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Posted Date: 29 November 2023

doi: 10.20944/preprints202311.1876.v1

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Article

# The Influence of the Production Process on the Natural Cork Stoppers Permeability and Its Impact on Still Wine Aging

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**Abstract:** The importance of oxygen role during aging process of wine has been long known. Many articles evaluating oxygen inlet for different wine stoppers have been published. However, none of these have studied the impact of the natural cork stoppers production process over oxygen inlet. At the moment, a standardized methodology widely accepted for measuring the oxygen transfer rate (OTR) and/or oxygen permeability of the stopper does not exist. Thus, this article is focused on the aging process (the final phase of the winemaking) and aims to test how some production process of natural cork stoppers impact on the stoppers permeability using chemiluminescence methodology and how also this affects wine ageing. In this study it has been seen that some of its processes such as the application of a surface treatment modify the permeability of the stopper. Surface plays an important role in the corks stoppers production and allows an easier insertion and extraction of the stopper into the bottle, avoiding leaks and homogenizing the stoppers. It was shown that the cork stoppers sample with surface treatment 1 had three times higher oxygen transfer rate than the cork stoppers sample with surface treatment 2 after one year of evolution. The two groups of stoppers had the same in terms of dimensions, density, visual class, batch and manufacture, the only difference between them was the surface treatment used. A red wine was corked with these two types of cork stoppers and its evolution was monitored up to 30 months of ageing. Differences were observed in the evolution of the wines both in certain oenological parameters such as the free SO<sub>2</sub>, the colour and the evolution of the polyphenols as well as at the sensory level. The wine corked with the stoppers of treatment 1 presented a faster evolution and a higher valuation in the sensory tastes of months 12th, while from the 18th month it presented an over evolution, and the score was below that of the wine with the corks of treatment 2. The data collected in this study shows that the differences observed with the chemiluminescence methodology of cork permeability test at a laboratory level are transferred to wine at the cellar. Therefore, it paves the way to standardize chemiluminescence as a methodology for permeability testing of cork stoppers. This opens a new field for the regulation of the permeability of natural cork stoppers by type of surface treatment applied.

**Keywords:** permeability; OTR; natural cork stopper; chemiluminescence

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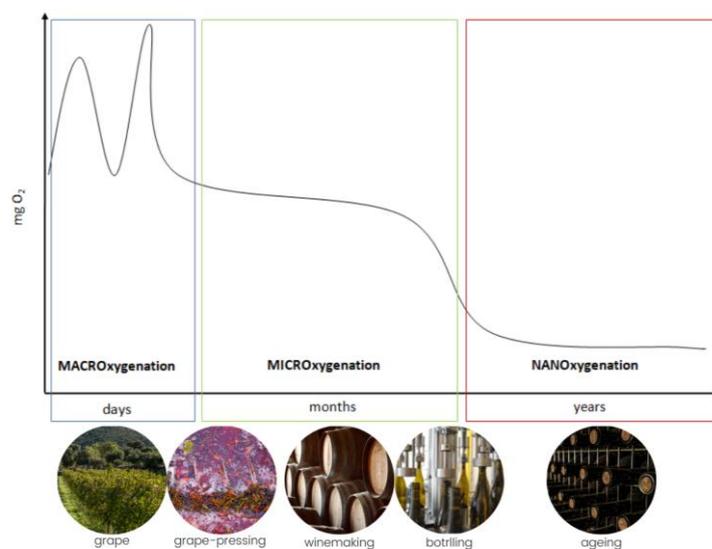
## 1. Introduction

For several centuries, cork has proved to be the most effective closure for wine, protecting its qualities and allowing it to develop and improve over time [1]. Cork is the outer bark of the oak (*Quercus suber* L.). Cork oak forests are located in the Mediterranean climate zone of Western Europe as well as in North Africa. Cork is a natural material with unique set of properties. It has attracted the attention of human since antiquity and has been used since then in various applications. Today, cork is widely known as the closure of wine bottles [2]. The cork sector in the Mediterranean area is important due to the relevance of the wine sector in the EU, where 60% of the world's wine production is concentrated [3].

