Wines Aging

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Wine is perhaps the most ancient and popular alcoholic beverage worldwide. Winemaking practices involve careful vineyard management alongside controlled alcoholic fermentation and potential aging of the wine in barrels.

Keywords: wine aging ; bottle aging ; oxygen permeability ; wine storage ; wine aroma

1. Introduction

The aging of spirits is a historical practice carried out for millennia, which makes alcoholic beverages and intrinsic element of many human cultures. Of these, wine is one of the most ancient and relevant today in many countries [1]. Wine aging has been improved over the centuries, and with the emergence of new technologies in recent decades, new methods and techniques can be applied to shorten the time of aging, as well as increase wine quality. Wines made from black or pink grapes are generally the ones subjected to the aging process, as they are rich in anthocyanins and other phenolic compounds (PC). This reflects upon the levels of total PC of red wines being among 1–5 g/L and 0.2–0.5 g/L in white wines [2]. Hence, white wines are not commonly subjected to aging, since they are far less resistant to oxidation, excepting some sparkling white wines which are fermented in the barrel and few dry whites [3]. By and large, the most commonly aged are red dry wines [4]. Nevertheless, multiple variations to the involved processes may be found, as there exist a wide variety of tastes for each group of consumers, as well as specific methodologies and selected grapes and/or fermentative yeasts used for some types of wines [5]. The winemaking process fundamentally comprise a careful selection of grape variety, harvesting, grape pressing to obtain wine must, alcoholic fermentation, barrel aging, and bottle storage [6]. The most significant chemical changes will take place during barrel aging and ultimately bottle storage, as along the latter the whole composition of wine is altered.

Generally, barrel-aged wines are stored from 3 to 22 months or even several years. There are different kinds of aging approaches, those using oak (*Quercus* sp.) wood barrels (traditional aging) or those using other vessels made of concrete or steel alongside oak wood pieces (accelerated aging). One or other aging process is selected depending on the grape variety, wine type, and aging desired [7][8]. Nevertheless, these may be subjected to regulations. For example, the use of wood chips for many EU wines labeled with designation of origin is not allowed, whereas the International Organisation of Vine and Wine (OIV) lists specifications to their usage [9][10]. The barrel aging step is also called oxidative aging, as low quantities of oxygen come into contact with the wine. Main chemical reactions that take place are linked to the transfer of oxygen and wood compounds. This oxidation can be performed passively, by oxygen ingress through the gaps of the barrel wood staves and wood micropores, or actively, by the supplementation of oxygen in small quantities during accelerated aging (microoxygenation) [11]. During this step, the wine undergoes controlled oxidation that allows a transfer of volatile (i.e., furfurals, norisoprenoids) and non-volatile compounds (i.e., ellagitannins) from the barrel to the wine and vice versa [12]. Therefore, this stage is crucial since this is the moment in which the unique aromatic outline is developed depending of the type of wood used and time of storage [13]. The most common woods employed for aging wine are obtained from different oak species such as *Quercus alba*, *Q. robur*, or *Q. petrea* but also from other species, known to contain high contents of ellagitannins, such as *Acacia*, *Castanea,* or *Prunus* [7][14][15]. Among the multiple variability of PC found in wood some of the most relevant compounds transferred to the wine during this step are ellagitannins, hydroxybenzoic acids, and hydroxycinnamic acids [12]. Other important reactions taking place during barrel storage are the condensation of tannins and flavonols, aldehyde transference and polymerization of pigments [16]. The degree and extent of these reactions relies upon the time of storage and wood used. Additionally, the practice to "toast" or burn barrel wood or wood pieces used in aging yields further compounds like furans, vanillin (a lignin degradation product) or lactones, albeit it may also result in degradation of ellagitannins and norisoprenoids. This is reliant on the toasting degree (low, medium, high) and the oak species employed [17]. The resulting levels of PC prior to bottling will display an impact on the need bottle aging but also on the overall oxidative stability of bottled wine. Thereafter, wines will respond differently to bottle aging, exhibiting a diverse flavor and aromatic profile. In general terms, barrel aged wines have an astringent and strong flavor, that through bottle storage will evolve towards a more fruity, softer flavor as a result of further oxidation of the wine [18]. Some examples of the few white wines benefitted from aging are typically Chardonnay or Sauvignon Blanc as these grapes are considered not very aromatic; or sparkling white wines like Champagne and Cava, which are fermented on the barrel [6][19]. As such, these types of whites acquire are more complex aroma by barrel aging. Nonetheless, as white wines are accounted for lower levels of PC (i.e., anthocyanins) that are of antioxidant nature, prolonged barrel or bottle storage may pose a quality issue, but a desired process for red or rosé wines [20]. In fact, if the desired white wine is enhanced by the aroma acquired barrel storage, it may acquire ellagitannins from the contact to the wood which also contribute to increase its resistance to oxidation. Another common practice, needed for sparkling white winemaking is aging on lees, whether on the barrel or bottle [21]. Wine shelf-life is a difficult matter to assess as bottles may be stored for long periods of time before consumption, even once the bottle aging phase has been concluded in the winery. As opposed to many foods and beverages, wine generally increases its quality the longer it is preserved in the bottle. This is due to complex chemical reactions that take place in long time term. Most of these reactions are due to the passage of environmental oxygen to the wine, that induce oxidative reactions, triggering further chemical interactions between wine compounds [22]. Typically, aged wine is stored in glass bottles and closed with cork stoppers, through which the oxygen will be transferred. There are other materials that can be used as stoppers with diverse gas transference properties, e.g., thermoplastics or aluminum. These materials will determine the oxygen transfer rate (OTR). However, even though the selection of the stopper is going to provide different oxidation degrees to wine, it is not the only factor responsible for the wine aging in the bottle. The most important condition of the susceptibility of the wine towards oxidation in the bottle heavily relies on their phenolic composition. The PC content of wine depends on the grape origin, characteristics of barrel aging and aging conditions. Besides, environmental parameters of storage such as type of closure, temperature, humidity, or exposure to light are going to strongly impact in the development of the wine aging bottle [23]. This diverse resilience of wines against oxidation also determines the choice of the closure. Aged red wines will require a higher OTR in order to ensure bottle oxidation, thus the stoppers are frequently made of natural cork or cork composites. Synthetic stoppers may also be used but as they tend to have higher permeability to oxygen (excepting screw caps), they are less frequently used in bottle aging as its use may impair accelerated/premature oxidation of the wine. The reactions induced by oxygen ingress includes polymerization of pigments, condensation of tannins, formation of new aromatic compounds, and degradation of molecules that lead to undesired aromas and off-flavors. These reactions take place over time, meaning the wine does not stay chemically still through the storage. Some perceivable changes of wine by storage in the bottle include darkening of color, increased fruit flavor, lower astringent and “reductive” flavor, or softer mouthfeel [18]. Hence the bottle acts as an active aging vessel. Altogether, these chemical changes will have a positive impact on the wine qualities. However, non-optimum storage conditions, faults on the qualities of the stopper or an excessive storage time can result in the development of undesirable chemicals and in some cases, even make the wine unfit for consumption [24]. The key aspects, relevance and outcomes of bottle storage of wines will be addressed in the following sections.

