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A review on understanding oxygen mass transfer in wine: influences, measurement methods, and implications

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ABSTRACT

The concentration of dissolved oxygen in wine is a critical parameter that must be controlled during the winemaking process to preserve quality post-packaging. However, oxygen uptake occurs at multiple stages, including production, stabilisation, and packaging. Conventional oxygen removal methods, such as boiling at atmospheric pressure and membrane contactors, are either detrimental to wine quality or cost-prohibitive. Consequently, sparging with an inert gas using a bubble column or an in-line sparger remains the primary approach for oxygen removal. While previous research has focused mainly on oxygen absorption, insights into desorption can be obtained from studies on gas–liquid mass transfer in wine and model solutions. Oxygen desorption is quantified by the volumetric mass transfer coefficient (k_La), which is influenced by factors such as viscosity, dissolved carbon dioxide, concentrations of ethanol, phenolic compounds (mainly in red wines), acids, and surfactants. Herein, we provide a comprehensive analysis of how variations in these parameters affect k_La. Additionally, the effects of hydrodynamics, design parameters, and operating conditions on k_La are investigated and presented in this comprehensive review.

KEYWORDS: oxygen mass transfer, wine, nitrogen sparging, desorption, mass transfer coefficient

INTRODUCTION

Oxygen exposure to must and wine occurs throughout the winemaking process and affects wine quality and stability. At the point of packaging, the dissolved oxygen in wine is the cumulative result of oxygen absorption during various winemaking stages, minus the oxygen consumed through oxidation reactions and by yeast (Ugliano, 2013). The steps that result in oxygen pickup include racking, pumping, cold stabilisation, filtration, and packaging (Steiner, 2013). Cold stabilisation and refrigeration are two significant steps that result in increased oxygen uptake due to higher oxygen solubility at lower temperatures (Coetzee & Du Toit, 2015). The saturation concentration is the maximum dissolved oxygen concentration that wine can contain at specific conditions.

At the end of wine processing, the dissolved oxygen concentration in wine ranges from 1 to 8.5 mg/L (Coetzee & Du Toit, 2015; Ugliano, 2013). While earlier recommendations of dissolved oxygen (DO) thresholds before packaging were based on non-peer-reviewed articles such as Steiner (2013), more recent peer-reviewed publications have maintained these levels. The modern recommendation is that the level of dissolved oxygen in packaged red wine should be less than 1.25 mg/L, and below 0.6 mg/L for white and rosé wines, respectively (del Barrio-Galán *et al.*, 2023). However, these levels can vary depending on the winemaker's tactics, the style of wine, and the winery's technology to implement practices limiting oxygen intake. In a study by Letaief (2016), an inspection of 18 wineries' bottling operations found a significant range: while some wineries had DO readings between 0.2 mg/L, others were over 1.5 mg/L and potentially impact wine quality. When DO concentrations exceed the levels recommended, they have to be lowered prior to packaging to preserve the chemical and sensory qualities of wine, as too high DO levels at bottling can lower certain fruit flavours and quality (Morozova *et al.*, 2014) and oxidised aroma (Dimkou, 2013).

During fermentation, a certain amount of oxygen is required to ensure complete fermentation (Zoecklein *et al.*, 1999). Dissolved oxygen is not inert in wine, and excess amounts of dissolved oxygen can result in oxidation reactions, causing browning and loss of fresh and fruity aromas in white wines (Coetzee & Du Toit, 2015; Saa *et al.*, 2013). It is suggested that too little oxygen exposure within the wine can result in reductive off-odours (Lopes *et al.*, 2009). In red wines, regulated oxygen exposure during maturation can enhance colour stability, promote phenolic polymerisation and reduce astringency (Lopes *et al.*, 2009; Singleton *et al.*, 1979). However, following maturation, excessive uptake of oxygen (particularly during bottling) may result in undesirable oxidative reactions that affect wine quality during bottle ageing. Therefore, control of dissolved oxygen level before packaging is important to preserve the sensory and chemical integrity of the wine.

1. Methods for the removal of dissolved oxygen

The primary method for removing dissolved oxygen from wine is sparging with an inert gas through a bubble column or an in-line sparger (Cant, 1960; Vidal & Moutounet, 2008). Other methods for removing dissolved oxygen include boiling at atmospheric or reduced pressure. These methods potentially negatively impact wine quality and can be energy intensive (Butler *et al.*, 1994).

Membrane contactors have been used in the wine industry for oxygen management since the early 2000s, offering a sophisticated alternative to traditional sparging techniques (El Rayess & Mietton-Peuchot, 2016). These systems employ hydrophobic microporous membranes, typically made of polypropylene or polyvinylidene fluoride (PVDF), to facilitate selective gas–liquid exchange, effectively controlling dissolved oxygen levels while preserving volatile aromatic compounds critical to wine quality (Oro *et al.*, 2025). This is particularly advantageous for premium wines, where aroma retention is paramount, unlike sparging, which can possibly strip these compounds due to gas bubbling (Zoecklein *et al.*, 1999). However, membrane contactors require significant capital investment and regular maintenance to mitigate membrane fouling, which can increase operational costs and complexity (Oro *et al.*, 2025). Recent advancements highlight their role in dealcoholisation and precise gas management, though challenges like fouling and cost remain barriers to broader adoption (Oro *et al.*, 2025; Ramirez & Selzer, 2024). Further research is needed to optimise membrane materials and cleaning protocols to enhance cost-effectiveness and scalability across diverse winemaking contexts.