In the wine market there are multiple closures types. However, these stoppers can be classified into three large families: cork or cork-based stoppers, screw caps and plastic-base stoppers. PricewaterhouseCoopers/Ecobilan quantified and compared the environmental impacts of cork stoppers versus aluminum and plastic closures on the UK market of wine [4]. According to the

researchers behind this study, when compared to aluminum and plastic closures, cork stoppers emerge as the most favorable alternative in terms of non-renewable energy consumption, greenhouse gas emissions, impact on atmospheric acidification, contribution to the formation of photochemical oxidants, influence on surface water eutrophication, and overall solid production [4]. It is clear that cork material plays a crucial role in preserving biodiversity, a key aspect of sustainable development. Additionally, it contributes to lowering greenhouse gas emissions and capturing CO<sub>2</sub>. The unique property of cork trees to regenerate after each harvest, where more than 50 percent of the bark is removed, enables them to endure such losses. The use of cork, derived from the living trees' bark, is preferred by policymakers due to its more environmentally friendly nature compared to synthetic alternatives [3,5]. Cork is recommended for bottles of reserve wines and wines that need to age in the bottle [1]. However, cork stopper is not an inert material and its permeability can lead to entry of quantity of oxygen molecules [6].

One of the most important aspects to maintain the quality of food products during the manufacturing, packaging and conservation process is the control and measurement of the oxygen permeability. Food oxidation is related to the loss of organoleptic properties and reduced durability. In the case of the wine sector, the evolution of the wine is extremely dependent on the oxygen amount the wine receives. Oxygen is crucially involved in the oenological process, from the collection of the grapes to the opening and consumption of the wine itself [7]. Oxygen determines the development of the winemaking process and defines the final sensory characteristics of the wine, among other factors Oxygen management and oxidation-reduction reactions are part of the main challenges that winemakers have to face during the production and aging of wine [8]. Pressing the grapes, the movements of the wine, filtration and bottling contribute to bringing large amounts of oxygen to the wine [5] (Figure 1). This study was focused on the final phase of the winemaking process: post-bottling and aging. Due to the extremely low rates of oxygen entry through a closure, this form of oxygen exposure has been called nano-oxygenation.



**Figure 1.** Diagram of the inlet of oxygen into the wine throughout the vinification process.

The main function of a stopper is to secure good seal to avoid any deterioration of the wine during storage and the ageing. Controlling the amount of oxygen is a critical aspect in the winemaking process from the harvest until the aging processing bottle, since oxygen can deteriorate or improve the characteristics of a wine [9]. Oxygen permeability of wine closures may vary depending on closure type and strongly influence the evolution of wines during bottling aging [10–15]. Godden et al. 2005 [16] described that different final products from the same wine could result with bottling under different closures.

Different authors studied the permeability of different types of stoppers (natural cork stoppers, agglomerated, microagglomerated or synthetic stoppers) [17–20] and others the permeability of the

natural stoppers of different qualities [21,22]. It had been determined most of the oxygen diffuses out of the cork into the bottles during the first 12 and 24 months of storage [23]. Internal research from ICSURO has also shown that a coated group of natural cork stoppers (flower class) with wax presented more oxygen inlet than the same group of stoppers without covered explaining that the oxygen that is transferred to the bottle comes from the inside of the stopper itself and not from the outside. However, the influences of the finishing operations of the production process of natural cork stoppers have not been studied.

After harvesting, the cork planks are stabilized, boiled, and stabilized again. After this resting period, the stoppers are punched and classified in graded. When the correct dimensions of the stoppers are obtained, the selected stoppers undergo to the finishing operations of the production process. The finishing operation starts with washing and disinfection stage, with a cleaning process of an aqueous solution of hydrogen peroxide most commonly [1]. Next, the stoppers are dried, and the stoppers may or may not be coloured coating. This process is not always carried out and has the purpose of put of a coloured layer on the stopper to give it a uniform colour [24]. Finally, the stoppers may be printed, and surface treated with paraffin or/and silicon to obtain the final product [3] (Figure 2).



**Figure 2.** Diagram of the process of manufacturing a natural cork stopper.

In this paper, the influence on stoppers permeability of different washing processes, different colored coatings, and different surface treatments and how also these affects wine ageing was studied. Synthetic alternatives stoppers can easily identify the OTR of the material and exists some synthetic closures into the market that report their permeability in their technical sheets. However, natural cork stoppers are not as well known as other alternative stoppers and this study aims to provide new data in the study of the permeability of natural cork stoppers that will allow them to be characterized and better understood. As has been said, cork stoppers are a natural material unlike other stoppers and therefore all research related to knowing the material and promoting its use has a direct link in the field of sustainability and the use of natural materials.

## 2. Materials and Methods

### 2.1. Cork stoppers samples

The stoppers studied were manufactured by the same batch. All of them were visual classified as flower class (the best visual class in range on classification). Seven groups of 150 natural cork

stoppers were made by: four washing processes (no wash, peroxide spray washing, peroxide immersion washing and peracetic acid spray washing); two different coloured coatings (not coated, water-based coated stoppers and solvent-based coated stoppers) and two different surface treatments (1 and 2) (Table 1). The formulations and the method of application of each process were carried out by the company according to its own production process, therefore it found under the trade secret.

**Table 1.** Study groups according to the washing process, the colored coating process and the application of surface treatment.

