to intense yellow as the storage time increased, and the A420 increase rate rose with increasing temperature. To observe the correlation of the A420 rise with the TP diminution during storage time, a regression study was carried out. The correlation coefficient between A420 and TP decreased with increasing storage temperature ($r_{15°C}$=-0.850; $r_{25°C}$=-0.945). Also, A420 at 25°C increased 1.3 times more than 15°C (Supplementary Figure 2). This would indicate that higher color development would be linked to the TP reduction, which can be accelerated by elevated aging temperatures (Ricci et al., 2017; Scrimgeour, Nordestgaard, Lloyd, & Wilkes, 2015; Li, Guo, & Wang, 2008). Besides, throughout the storage, C-TRw-25 and SyC-TRw-25 presented a superior browning degree to the rest of the wines; but between them, there were not any significant differences. Also, the A420 values for SC-TRw-25 were greater in comparison with C-TRw-15 and SyC-TRw-15; however, among these treatments, there weren't any statistical differences (p-value>0.05). Whereas, the A420 for SC-TRw-15 was increased slowly along time and was lesser than the other wines. Moreover, from the beginning to the end of the storage, the A420 mean percentual rise was 38.70% for C-TRw-25 and SyC-TRw-25, 32.58% for SC-TRw-25, 31.51% for C-TRw-15 and SyC-TRw-15, and 24.98% for SC-TRw-15. Furthermore, Torrontes Riojano wines sealed with screwcaps had the lowest CO concentration during aging (Figure 1), which would mean that the use of this closure type and low storage temperatures might provide better color preservation for these white wines over time.

**Table 2**

**Figure 2**

The color of Torrontes Riojano wines was also assessed employing the CIELab coordinates. During aging, lightness (L*), chroma ($C^*_{ab}$), and hue ($h_{ab}$) evolutions depended on closure type-aging time and storage temperature-aging time interactions, whereas the green/red (a*) and yellow/blue (b*) color components were mainly affected by the storage temperature (Table 2 and Supplementary Table 2). Figure 2B shows the distribution of wines from different aging treatments throughout conservation on the CIELab a*b* color plane. In this plot, the samples were located in the $h_{ab}$ region defined between 91° and 99°, related to yellow tones with a very slight tendency to green. In general, over the 18 months of storage, the wines showed characteristic color changes associated with aging (decreases of $h_{ab}$ and L*, and increases of $C^*_{ab}$). During the first months of storage, the wines kept at 15°C were grouped closer to the origin of coordinates and had lower $C^*_{ab}$ values (associated with higher white light transmission), which determined the final color of these wines to be in the pale yellow category. In opposite, at the same aging period, wines stored at 25°C presented higher b* and $C^*_{ab}$ values, related to the major browning degree that they showed. Also, as the b* and $C^*_{ab}$ coordinates increased during time storage (C-TRw-25 and SyC-TRw-25 had the highest values, SC-TRw-15 the lowerest, and the other wines intermediate values), result in Torrontes Riojano wines´ color became more yellow and more intense (lower L*). Besides, during the aging at both storage temperatures, SC-TRw treatment displayed a lower decrease of $h_{ab}$ compared to C-TRw and SyC-TRw, showing that screwcaps could favor the preservation of wines´ yellow-greenish nuances. Furthermore, for SC-TRw-15, the L* values were not significantly affected across conservation; and, at the end of the trial, they remained higher than for the rest of the treatments, which were not differentiated (p-value>0.05) neither by closures nor storage temperatures.

Additionally, the color difference ($\Delta E^*_{ab}{}^{(f\text{-}i)}$) between specific aging times (6-0, 12-0, 18-0) was evaluated. The $\Delta E^*_{ab}{}^{(f\text{-}i)}$ 3 has been quoted as a minimum value to discriminate between wines by an average-observer (Giuffrida de Esteban et al., 2019; Martínez, Melgosa, Pérez, Hita, & Negueruela, 2001). However, such estimates must be done with caution, as those $\Delta E^*_{ab}{}^{(f\text{-}i)}$ limit values were used to differentiate red wines. Other authors employed $\Delta E^*_{ab}{}^{(f\text{-}i)}$ 1 and $\Delta E^*_{ab}{}^{(f\text{-}i)}$ 2 for white wines comparations (Šottníková, Hřivna, Jůzl, Cwiková, & Šottníková, 2014; Lopes et al., 2009), and these values might be more appropriate to evaluate the color evolution of Torrontes Riojano wines during aging. All wines conserved at 25°C showed $\Delta E^*_{ab}{}^{6\text{-}0)}$, $\Delta E^*_{ab}{}^{(12\text{-}0)}$, and $\Delta E^*_{ab}{}^{(18\text{-}0)}$ values above to 2; although at each point, the $\Delta E^*_{ab}{}^{(f\text{-}i)}$ values for SC-TRw-25 were always 1.5 times less than the C-TRw-25 and SyC-TRw-25. While C-TRw-15 and SyC-TRw-15 exceeded that reference value from month 12, whereas SC-TRw-15 only showed $\Delta E^*_{ab}{}^{18\text{-}0)}>2$ (Supplementary Table 2). These results confirm that the Torrontes Riojano white wine color changes during storage, being particularly important from 6 months onwards, especially for those closed with natural cork and coextruded synthetic cork. Thus, the color differences observed for the C-TRw and SyC-TRw treatments could be related to the higher oxygen consumption they had during aging (which facilitated the phenolic compounds' oxidation) compared to wines sealed under more airtight conditions (Fig. 1).