An alternative is the use of a sparged gas to strip the oxygen out of the solution. Several gases can be used for sparging, with nitrogen, carbon dioxide, and argon being the most used because of availability and inertness (Zoecklein *et al.*, 1999). Sparging with carbon dioxide can alter the sensory characteristics of the wine, making it taste spritzy or fizzy (Ough, 2018). Nitrogen and argon are less soluble in wine than carbon dioxide and do not have this effect. Unfortunately, nitrogen and argon can reduce dissolved carbon dioxide levels and affect the wine's taste and astringency (Gawel *et al.*, 2020). To avoid excess loss of carbon dioxide, wineries often use a blend of nitrogen and carbon dioxide or re-sparge the wine with carbon dioxide after nitrogen sparging (Zoecklein *et al.*, 1999). The optimal range of carbon dioxide in still wine is between 500 and 1800 mg/L for white wine and between 500 and 1000 mg/L for red wine (Gawel *et al.*, 2020).

The most widely used methods for oxygen removal in wine are in-line sparging or sparging through a vessel. Bubble vessels/columns are considered particularly effective methods for oxygen removal due to their high height-to-diameter ratio, which allows for greater gas–liquid contact time (Besagni *et al.*, 2018). The advantages of the bubble column are that they typically have excellent mass transfer characteristics (Kantarci *et al.*, 2005). Small gas bubbles

enter the column or in-line sparger through the sparger/sinter and flow directly into a stream of wine. The efficiency of a bubble column versus an in-line sparger depends on numerous factors. Cant (1960) found that bubble columns reduced dissolved oxygen more effectively than in-line spargers for oxygen-saturated wine, though both methods were similarly effective for wine with lower oxygen levels. However, the study results are not necessarily indicative of one unit being more effective than the other, as numerous unaccounted-for factors will affect the efficiency of each process. The process behind each unit is the same, facilitating liquid–gas mass transfer through the contact of an inert gas with wine.

A basic diagram of a bubble column and an in-line sparger is shown in Figure 1 below.

Currently, either of these two units is used in winemaking. As the dissolved oxygen level is an important parameter that requires controlling, it is helpful to understand the mechanics behind sparging with an inert gas.

Wineries use various techniques to estimate nitrogen requirements for sparging operations, a critical process for reducing dissolved oxygen (DO) levels to prevent oxidation and maintain wine quality. The volume of nitrogen or mixed gas (N_2/CO_2) required depends on factors such as operating temperature, equipment type, pressure, gas flow rate, and wine composition (*e.g.*, alcohol content, pH, and phenolic profile) (Walls *et al.*, 2022; del Barrio-Galan *et al.*, 2023; Nordestgaard, 2017). Figure 2 illustrates this dependency, showing the total gas volume needed to lower DO from 3 mg/L to below 0.5 mg/L under varying conditions. Equipment type significantly affects efficiency: a diffusion stone, a porous material creating fine bubbles, improves gas–liquid contact and reduces nitrogen use (*e.g.*, ~1 mL/L at 120 mL N_2/L wine flow rate), whereas no diffusion stone relies on larger bubbles from direct gas injection, requiring more gas (*e.g.*, ~7 mL/L) due to less efficient oxygen displacement (Walls *et al.*, 2022). Gas flow rates (*e.g.*, 120–280 mL N_2/L wine) and the use of mixed gases (N_2/CO_2) further influence outcomes, with higher flow rates and mixed gases increasing gas consumption (del Barrio-Galan *et al.*, 2023). Studies report nitrogen use ranging from 0.09 to 3.41 L/L wine, depending on whether a diffusion stone is used, the tank size, and initial DO, highlighting the need for optimised equipment selection (del Barrio-Galan *et al.*, 2023; Walls *et al.*, 2022). Understanding these variables is essential for achieving target DO levels (*e.g.*, <0.6 mg/L for whites, <1.25 mg/L for reds) and improving cost-effectiveness.

2. Desorption

The mechanism by which nitrogen removes dissolved oxygen from wine relies on liquid–gas mass transfer, a process governed by the principles of gas solubility and diffusion. Absorption is the transfer of a gaseous solute (*e.g.*, oxygen) from the gas phase into the liquid phase, driven by a concentration gradient where the partial pressure of the gas exceeds its equilibrium value in the liquid, as described by Henry's law (Elhajj *et al.*, 2014; Sander, 2015). Desorption, conversely, is the release of dissolved gaseous compounds (*e.g.*, oxygen) from the liquid phase into the gas phase, facilitated when the liquid-phase concentration exceeds the equilibrium solubility determined by the gas phase's partial pressure.