Washing	Colored coating	Surface treatments
No coated	Not coated	Surface treatments 1
Peroxide spray washing	Not coated	Surface treatments 1
Peroxide immersion washing	Not coated	Surface treatments 1
Peracetic acid spray washing	Not coated	Surface treatments 1
Peroxide spray washing	Water-based coated	Surface treatments 1
Peroxide spray washing	Solvent-based coated	Surface treatments 1
Peroxide spray washing	Solvent-based coated	Surface treatments 2

All closures were analysed prior to bottling for dimensions, density, and moisture (ranged from 4% to 6%) according to UNE 56921:2021 methodologies [25]. A selection of ten stoppers of each group was made according to the most homogeneous values obtained for dimensions, weight, and density.

## 2.2. Glass bottles

A set of 375 mL Extra white (colourless) "bordelaise" classic bottles with standardised bottleneck bores, 18.5 mm diameter were used. Bottles were coded to ensure traceability to the specific cork closure tested in each bottle. Bottle internal neck profiles were measured using a PerfiLab® with Kroeplin instrument (Egitron, Portugal). Measures were taken from the rim to 50 mm depth at a 1 mm pace along the bottleneck. All the bottles that were used complied with the internal diameter of the bottle according to UNE standard at 3 and 45mm [26].

## 2.3. Corking operation

A semi-automatic corker Moeller model M500 with corking jaws at 16 mm compression, was used for all closures. All bottles were bottled under controlled conditions and were sealed with different closures under a continuous flush of nitrogen (2.5 bar). An initial reading of the sensor was done in order to control degas oxygen process in the empty bottles. All bottles were left upright for 1 h after bottling and then stored horizontally over 12 months.

## 2.4. Storage conditions

Bottles were stored horizontally in darkness conditions under constant temperature of  $20 \pm 2^\circ\text{C}$  and a constant relative humidity of  $65 \pm 20\%$  over this period.

## 2.5. Monitoring oxygen ingress

Oxygen measurements were taken using chemiluminescence by an analyzer oximetric model NOMASenseO2P6000 with Fibox 3 LCD Tace V6 fiber and Pts6 sensors (Wine Quality Solutions). The system has a transmitter/receiver probe. This flow is directed to a sensor (called spot) previously placed inside a transparent bottle. The spot was placed inside the bottles and left for 24 hours in the dark before starting the test. An initial reading of the sensor was done in order to control the inserting process just after corking the empty bottles. During the first 1 hour, the empty bottles are measured every few minutes to verify that the corking process was done correctly and that there were not leaks. Each bottle has its initial oxygen value. This amount of oxygen was subtracted from the following reading in order to know the amount of incoming oxygen. The measurements were taken daily for the first 90 days and then every 15 days up to 12 months. The device expressed the result as partial

pressure of oxygen ( $PO_2$ ). Partial pressure of oxygen inside the empty bottles was converted to milligrams of oxygen by the general law of ideal gases and the molecular mass of oxygen.

### 2.6. Modelling and data analysis

All readings are records in Microsoft Excel and they were transformed from hPa to mg O<sub>2</sub> with the same program. Thanks to the Excel functions, the increments were calculated and the modalization over time for each bottle or for a pool was done. Thus, analytical data were obtained with Microsoft Excel. Analysis of variance (ANOVA), Turkey test, Kruskal Wallis and Welch's t-test for two samples were developed by R commander package for Windows of R software (ver. 3.5.3, 2019).

### 2.7. Red wine

A young red wine from the DO Empordà was used in the second part of the study. The oenological analysis of the wines was carried out by a wine specific laboratory. The analyses were carried out according to international standards. The Instituto de Ciencias de la Vid y el Vino of the Universidad de la Rioja studied the evolution of the monomeric phenolic compounds of the wines according to their internal protocols.