### 3.4. Sensory analyses

In general, Torrontes Riojano young wines are sensory characterized by a pale golden-yellow color, a slightly bitter aftertaste, and a high aromatic intensity, regardless of the agro-ecological zone where the grapes come from (Fanzone M., Griguol R., Mastropietro M., Sari S., Pérez D., Catania A., Jofre V., Assof M., 2019). Those sensory attributes could be related to the non-volatile ((+)-catechin, caffeic acid, quercetin-3-glucuronide) and volatile (holotrienol, linalool, geraniol, nerol, β-citronellol, β-cyclocitral, β-damascenone) varietal composition identified in these young wines, and to fermentative compounds (tyrosol, ethyl esters, higher alcohol acetates, higher alcohols) arising from winemaking (Pérez, Assof, Bolcato, Sari, & Fanzone, 2018; R. Romano, 2013). In the present study, to observe whether the different conservation treatments were beginning to affect the sensory profile of Torrontes Riojano wines, the Triangular test was performed at 3 months of storage. In these ABA tests, only C-TRw-15 from C-TRw-25 and SyC-TRw-15 from SyC-TRw-25 were discriminated against, evidencing that closure types and storage temperatures were beginning to affect the sensory characteristics of Torrontes Riojano wines. Then, at 6, 12, and 18 months of storage, the sensory profiles of the wines were carried out through descriptive sensory analyses. The attributes selected by the judging panel to describe the wines depended on the evaluated aging time. At 6 months of wines storage, the consensus attributes were greenish hue (GH), yellowish hue (YH), fresh fruits (FF), linalool (LL), oxidized character (OX), bitterness (BT), acidic taste (AC), and aromatic intensity (AI). At 12 months were GH, YH, FF, LL, OX, BT, AC, AI, orange blossom (OB), chemical aromas (CA), color intensity (CI), and sweetish taste (SW). And, at 18 months were YH, FF, OX, BT, AC, AI, CI, floral (FL), herbaceous (HB), and brownish hue (BH). To analyze the influence of different factors over the sensory attributes of Torrontes Riojano wines across aging, the Multifactorial ANOVA was conducted (Table 3). The factors storage temperature, aging time, judge (randomly enumerated in each session), and session (replicates) were nested within closure type factor. It was observed that the judge and replicate factors had no significant effect (p-value>0.05) on the sensory differences found among the treatments.

**Table 3**

Then, at each evaluated aging time, to highlight the similarity of the samples, and to determine the main attributes that contributed to differentiating them, the Principal Component Analysis (PCA, unsupervised pattern recognition technique) was performed (Supplementary Figure 3). At 6 months of storage, the PCA analysis explained the 94.45% of the data variance. The Principal Component 1 (PC1, 87.19%), related to the high GH, LL, FF, AI, OX, and YH eigenvalues, allowed to discriminate wines by the storage temperature effect; and the PC2 (7.26%) was mainly correlated to AI and BT eigenvalues, which contributed to separate the wines by the closure type effect. Torrontes Riojano wines kept at 15°C were characterized by GH, LL, and FF attributes, although C-TRw-15 and SyC-TRw-15 had lower aromatic intensity than SC-TRw-15. On the other hand, C-TRw-25 and SyC-TRw-25 were associated with oxidized characters (caramelized, honey aromas) and yellow nuances; but SC-TRw-25 presented higher AI and lower OX and YH scores than the other wines stored at 25°C. At 12 months of aging, the PCA explained the 90.95% of the data variance and showed that the judging panel was able to differentiate the wines by the temperature effect. The C-TRw-15, SyC-TRw-15, SC-TRw-15 were not discriminated by closure types and were associated with FF, LL, and OB attributes. In opposite, C-TRw-25, SyC-TRw-25, and SC-TRw-25 were showed higher scores of color intensity, oxidized character, and yellowish hue. In this aging time, wines kept at 25°C also presented rubbery, sulfurous, and metallic flavors, which the panel described as chemical aromas. At 18 months of storage, the PCA analysis explained 86.46% of the total variance. The judges were unable to discriminate among C-TRw-15, SyC-TRw-15, and SC-TRw-15, and associated them with high FL, FF, and AI attributes. At this aging time, the yellowish nuances evolved into browning hues evidencing that the oxidation process was advanced, especially at higher conservation temperatures. The SyC-TRw-25 had the highest BH and CI scores, whereas C-TRw-25 and SC-TRw-25 presented more oxidized character; thus at 25°C, the Torrontes Riojano wines could be differentiated by the closure type effect.