The driving force for mass transfer in both absorption and desorption is the difference in chemical potential or concentration between the gas and liquid phases, with nitrogen sparging reducing the partial pressure of oxygen to promote desorption of dissolved oxygen from the wine. Equilibrium is reached when the net flux of gas molecules between phases is zero, corresponding to equal rates of transfer in both

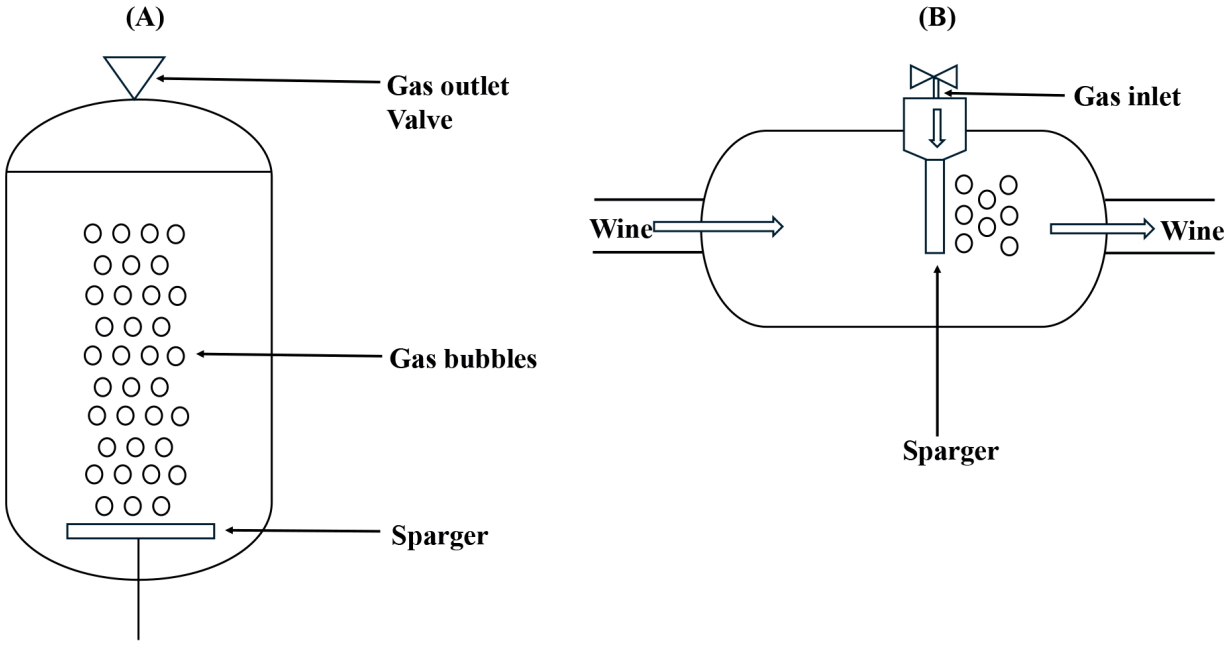


FIGURE 1. Basic diagram of a bubble column (A) and an in-line sparger (B).