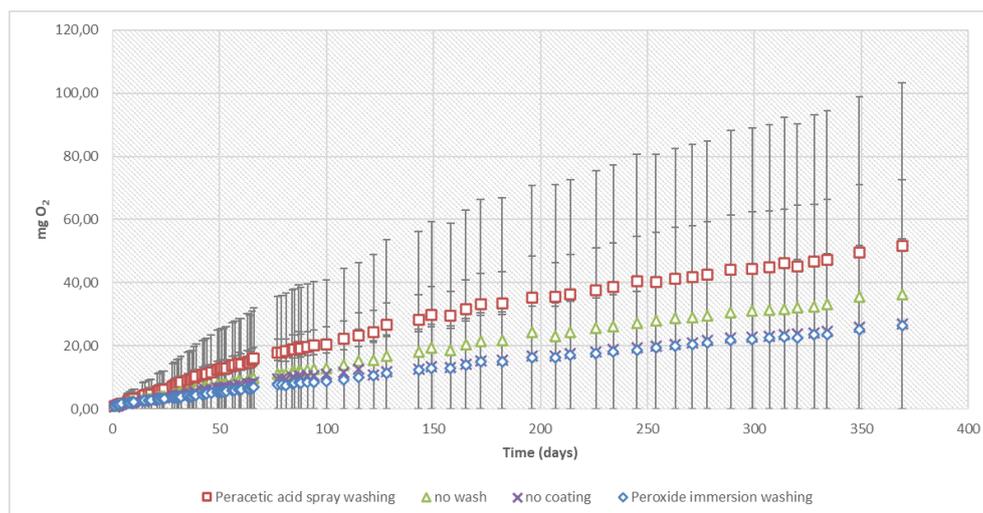
## 3. Results

Cork stopper finishing operations include a stage of washing and optionally colored coating with subsequent drying, classification into commercial quality classes and a final impression with the client's brand followed by the final surface treatment, which is normally a mixture of paraffin and silicone [2]. The influence of these three final stages on the permeability of natural cork has been studied separately. The results obtained are shown below.

### 3.1. Washing stage

The washing stage of cork stoppers plays the defining role in cleaning/disinfection surface dust or loosened lenticular material and appearance (primarily the colour homogeneity and whiteness) [27]. Nowadays the standard cork washing and bleaching is done by using a hydrogen peroxide aqueous solution (about 10% of 130vol. H<sub>2</sub>O<sub>2</sub> solution) with 1% sodium hydroxide, with a neutralization agent. Washing with other chemicals such as sodium metabisulphite, sulphamic acid or peracetic acid is more rarely and depends on client's demand [2]. Moreover, the washing process can be applied by spray washing or immersion washing. In the present study, 4 groups had followed: no wash, peroxide spray washing, peroxide immersion washing and peracetic acid spray washing (Figure 3). The stoppers studied were manufactured by the same company, from the same batch, with the same surface treatment and colourless coating (solvent-based coated). Both peroxide washes seemed to have a similar behaviour over time, presenting comparable oxygen input values. However, the groups not washed and washed by spraying with peracetic acid, presented higher values throughout the study time, being peracetic washing the group that presents the highest oxygen inlet.

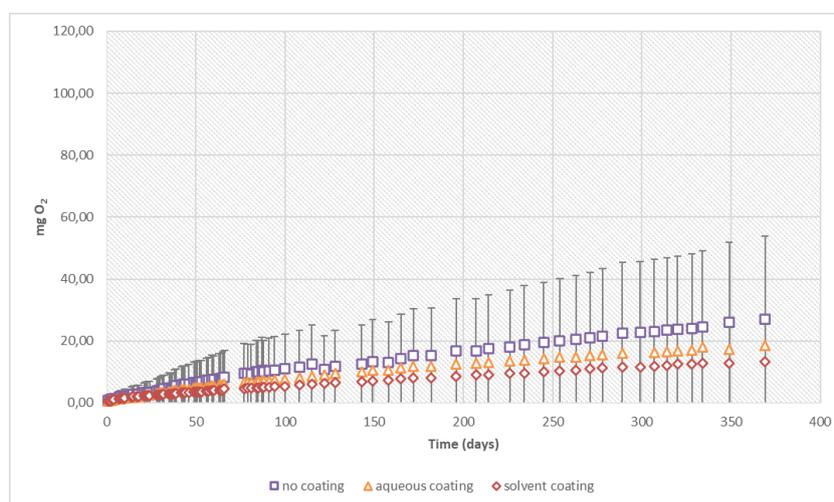
The analytical data showed significant different between the washing trials groups ( $p$  value > 0.05). The multiple comparisons of means: Tukey Contrasts presented the group washed with peroxide by immersion and washed with peracetic spray with the most different groups. The statistical pooling, therefore, gave different pools to these trials. In addition, it put no washed and peroxide immersion washing in the same pool, which in turn these two share characteristics with the other pools. To the best of our knowledge, no study has been published on the interaction or relationship that the washing process of natural cork stoppers may have with their permeability. For this reason, we believe that this study may open a new field of research. Some authors have pointed out the importance of the washing process and the alterations it can cause on the surface of the stopper [26].



**Figure 3.** Monitoring of oxygen ingress along time (dots for average, bars for standard deviation) of natural cork stoppers with different washing system: no wash, peroxide spray washing, peroxide immersion washing and peracetic acid spray washing (n=8).