2. Influence of Closure

Once the oxidative aging process has concluded, wine is placed in glass bottles of varying volume (generally containing 0.75 L of wine). The vessel/wine volume ratio is relevant, as it contributes to shape the resistance to oxidation, as well as the available gaseous phase in the bottle headspace [25]. For this matter, under the same storage conditions and time, an extended oxidation is observed in bottles of 0.375 L than in 0.75 L [24]. Although wine can be stored in plastic bottles or plastic/cardboard containers (Bag in Box), glass remains to be the main packaging material used [26][27]. As glass is a hermetic material, the passage of oxygen is only possible through the stopper. In this sense, the stopper of choice can make a difference on the transference of oxygen to the bottled wine, as the porosity of the material used directly affects this parameter [28]. As the stopper is generally gas-porous, it acts as a permeable barrier for different gases, such as alcohol or water vapors from the wine that may be dissipated out of the bottle (Figure 1). Regarding oxygen, it comes into contact with the wine along several steps in winemaking and bottling process, hence when wine is bottled it already contains dissolved oxygen. Besides, after wine has been bottled oxygen will be present in the headspace [29]. To better control the storage and aging, oxygen in the headspace can be evaporated by vacuum and replaced by an inert gas, such as nitrogen. This procedure also avoids pressure difficulties when the bottle is opened and minimize evaporation of water and alcohol [30]. The occupation of the headspace with another gas saturates gas pressure in the bottle and hinders the aging process since it results in negative tones for some wines. Nevertheless, when bottle aging is required to enhance flavor and aroma of wine, oxygen can ingress into the bottle through the stopper.

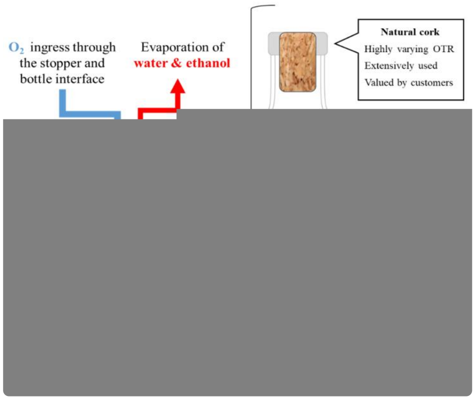

Figure 1. Ingress of oxygen to the bottle through the stopper and characteristics of stoppers.