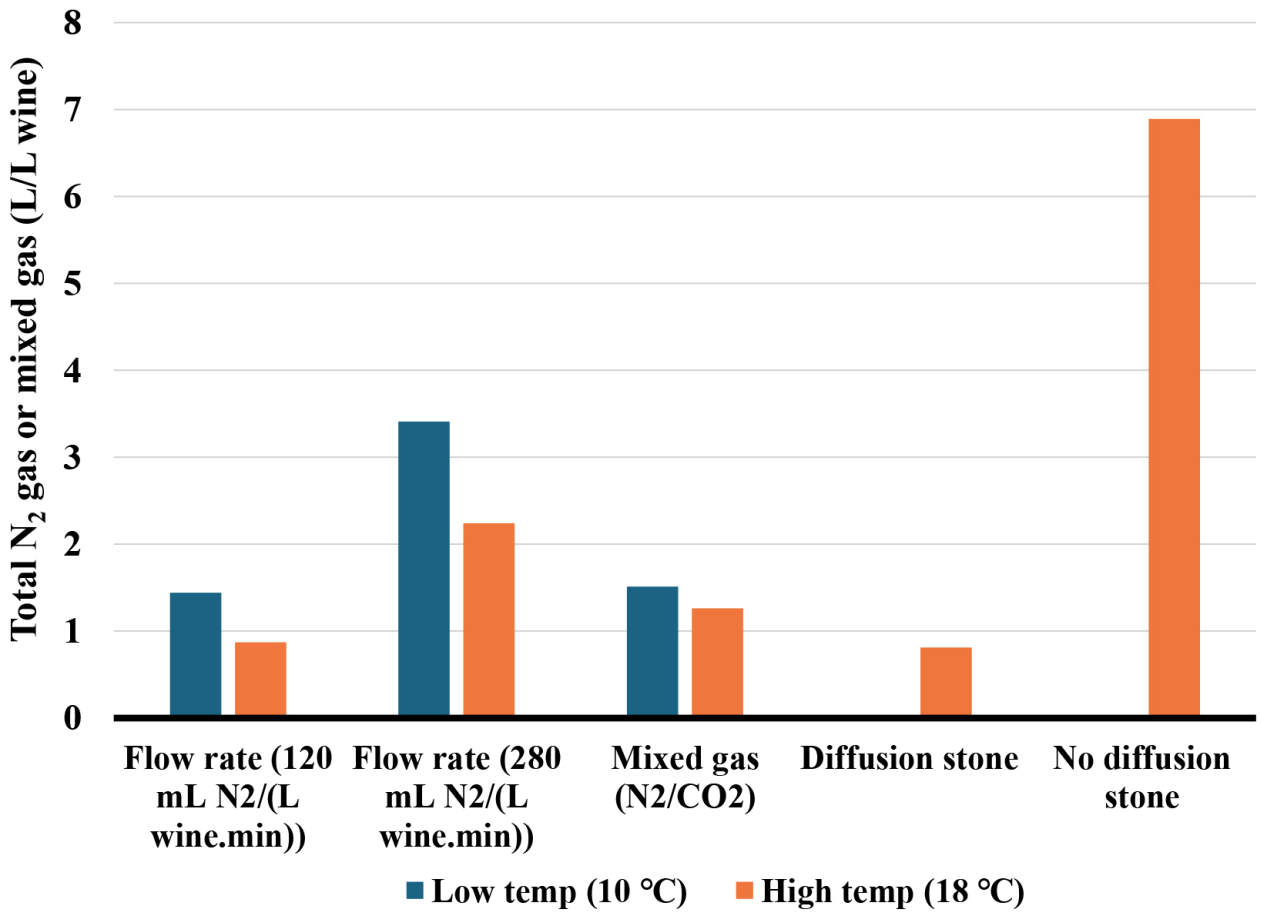


FIGURE 2. The total volume of nitrogen or mixed gas (N_2/CO_2) required in each experiment to lower the oxygen concentration of wine from 3 mg/L to below 0.5 mg/L. Data taken from Walls *et al.* (2022).

directions, determined by the system's temperature, pressure, and the liquid's composition (Sander, 2015; Garcia-Ochoa & Gomez, 2009). In air-exposed wine, the equilibrium dissolved oxygen concentration aligns with the oxygen saturation level (approximately 6-8 mg/L at 20 °C and 1 atm, depending on wine composition), which nitrogen sparging aims to lower (Zoecklein *et al.*, 1999).

Figure 3 illustrates the basic desorption process, showing oxygen transferring from a bulk liquid into a nitrogen bubble, which forms the basis of nitrogen sparging in wine.

Aside from the difference in partial pressures of oxygen in the liquid and gas, another critical parameter in understanding liquid–gas mass transfer is the volumetric mass transfer coefficient (k_La). This composite parameter describes the resistance experienced by a compound as it moves through the boundary layers between the liquid and gas phases (Garcia-Ochoa & Gomez, 2009). Understanding this coefficient is important for understanding liquid–gas mass transfer processes.

The literature explicitly examining gas–liquid mass transfer mechanics in wine (particularly the volumetric mass transfer coefficient) has primarily focused on enhancing micro-oxygenation during fermentation (Chiciuc *et al.*, 2010; Devatine, Chiciuc, and Mietton-Peuchot, 2011; Devatine *et al.*, 2007; Moenne *et al.*, 2014; Saa *et al.*, 2013). While several studies have investigated the addition and, to a lesser extent, the removal of oxygen in various aqueous mixtures, these studies provide valuable insights into how changes in liquid and gas properties influence mass transfer. They also illustrate methods for assessing and quantifying mass transfer in bubble columns, with comprehensive reviews available in the works of Kantarci *et al.* (2005), Manjrekar (2016), and Besagni *et al.* (2018).

3. Two-film theory and the mass transfer equation

Two-film theory is the most widely used model to describe liquid–gas mass transfer phenomena (Hamborg *et al.*, 2010; Pittoors *et al.*, 2014). While absorption is the most studied liquid–gas mass transfer process, desorption can also be effectively modelled using the two-film theory, similar to absorption (Hamborg *et al.*, 2010).

In the two-film theory, the "films" refer to the gas and liquid films on either side of the gas–liquid interface, as indicated in Figure 3. Beyond the gas film is the bulk gas phase, and beyond the liquid film lies the bulk liquid phase. The edge of each film is known as the boundary layer, and the liquid and gas films control the transport of molecules across the interface (Pittoors *et al.*, 2014).