### 3.2. Coloured coating stage

The colored coating is the application of a colored layer on the stopper surface to give it a uniform color [24]. This stage is not carried out routinely; it is only executed in some cases or at the request of the client. In this study, different colored coating of natural cork stoppers were compared: not coated, water-based coated stoppers and solvent-based coated stoppers (Figure 4). The stoppers studied were manufactured by the same company, from the same batch, with the same washing (peroxide spray) and the surface treatment. No differences were observed nor were there statistical differences between the three colored coating study groups in reference to its permeability.

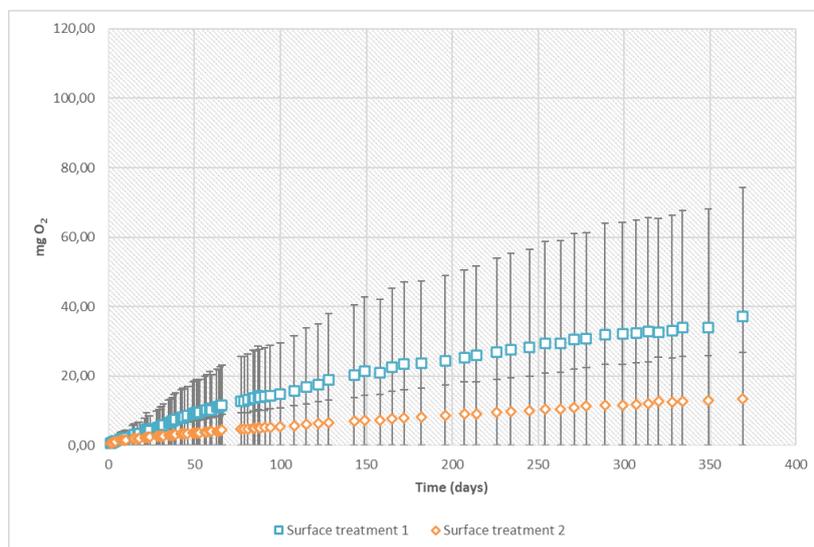


**Figure 4.** Monitoring of oxygen ingress along time (dots for average, bars for standard deviation) of natural cork stoppers with different colored coating: not coated, water-based coated stoppers and solvent-based coated stoppers (n=8).

### 3.3. Surface treatment stage

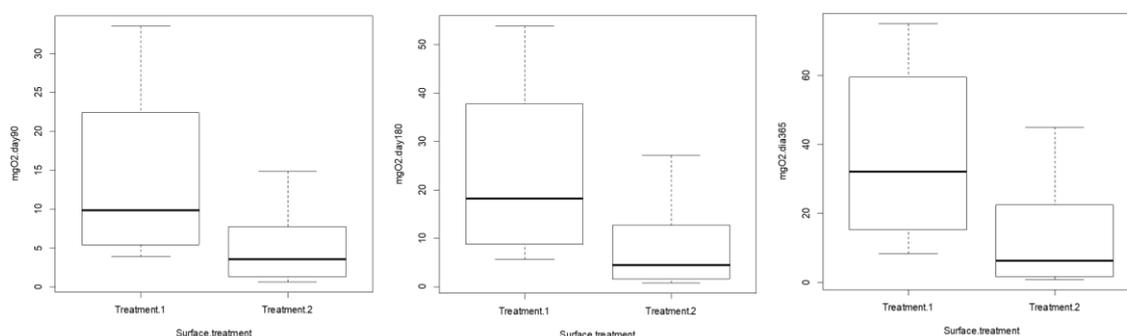
The surface treatment has the objective to coat the stopper with a lubricant film to reduce friction. It allows an easier introduction and extraction into and out the neck of the bottle [2]. Previous studies from our research centre showed that stoppers without surface treatment had higher permeability than stoppers with surface treatment (this data not shown). This fact would suggest that the surface

treatment played an important role in the transfer of oxygen, thereby acting like a kind of barrier. For this reason, two groups of natural cork stoppers with two different surface treatments (1 and 2) were chosen (Figure 5). The stoppers studied were manufactured by the same company, from the same batch, with the same washing (peroxide spray) and the same-coloured coating (solvent-based coated).



**Figure 5.** Monitoring of oxygen ingress along time (dots for average, bars for standard deviation) of natural cork stoppers with different surface treatment: surface treatment 1 and surface treatment 2. (n=8).

Surface treatment 1 presented twice inlet oxygen as treatment 2 over time. The analytical data showed statistical differences ( $p$  value  $>0.05$ ) from the day 90 from the end on the study (Figure 6). As happened in the case of the study groups with different washes, the authors are not aware that this phenomenon has been previously reported. Therefore, the results of this study show that the finishing stages of cork stoppers could modulate oxygen entry. Thus, knowing and characterizing the different stages of the industrial finishing process can help to have stoppers with different tendencies of oxygen permeability.