Types of Closure

As mentioned, a great number of studies have determined that stopper characteristics greatly influence the bottle aging process and preservation of the wine [28][31][32]. Oxygen may access the bottle by two different mechanisms: By permeation and diffusion. Permeation depends on the gas pressure in the bottle, but this entry pathway can be partially countered by introducing an inert gas, like nitrogen or in the case of sparkling wine, the contained carbon dioxide. This process is less feasible to happen in isothermal conditions, that is why is so important to store wine bottles at steady temperature [33]. The diffusion pathway relies on the oxygen concentration gradient and can occur against pressure gradient. Thus, despite of measures taken to lower oxygen transference to the wine, oxygen ingress may occur anyway albeit at lowered rates that allow an extended control over the aging process [34]. Both these routes hinge on the transmission properties of the closure, hence the stopper plays a critical role on modulating oxidation of the stored wine, based on its oxygen permeability. Moreover, gaseous exchange may occur not only through the stopper, but also via the stopper–glass interface, which needs to be tightly closed [35]. In fact, this path of entry for oxygen has been found to be a major issue when stoppers loose tightness over time or inadequate storage conditions [28]. For this reason, over the years, winemakers have analyzed the performance of different closures and the physical alterations that improve their enclosing properties. The usual closure system consists of cork stoppers. However, cork may be subjected to different treatments of its structural conformation and particle size that leads it to have different permeability to oxygen. Wineries use other materials to enclose their bottles as well, like synthetic composites, screw caps made of aluminum with a thermoplastic layer, or even caps made solely of polyethylene [28]. Yet, porous stoppers remain the most used, since they allow a proper aging of the wine, while screw caps are almost airtight and greatly limit the ingress of oxygen. In turn, the very low oxygen ingress allowed by screw caps affects the wine chemical environment and yield more reductive characters [36]. In contrast, polyethylene caps are excessively porous, yielding a premature oxidation of the wine [37]. When made of permeable materials (cork, synthetics), stoppers require a mechanical compression that will additionally reduce their permeability in their contact interface with the glass, yet not the permeability of the stopper [35]. The size of the stopper is relevant too, as it determines its available surface (diameter) and filter thickness (length) [38]. A stopper size may vary from 22–24 mm of diameter to 28–46 mm of length, cork stoppers usually being the larger [35][39]. Cork is composed of suberin, lignin, cellulose, and hemicellulose along with minor quantities of tannins or waxes. Synthetic stoppers are commonly made of low density polyethylene if they are produced by molding process, either styrene–butadiene–styrene or styrene–ethylene–butylene–styrene in a molding process or rather a mixture of low density polyethylene and ethylene vinyl acetate [38]. The mechanical and chemical properties of these materials make them convenient for their use as microporous closures.

Regarding their structure, stoppers show diverse particle size that will later influence their permeability to oxygen. Cork stoppers may be extracted from cork oak bark as a single piece (natural cork), macroagglomerated particles (2–8 mm size) or microagglomerated particles (≤2 mm) jointed together as cork composites by blended with polyurethanes and isocyanates [38]. Microagglomerated stoppers are also called technical stoppers. Besides this, in the case of sparkling wines, the stopper is usually a multilayered cylinder with a central body of natural cork or macroagglomerated cork and two microagglomerated disks at each end. This configuration allows for an improved control over the gas transference from and to the wine [40]. There are several methods and measures to address the transference of gases such as diffusion coefficient or permeability. However, the most used and practical value is OTR [41] that may be calculated by physical measures of the stopper properties, i.e., inferring from their effective diffusion or rather indirectly by determining the oxygen concentration in the bottle or the degree of degradation of compounds in the wine or even apparent characteristics (i.e., yellow color by measuring absorbance at 420 nm or chemiluminescence) [34][42]. The units of OTR are usually given as mg or ml of O_2 per day, month or year [29]. This allows not only to determine the passage of oxygen, but also to easily conceive what amount may be added to the wine through bottle aging and best fits each type of wine. In turn, a more efficient selection of the stopper and closure used is possible [33]. As cork is a natural material, it is also heterogeneous and natural cork stoppers show a broad spectrum of OTR, since the microscopic structure of its cells varies greatly. Agglomerated cork, or technical stoppers have tightened range of OTR, as they are more homogeneous [43]. Yet, the OTR values may still differ, for the material permeability is still linked to the microscopic structure despite a homogenization of particle size [38]. In summary, evidence suggests that in general terms, natural cork stoppers have a varying yet good OTR that can be homogenized by microagglomeration while synthetic stoppers offer in many cases excessive OTR for long-aging wines. On the other hand, while screw caps may be a good option to preserve wine in non-optimal conditions of storage, are prone to induce the development of “reductive” characters [44]. A general overview on the OTR values of stoppers is presented in Table 1.

Table 1. Mean oxygen transfer rate of several types of stoppers *.