On the other hand, to evaluate how the sensory attributes of Torrontes Riojano wines evolved through conservation, the Linear Discriminant Analysis (LDA) was performed. LDA is a supervised pattern recognition method based on the determination of linear discriminant functions, which maximize the ratio of between-class variance and minimize the ratio of within-class variance (Berrueta et al., 2007). For this purpose, and to assure independence among the variables (a mandatory requirement for the LDA technique), the information obtained from the exploratory analyses (PCA) and the Multifactorial ANOVA (Table 3), was submitted to correlation studies (Pearson's test, $\alpha$=0.05). Considering the correlation coefficients among variables in the correlation matrix (data not shown), the attributes CI, LL, BH, OX, FF, AI, GH, and YH were selected as predictor variables.

From the selected predictor variables, three discriminant functions were obtained (Supplementary Table 4), which together, represented 99.01% of the total variance ($\alpha$=0.01). The Discriminant Functions 1 and 2 explained 87.25% and 9.36% of the total variance, respectively, and had Wilks Lambda values lesser than $1.10^{-5}$, which shows that Torrontes Riojano wines could be discriminated against throughout the conservation. Thus, considering the aging process as a whole, the aromatic intensity of wines alongside fresh fruit, yellowish and greenish nuances decreased, while brownish hue, color intensity, linalool, and oxidized character rose as storage time increased. Figure 3 shows that the wines were sensory discriminated mainly by time and storage temperature; although at 18 months of conservation, the closure type effect allowed to sensory separate those aged at 25°C.

**Figure 3**

## 4. Conclusion

During the aging stage, different physical and chemical processes take place that modifies the organoleptic characteristics of bottled Torrontes Riojano white wines. The permeability of stoppers to the surrounding air affects the intake and consumption of oxygen and, therefore, the stability of compounds associated with the color and sensory properties of wines. The higher the permeability degree, the more oxygen and sulfur dioxide are consumed, since oxidizing and antioxidant reactions are promoted, respectively. As a consequence, during aging, Torrontes Riojano wines sealed with corks and synthetic stoppers show an increase in the browning index, hue, and chroma, and a decrease in lightness, freshness, fruity aromas, and aromatic intensity in a shorter period than those sealed with screwcaps. Also, the storage temperature affects the rate of the reactions mentioned above. The 25°C aging temperature facilitates the development of oxidative reactions, increasing the occurrence velocity of organoleptic defects, and decreasing the quality of the products. In this sense, the interaction between closure type and storage temperature is critical to the organoleptic properties stability of these wines during their bottle aging. Thus, to preserve and increase the shelf-life of Torrontes Riojano wines, the best aging conditions are the use of screwcaps and low storage temperatures.

## CRediT authorship contribution statement

**E. Romina Castellanos:** Validation, Formal analysis, Investigation, Data Curation, Visualization, Writing - original draft. **Viviana P. Jofre:** Conceptualization, Writing, Review & Editing, Formal analysis, Visualization, Supervision, Project administration, Funding acquisition, Writing - original draft. **Martín L. Fanzone:** Conceptualization, Funding acquisition. Review. **Mariela V. Assof:** Conceptualization, Formal analysis, Validation. Review. **Anibal A. Catania:** Formal analysis, Validation. **A. Mariela Diaz-Sambueza:** Formal analysis, Validation. **Francisco J. Heredia:** Formal analysis. Review. **Laura A. Mercado:** Formal analysis, Validation.

## Conflict of interest

The authors declare no conflict of interest.