**Figure 6.** Box plot of the oxygen ingress of natural cork stoppers with different surface treatment along the time. (n=8).

#### 4. Discussion

Do these differences observed translate into the wine? The results obtained that have been previously presented were shared with the company that manufactured the stoppers and it was decided to extend the study for a few more months. It was chosen to continue studying the surface treatments. In this case, the company produced two groups of natural cork stoppers (superior class,

the second visual class in range on classification) from the same batch, with two different surface treatments (1 and 2) but with the same washing (peroxide spray) and the same colored coating (solvent-based coated). One hundred and twenty bottles of the same young red wine were bottled: sixty of them with cork stoppers from treatment 1 and sixty more with cork stoppers from treatment 2. The bottling process in this case was conducted in the winery with its own industrial bottling line. After bottling, 6 bottles samples were taken to analyze the initial wine. The wine bottles were aged under cellar conditions in the winery itself during the 30 months of study. During this time, 4 samplings were done to analyze some oenological parameters of the wine followed by a sensory test. The studied wine was a young red wine that is normally on the market a few months after bottling and its shelf-life usually is between 12 and 18 months. The sanitary conditions experienced throughout the years 2020 and 2021 with COVID led to the modification and expansion of the study.

An oxygen permeability analysis of this new set of stoppers was performed. The oxygen transfer rate (OTR) was showed below (Table 2). The differential trend observed in the first part of the study remained. The two surface treatments behave the same in this new batch of stoppers: surface treatment 1 presented more permeability than surface treatment 2. A clear evolution of the oenological parameters from the initial sample to the later sample points were seen in Table 3. There was a decrease in total and free sulphur dioxide, an increase in parameters related to coloring and some variations in total polyphenols, anthocyanins, and tannins. These changes were typical of the evolution in wine chemistry and of maturation itself. The red-purple colour of young red wines changes towards brick-red, which is attributed to the progressive formation of new pigments [28,29] from the initial free anthocyanins [30,31] causing the reduction of these and therefore reducing the violet notes of the wines. This evolution of the color was observed over time, being more accelerated in the bottles of treatment 1 stopper than in 2 (see months 12 and 18 of evolution). In addition, total sulphur dioxide is higher in the samples from the surface treatment 2 than the stoppers with surface treatment 1 at 30 months. The stoppers with surface treatment 2 had lower oxygen permeability, therefore it was expected that the total sulphur dioxide value would be higher since this compound can be considered an antioxidant agent.

**Table 2.** Mean of the oxygen transfer rate in mg/day by the oxo-luminescence method of the natural cork stoppers with different surface treatments: surface treatment 1 and surface treatment 2. (n=4).

	Oxygen transfer rate (mg/day)				
	1 month	2-12 months	12 months	12-24 months	24 months
Surface treatment 1	0.103±0.059	0.033±0.047	0.041±0.051	0.021±0.027	0.031±0.039
Surface treatment 2	0.054±0.020	0.007±0.007	0.012±0.007	0.005±0.006	0.008±0.006

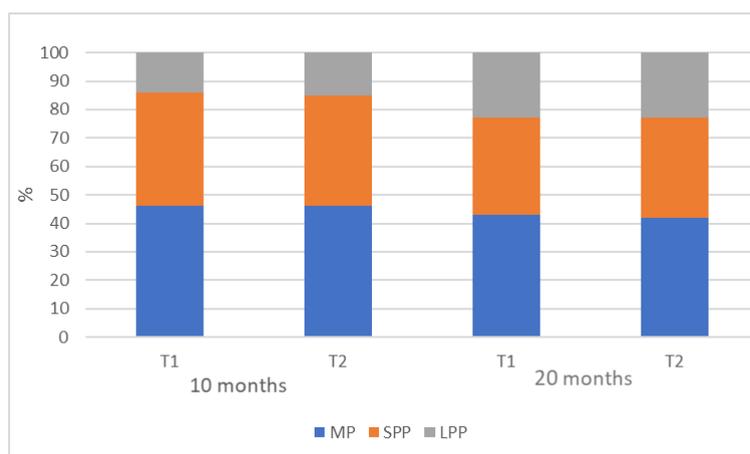
**Table 3.** Oenological parameters analyzed over time of the young red wine aged closed with the two types of natural cork stoppers with different surface treatments: surface treatment 1 and surface treatment 2.