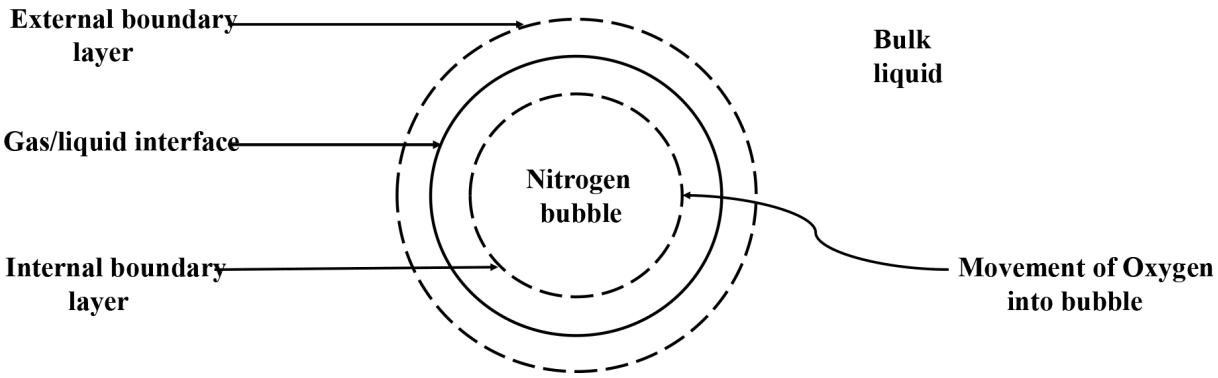


FIGURE 3. Diagram demonstrating the desorption of oxygen into a nitrogen bubble.

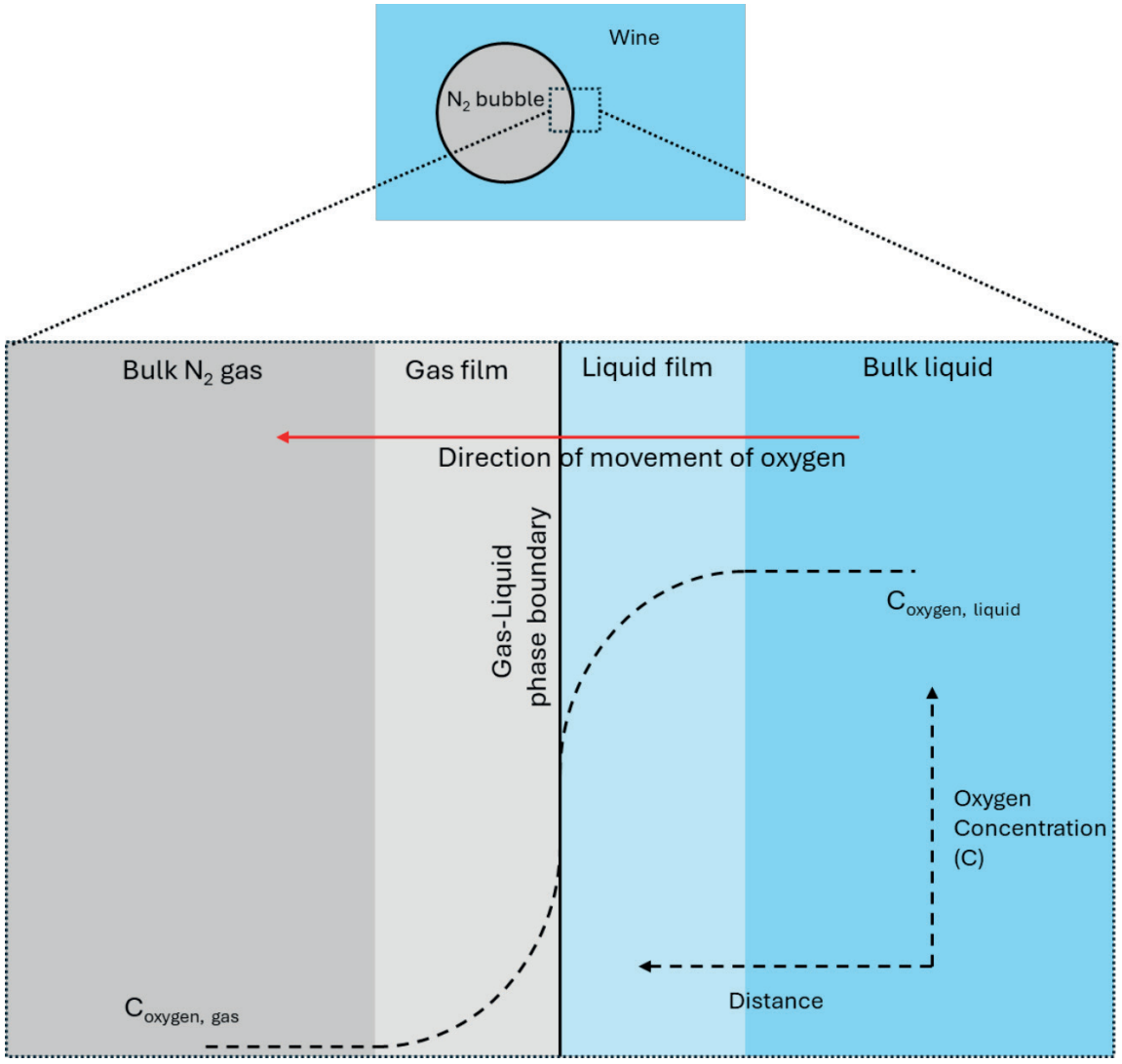


FIGURE 4. Diagram illustrating two film theories as applied to oxygen desorption.

If, during mass transfer, the compound transferring from one phase to another were instantly mixed into the bulk of that phase, no film would be present. However, due to slow mixing, a concentration gradient forms between the interface and the edge of the film. The edge of the film is the point where the concentration of the compound is the same as the concentration of that compound within the bulk phase. The slower the movement of the compound from the interface to the bulk phase, the more significant the concentration difference between the two, leading to increased resistance to mass transfer (Wiesmann *et al.*, 2006). Figure 4 below shows how oxygen concentration varies across the films during a typical desorption process, with the film's end marked by flattening of the concentration.