Type	Size Range *(Length × Diameter) (mm)*	Oxygen Transfer Rate *(mg/Year) ***	References
	49 × 24	5.5	[28]
	45 × 24	6.37	[45]
Natural cork	44.7 × 24	2.03	[46]
	45 × 24	14.25	[34]
	45 × 24	3.65	[37]
	45 × 24	2.62	[45]
	45 × 24	1.8	[47]
Agglomerated cork	Not mentioned	0.78	[48]
	45 × 24	1.68	[49]
	45 × 24	2.68	[49]
	43.6 × 23.7	2.03	[46]
	44 × 24	1.8	[29]
Technical stopper	44 × 24	1.9	[47]
	49 × 24.2	0.61	[43]
	49 × 24.2	0.38	[43]

	44.6 × 21	6.8	[46]
	44.7 × 21	20.8	[45]
Molded Synthetic	43 × 22	6.5	[47]
	Not mentioned	22.25	[42]
	Not mentioned	6.95	[42]
	43 × 22	13.65	[45]
	23 × 38	3.28	[50]
Extruded synthetic	23 × 38	6.57	[50]
	38 × 24	9.38	[37]
	38 (length)	4.34	[25]
	31.5 (diameter)	2.52	[29]
	31.5 (diameter)	1.82	[29]
Screw cap	Not mentioned	0.23	[46]
	60 × 30	0.5	[47]
	Not mentioned	0.31	[37]

* Data shown indicate measures taken on the closure alone with appropriate seals and equipment (i.e., metal tubes/rings), chemical oxygen determination methods in wine or data provided by producers. ** Oxygen transfer rate units from literature have been calculated and/or extrapolated to mg year^{-1} for practical purposes, when necessary.

Stoppers made of cork are commonly subjected to physico-chemical treatments to improve their properties and sanitize them, preventing the transference of undesired compounds to the bottled wine. The foremost method, used at industrial scale, is CO_2 supercritical treatment. It has proven to be very successful to preserve the wine without negatively affecting permeability of cork stoppers [51]. On the other hand, stopper surface treatments are done for various purposes, like ease the extraction of the stopper or avoid liquid leakage. On top of that, surface coatings have also been found to lower the oxygen diffusion through the stopper–glass interface of the bottleneck [43]. Such surface treatments are carried on cork stoppers and are commonly made with paraffin waxes or silicon [52]. Another way to lower gas permeability is to cover the closure with a metallic or plastic layer (capsule) over the stopper, while this operation is also done to protect the stopper during handling and transportation [6][53]. Nevertheless, encapsulation of the closure has proven to be an effective measure to limit excessive oxidation and preserve wines from undesired aromas (i.e., haloanisoles), extending their shelf-life [54].

The contact with the wine and environmental moisture can affect the permeability of cork to oxygen, which is a common feature of filters. Humidity retained in the cork pores affects its mechanical properties, which in turn, alter the permeability. Yet, the absorption capabilities of the stopper are heavily reliant on temperature [55]. Synthetic stoppers, like those made from expanded polyethylene, generally show a higher permeability in comparison with cork. This is a widely known fact, extensively reported in scientific literature but more pronounced in long periods of storage [38]. For example, a study carried out by Silva et al. measuring oxidation of wines after 2 years of storage found that wines enclosed with synthetic stoppers showed greatly higher levels of oxidative markers in comparison to stoppers made of cork [56]. Extruded synthetic stoppers are reported to be more permeable to oxygen in comparison to natural or technical cork, showing more oxidative characters when compared to cork stoppers in the same aging time [31]. Moreover, synthetic stoppers tend to harden over time, loosing tight in the stopper–glass interface, which may result in a premature oxidation [57]. Still, synthetic stoppers could be valuable for young wines or those simply needing short aging periods. Conversely, albeit screw caps frequently contribute to the development of "reductive" aromas, also heavily minimize oxidative degradation of the wine [47]. This can be of interest for wines more sensible to oxidation and expected to be consumed in a short period after bottling, as is the general case of white wines.

Although synthetic materials or alternatives to cork offer some benefits like their affordability and absence of off-flavor compounds; natural cork closures remain the most popular for their presence is considered a quality feature among consumers, whereas synthetic stoppers are generally associated with "cheaper" or "lesser" wines [58]. The higher permeability to oxygen that synthetic stoppers display over time also tends to make them less preferred. Yet, it should be considered that many researchers in the field consider that role and influence of many parameters and materials not fully determined, as in the case of stoppers [28][38]. That explains why many successful wineries still face unpredicted issues in their products such as faults and taints that would be more easily controlled in other foods and beverages [59]. Nevertheless, there is an ever-growing interest on defining key winemaking parameters in order to refine and hold more control over the final product quality. For this matter, research in this field has sprouted in recent years.

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