	In	4 months		12 months		18 months		30 months	
		treatm ent 2	treatm ent 1						
SO <sub>2</sub> free (mg/L)	24,8	14,2	13,7	<10	<10	10	<10	<10	<10
SO <sub>2</sub> total (mg/L)	54,0	38,5	37,3	32,0	32,0	29,0	21,7	23,3	26,3
volatile acidity (g/L)	0,5	0,5	0,5	0,5	0,5	0,6	0,60	0,6	0,6
Absorbance <sub>420n</sub>	2,	2,717	2,700	2,740	2,770	2,850	3.083	3,417	3,253

<b>m</b>	483								
<b>Absorbance520nm</b>	2,950	3,017	3,033	3,200	3,210	3,173	3,433	3,500	3,433
<b>Absorbance620nm</b>	0,683	0,800	0,800	0,750	0,750	0,773	0,847	0,877	0,847
<b>Colour intensity</b>	6,117	6,533	6,533	6,690	6,730	6,797	7,363	7,793	7,533
<b>Colour intensity</b>	-	-	-	0,856	0,863	0,9	0,9	1,0	0,9
<b>L*</b>	-	-	-	22,60	22,6	22,0	20,0	19,3	19,9
<b>C*</b>	-	-	-	64,40	64,4	62,9	60,3	58,7	59,8
<b>h*</b>	-	-	-	34,80	34,8	34,9	33,7	33,8	34,1
<b>a*</b>	-	-	-	52,80	52,9	51,6	50,1	48,7	49,5
<b>b*</b>	-	-	-	36,70	36,7	35,9	33,5	32,6	33,5
<b>Totalpolyphenolindex (GAE)</b>	45,6	44,5	44,3	44,9	48,3	45,3	45,9	45,2	45,7
<b>Total anthocyanins (mg/L)</b>	275,2	201,6	202,2	389,0	334,0	318,0	292,0	199,0	207,7
<b>Totaltannins (g/L)</b>	2,3	1,7	1,7	1,7	1,5	2,0	2,4	2,5	2,5

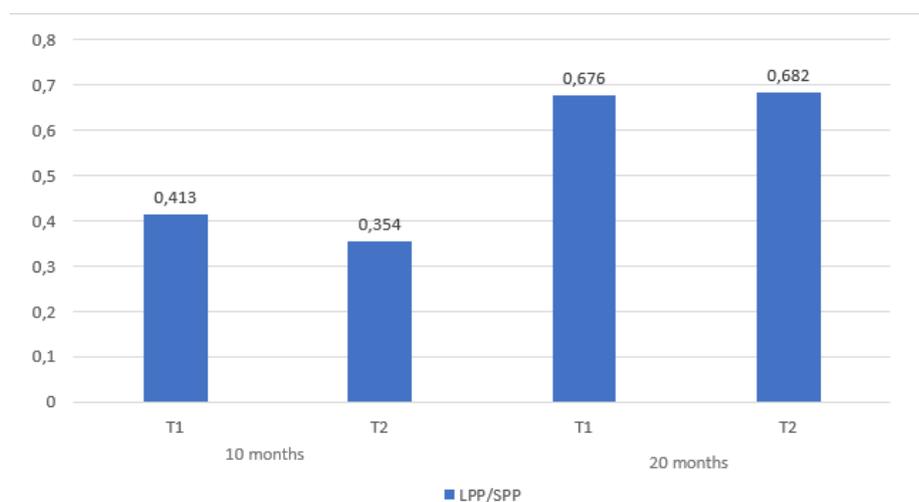
During the corresponding sensory tastings, the testing panel described that the wines corked with the stoppers with the surface treatment 1, were more developed after 12, 18 and 30 months of aging in general terms. On the other hand, the wines corked with surface treatment 2 seemed underdeveloped after 12 and 18 months and still within the shelf-life range in 30 months.

At the same time, different samples of the wines were sent to the Instituto de Ciencias de la Vid y el Vino of the Universidad de la Rioja to study the evolution of the monomeric phenolic compounds of the wines. The combination of anthocyanins and flavanols/tannins can take place by direct condensation or by formation of ethyl bridges formed from acetaldehyde (reaction favoured by the presence of oxygen). The formation of these combinations allows the change of the red-blue tones in young wines to brick-red. In general, these new pigments are more stable than the anthocyanins from which they come from, which imply that their formation increases colour stability in aged red wines. Pigments formed from anthocyanins have long been recognized as the stable form of colour in aged red wines. After a period of aging of the wine, most of the colour is due to the pigments formed from the anthocyanins from the grape [32,33]. In the studied wines, the large polymeric pigments (LPP) increase with the storage time of bottled wines, while small polymeric pigments (SPP) and monomeric pigments (MP) decrease (Figure 7). At 10 months LPP had a proportion of around 15% and SPP of 39%, being MP of 46%. At 20 months, the LPP increased to 23% and, the SPP and the non-combined MP decreased by around 34% and 42%, respectively. The percentages for each group of compounds change significantly with aging time. This fact can be explained because during aging anthocyanins, dimeric and trimer flavanols tend to decrease, since these can continue to polymerize, forming high molecular weight polymeric pigments [34], as observed with the increase in the LPPs.