Mass transfer resistance may lie on either side of the interface, but for low-solubility gases like nitrogen or oxygen, resistance is primarily located in the liquid phase due to the high value of the Henry's law constant.

The mass transfer flux J ($mol.m^{-2}.s^{-1}$) can then be expressed through Equation 1:

$$J = k_L\,(C^* - C_L) \qquad (1)$$

where C* ($mol.m^{-3}$) is the equilibrium dissolved oxygen concentration, C_L ($mol.m^{-3}$) is the bulk liquid concentration, and k_L ($m.s^{-1}$) is the liquid-side mass transfer coefficient.

To account for interfacial area effects, the volumetric mass transfer rate is given by:

$$OTR = \frac{dC_{oxygen}}{dt} = k_L a\,(C^* - C_L) \qquad (2)$$

where OTR ($mol.m^{-3}.s^{-1}$) is oxygen transfer rate and ($m^2.m^{-3}$) is the specific interfacial area between gas and liquid, $k_L a$ (s^{-1}).

In desorption scenarios, when an inert gas such as nitrogen is used, the equilibrium concentration C* approaches zero, and Equation (2) simplifies to:

$$OTR = \frac{dC_{oxygen}}{dt} = k_L a\,(-C_L) \qquad (3)$$

Assuming first-order kinetics and isothermal conditions, integrating the rate expression gives: Absorption:

$$\ln\left(1 - \frac{C_{L,t}}{C^*}\right) = -k_L a.t \qquad (4)$$

Desorption:

$$\ln\left(\frac{C_{L,t}}{C_{L,O}}\right) = -k_L a.t \qquad (5)$$

where $C_{L,t}$ ($mol.m^{-3}$) is the concentration of oxygen in the liquid at time t (s), and $C_{L,0}$ ($mol.m^{-3}$) is the initial concentration of dissolved oxygen in the liquid.

The two-film theory offers an effective method for determining the volumetric mass transfer coefficient by measuring the change in dissolved oxygen concentration over time. Accurate measurement of $k_L a$ across different systems and conditions allows for evaluating factors that may influence $k_L a$.

4. Effect of sparging on wine composition

Changes in the chemical composition of wine due to sparging to remove dissolved oxygen are a concern for wine producers. Gases such as nitrogen and carbon dioxide used in sparging can interact with different wine components, thereby altering the wine's taste, aroma, stability, and chemical composition (Girardon, 2019; del Barrio-Galan *et al.*, 2023).

Walls *et al.* (2022) also reported that sparging with nitrogen gas at high flow rates resulted in high losses of CO_2, thereby altering the wine's mouthfeel and general sensory balance. Winemakers now consider sparging with mixed gases (N_2/CO_2) to maintain CO_2 at optimal levels while removing oxygen (Walls *et al.*, 2022; del Barrio-Galan *et al.*, 2023). The mixed gas balancing helps preserve the wine's sensory profile and structure.

Wine acidity and pH can also be affected by sparging. Del Barrio-Galan *et al.* (2023) report that sparging with CO_2 to remove dissolved oxygen increased total acidity by 2 % in wines. The interaction between CO_2 and other dissolved gases with acids in the wine possibly contributes to this.

Conflicting results have been reported in the literature regarding the effects of sparging on the volatile composition of wine. Recent studies have shown that nitrogen sparging does not remove significant amounts of aromatic compounds in wines (Walls, 2020). This study by Walls (2020) was performed on a Chenin blanc and a Sauvignon blanc wine, in which sparging was performed for an hour from a stone sparger. However, this might not apply entirely to all types of wine. However, del Barrio-Galan *et al.* (2023) reported that compounds such as hexanoate and ethyl octanoate in white wines decreased during sparging, and these are the majority compounds within the ethyl esters and red wines. Sparging also had a negative effect on aromatic compounds of interest, such as alcohol acetates and terpenes. These compounds are vital for the wine's aroma and flavour profile in red and white wines (del Barrio-Galan *et al.*, 2023). High sparging rates or extended sparging times may result in the loss of some of these compounds, particularly those with low molecular weight, which are more prone to volatilisation. Therefore, optimising sparging conditions to minimise or avoid such effects is crucial. These conflicting results might be due to differences in sparging regimes, such as gas flow rate, wine temperature, and sinter used, while differences in the wine matrix might also play a role. However, it must be stated that, according to our knowledge, sound sensory studies on the effects of sparging on the aromatic characteristics of wine have not been performed yet.

FACTORS THAT AFFECT DESORPTION

Several factors affect the volumetric mass transfer coefficient ($k_L a$) and components. The volumetric mass transfer coefficient is a composite parameter consisting of the mass transfer coefficient (k_L) and the interfacial area (a). The interfacial area measures the total area of gas exposed to the liquid per unit volume of gas in the liquid at any moment. As gas bubbles rise through the column, they may coalesce, break up (if encountering an obstacle such as an impeller), or move in a different direction, and all these changes will affect the total interfacial area.