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## Table Captions

**Table 1:** Chemical and physical parameters of Torrontes Riojano wine at bottling.

**Table 2:** Multifactorial ANOVA for color parameters of Torrontes Riojano wines across the aging process.

**Table 3**: Multifactorial ANOVA for sensory attributes of Torrontes Riojano wines during aging.

**Supplementary Table 1**: Reference standards for sensory analyses of aged Torrontes Riojano wines.

**Supplementary Table 2:** Chemical and physical parameters of Torrontes Riojano wines from aging treatments at different storage times.

**Supplementary Table 3:** Microbiological stability of Torrontes Riojano wines during aging.

**Supplementary Table 4:** Standardized coefficients for discriminant functions based on sensory attributes of Torrontes Riojano wines aged under different conditions for 18 months.

## Figure Captions

**Figure 1:** Evolution of consumed oxygen throughout 18 months of storage for Torrontes Riojano wines aged under different conditions. **A.** CO evolution at 25°C. **B.** CO evolution at 15°C.

CO consumed oxygen (mg/bot equivalent to mg/750 mL). C-TRw Torrontes Riojano wines sealed with natural cork, SyC-TRw with coextruded synthetic cork, SC-TRw with screwcap. 15 and 25 are storage temperatures, 15°C, and 25°C, respectively.

**Figure 2:** Evolution of color parameters throughout 18 months of storage for Torrontes Riojano wines aged under different conditions. **A**. A420 nm (absorbance at 420 nm) evolution during aging. **B**. The CIELab a*b* color plane (a* green/red color component; b* yellow/blue color component; $h_{ab}$ hue angle).

C-TRw Torrontes Riojano wines sealed with natural cork, SyC-TRw with coextruded synthetic cork, SC-TRw with screwcap. 15 and 25 are storage temperatures, 15°C, and 25°C, respectively.

**Figure 3:** Supervised pattern recognition (LDA) based on sensory attributes of Torrontes Riojano wines aged under different conditions for 18 months.

LDA Linear Discriminant Analysis, DF1, and DF2 are Discriminant Function 1 and 2, respectively. Dotted lines are confidence ellipses (statistical significance at 95%). C-TRw Torrontes Riojano wines sealed with natural cork, SyC-TRw with coextruded synthetic cork, SC-TRw with screwcap. 15 and 25 are storage temperatures, 15°C, and 25°C, respectively.

**Supplementary Figure 1:** Oxygen evolution during the firsts 4 months of storage for Torrontes Riojano wines aged under different conditions. **A.** Oxygen evolution for C-TRw-15. **B.** Oxygen evolution for SyC-TRw-15. **C.** Oxygen evolution for SC-TRw-15. **D.** Oxygen evolution for C-TRw-25. **E.** Oxygen evolution for SyC-TRw-25. **D.** Oxygen evolution for SC-TRw-25.

DO dissolved oxygen, HSO headspace oxygen, and CO consumed oxygen (units: mg/bot equivalent to mg/750 mL). C-TRw Torrontes Riojano wines sealed with natural cork, SyC-TRw with coextruded synthetic cork, SC-TRw with screwcap. 15 and 25 are storage temperatures, 15°C, and 25°C, respectively.

**Supplementary Figure 2:** Regression analyses between A420 and total phenols of Torrontes Riojano wines aged at 15°C (**A**), and 25°C (**B**) during 18 months.

A420 absorbance at 420 nm, TP total phenols concentration expressed in g/L gallic acid equivalents. Regression parameters: r correlation coefficient (Pearson test), $R^2$ determination coefficient, DW Durbin-Watson test, model from ANOVA test, LOF lack of fit test.

**Supplementary Figure 3:** Unsupervised pattern recognition (PCA) based on sensory attributes of Torrontes Riojano wines aged under different conditions for 18 months. **A.** PCA plot at 6 months of storage. **B.** PCA plot at 12 months of storage. **C.** PCA plot at 18 months of storage.

PCA Principal Component Analysis, PC1, and PC2 are Principal Components 1 and 2, respectively. Lines are confidence ellipses (statistical significance at 95%). C-TRw Torrontes Riojano wines sealed with natural cork, SyC-TRw with coextruded synthetic cork, SC-TRw with screwcap. 15 and 25 are storage temperatures, 15°C, and 25°C, respectively. Sensory attributes: greenish hue (GH), yellowish hue (YH), fresh fruits (FF), linalool (LL), oxidized character (OX), bitterness (BT), acidic taste (AC), and aromatic intensity (AI), orange blossom (OB), chemical aromas (CA), color intensity (CI), sweetish taste (SW), floral (FL), herbaceous (HB), and brownish hue (BH).