**Figure 7.** Comparison of the percentage of large and small polymeric pigments (LPP and SPP) and monomeric pigments (MP) at 10 and at 20 months from corked wine with cork stoppers with surface treatment 1 (T1) and surface treatment 2 (T2).

The ratio of LPP/SPP polymeric pigments was observed to increase by a factor of 1.6 and 1.9 with time, indicating an increase in LPP relative to SPP in the aging period. After 10 months of evolution, a difference of 0.5 was observed between the wines from the different stoppers (Figure 8). This may indicate a differential evolution between the wines, since the wines from treatment 1 present a greater proportion of LPP than those from treatment 2, indicating that the pigments have evolved more.



**Figure 8.** LPP/SPP ratios in wines bottled with cork stoppers with surface treatment 1 (T1) and surface treatment 2 (T2) and aged for 10 and 20 months (1 and 2).

According to the results presented, the corks produced in the same way by the company with two surface treatments show different oxygen permeability. After one year of evolution, the sample of stoppers with surface treatment 1 presented three times more oxygen inlet than the stoppers with surface treatment 2. The surface treatment seems to be a determining factor for OTR oxygen permeability. And, what's more, the differences detected in the permeability test are translated into a differential evolution of the same wine corked with the two types of study stoppers with different surface treatment both at the sensory level and in some analytical parameters.

It should always bear in mind that the amount absorbed in the bottling stage is about 1-3mg/L and depends on the best practices in the bottling and corking process (the method of bottling, the use of inert gases, the type of machine used, the corking system, maintenance of the jaws, control of the

volume of bottling and control the head space) [35]. The amount of oxygen absorbed during bottling, and this found in the free space in a bottle (Total Oxygen Package, TPO) usually reaches a values of 1-9mg/L [36,37]. Our study is focused on ageing oxygen. Oxygen penetrates through the natural cork during the aging in bottles in an amount of 0,005-5mg/L/year [38] process and can only be understood within a context of good bottling practices, since if only this last step of the process was controlled the contribution of oxygen stoppers could be ridiculous compared to all other processes.

## 5. Conclusions

Chemiluminescence was validated as a method of measuring the oxygen permeability of natural cork stoppers, since the conclusions drawn at the laboratory level can be extrapolated to wine. Cork stopper finishing operations include a stage of washing, optionally coloured coating, and surface treatment. The influence on stoppers permeability of different of these finishing operations was studied in this paper by chemiluminescence method. The analytical data showed that the type of surface treatment and washing process has a significant effect on the permeability of cork stoppers, unlike the colouring process. To the best of our knowledge, no study has been published on the relationship that the washing process or the surface treatment of natural cork stoppers may have with their permeability. Therefore, the knowledge acquired during the project study should allow modulating the production processing benefit of the homogeneity of the natural cork stoppers.

A red wine was corked with these two types of cork stoppers and its evolution was monitored up to 30 months of ageing. Differences were observed in the evolution of the wines both in certain oenological parameters such as the free SO<sub>2</sub>, colour and evolution of the polyphenols as well as at the sensory level. The wine corked with the stoppers of treatment 1 presented a faster evolution and a higher valuation in the sensory tastes of months 12th, while from the 18th month it presented an over evolution, and the score was below that of the wine with the corks of treatment 2.

Highlight the OTR results can only be understood within the context of best bottling practices. The method of bottling, the use of inert gases, the type of machine used, the corking system, maintenance of the jaws, control of the volume of bottling and control the head space are essential processes to have a desired wine evolution into the bottle, where the oxygen of the cork also has a relevant contribution in still wine. Thus, it means that aging stage contributes very little oxygen inlet compared to the rest of the process. For this reason, it has become evident the importance of the correct bottling process by the wineries to ensure the correct conservation of their wines in bottles.

**Acknowledgments:** This research has been carried out within the framework of the Operational Groups of the European Association for Innovation (AEI)(GO OTR-56.26043.2017.3A), with the financial support of Operation 16.01.01 (Cooperation for innovation) through the Rural Development Program of Catalonia 2014-2020 with the participation of the EAFRD fund. This work was co-financed by AECORK and its companies. We express our gratitude to Manuel Serra and J. Vigas for providing the stopper in the study. We thank the winery (Vinyes dels Aspres) for agreeing to collaborate in the study. We would also like to thank our coworkers from ICSuro and all the students and scholars who helped with the laboratory work. Last but not least, we thank Marta M<sup>a</sup> Inés Dizy, Maria Purificación Fernandez y Shuyan Liu from Instituto de Ciencias de la Vid y el Vino of the Universidad de la Rioja.

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