The mass transfer coefficient (k_L) measures the rate at which a compound diffuses across the gas–liquid interface. For example, when dissolved oxygen encounters a nitrogen gas bubble, the concentration gradient drives the oxygen molecule to diffuse into the gas phase. The outer bubble film acts as a barrier to diffusion, making k_L a critical parameter for evaluating this resistance. Many studies examining gas–liquid mass transfer focus solely on measuring the $k_L a$. Understanding the factors that affect the components of the $k_L a$ allows for a better understanding of the mechanics of mass transfer in gas–liquid systems.

The factors affecting the volumetric mass transfer coefficient can be categorised into physico-chemical, operating, design, and hydrodynamic factors. Notably, hydrodynamic factors are significantly influenced by the other three categories (Garcia-Ochoa & Gomez, 2009). This section will explore how each of these factors impacts k_La.

1. Physico-chemical factors

Physico-chemical properties such as liquid viscosity, liquid phase diffusivity, liquid and gas density, and surface tension have been shown to affect k_La (Akita & Yoshida, 1973). These properties can influence either or both k_L and a. It has been widely demonstrated that increasing viscosity results in a decrease in k_La, primarily due to a reduction in gas–liquid interfacial areas (Besagni *et al.*, 2018; Kantarci *et al.*, 2005). Viscosity also has a dual effect on bubble size. Higher viscosities cause bubbles to detach later from the sparger base when larger, leading to reduced interfacial areas (Besagni *et al.*, 2018). When viscosity increases significantly, system turbulence increases, leading to more bubble coalescence. However, if viscosity remains below a certain threshold, the rising bubbles stabilise, reducing coalescence and increasing boundary layer thickness (Fukuma *et al.*, 1987). Ruzicka *et al.* (2003) estimated that viscosities between 1 and 3 mPa·s stabilise bubble flow (reducing coalescence), while viscosities between 3 and 22 mPa·s enhance turbulence, increasing coalescence. Wine viscosity is influenced mainly by the alcohol content, with 1-1.7 mPa·s being reported for an alcohol content ranging from close to 0 to 14 % *v/v* (Pickering *et al.*, 1998); thus, coalescence is not the major contributing factor for wine sparging.

The volumetric mass transfer coefficient (k_La) in nitrogen sparging for oxygen desorption from wine is influenced by several physicochemical properties, with effects varying depending on wine type. Viscosity indirectly affects k_L by altering liquid diffusivity; higher viscosity reduces the rate of molecular movement across the gas–liquid boundary layer, lowering both k_L and k_La (Song *et al.*, 2014). In winemaking, viscosity differs across wine types: red wines, with higher tannin and polysaccharide content, exhibit greater viscosity (*e.g.*, 1.5–2.0 mPa·s) than white wines (1.0–1.5 mPa·s), potentially reducing k_La more significantly in reds during sparging (Jackson, 2014). Gas density, influenced by gas type (*e.g.*, nitrogen, CO_2) and system pressure (related to liquid height), generally increases k_La with higher density (Jordan & Schumpe, 2001; Kantarci *et al.*, 2005). This effect is more pronounced in sparkling wines, where CO_2 addition increases gas density, enhancing oxygen removal efficiency compared to still wines using pure nitrogen. Lower surface tension typically produces smaller bubbles, increasing the interfacial area and thus k_La (Matsunaga *et al.*, 2009). White wines, with lower ethanol and phenolic content, often have higher surface tension than red wines, leading to larger bubbles and reduced k_La unless adjusted with surfactants or fine diffusion stones (Zoecklein *et al.*, 1999).

Numerous correlations for estimating k_La highlight the impact of these physicochemical factors, though they are often system-specific (Akita & Yoshida, 1973). Liquid diffusivity, viscosity, surface tension, and density are integrated into a broad correlation by Akita and Yoshida (1973), while gas density is emphasised in models by Jordan and Schumpe (2001) and Matsunaga *et al.* (2009). Kantarci *et al.* (2005) provide a comprehensive review of bubble column correlations, highlighting their dependence on liquid properties. For wine, these variations are critical: higher sugar content in sweet wines (*e.g.*, dessert wines) might increase viscosity and density, further lowering k_La, whereas low-sugar, high-alcohol wines (*e.g.*, certain fortified wines) may enhance diffusivity, increasing k_La. Thus, the physicochemical properties of wine, modulated by type and composition, significantly influence oxygen desorption k_La, necessitating type-specific optimisation of sparging conditions.

2. Hydrodynamic conditions

Another consideration for determining the mass transfer characteristics within a vessel is the range of hydrodynamic conditions that occur within a vessel (Besagni *et al.*, 2018). These conditions define the interactions between gas and liquid within the system and are important in understanding liquid–gas mass transfer. The way gas bubbles interact with each other is fundamental. A common approach to evaluating hydrodynamic conditions within a vessel is to classify these conditions into flow regimes (Kantarci *et al.*, 2005).

Hydrodynamic conditions are typically classified into two or three flow regimes. Manjrekar (2016) identifies three flow regimes: homogeneous, transition, and heterogeneous. Similarly, Kantarci *et al.* (2005) describe three regimes: bubbly flow (homogeneous), turbulent flow (heterogeneous), and slug flow. Factors affecting the vessel's flow regime are superficial gas velocity, sparger configuration, liquid and gas phase properties, operating pressure, and column dimensions (Besagni *et al.*, 2018). Understanding which flow regime is present in a system is important, as results for one system may only be valid for another system if the flow regime occurring within both is the same.

The superficial gas velocity is a function of the gas flow rate and the vessel's cross-sectional area. The superficial gas velocity significantly affects the type of regime present. A bubbly or homogeneous flow regime is typically encountered at superficial gas velocities below 5 cm/s. This general guideline can vary depending on the physico-chemical properties of the gas and liquid (Besagni *et al.*, 2019). In this regime, bubble sizes are relatively uniform, and bubble rise velocity is consistent, with minimal bubble break-up or coalescence (Kantarci *et al.*, 2005).

A system transfers from the homogeneous to the heterogeneous regime as the superficial gas velocity is increased (Ruzicka *et al.*, 2003). Within the heterogeneous regime, bubble size is more significantly varied throughout the column, and the radial dispersion of bubbles increases significantly (Kantarci *et al.*, 2005). Slug flow typically occurs

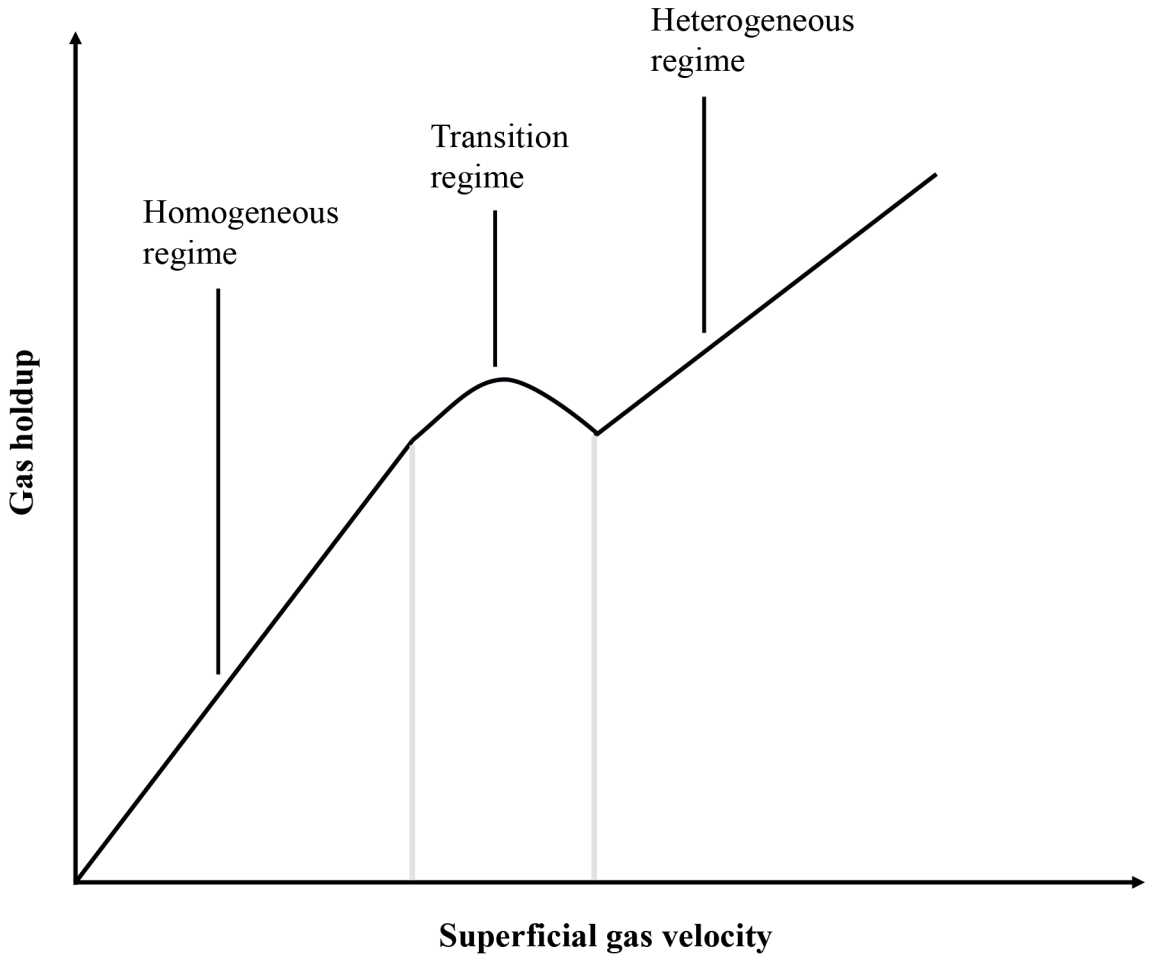


FIGURE 5. Demonstrates how increases in superficial gas velocity affect gas hold-up in the homogeneous, transition, and heterogeneous regimes—adapted from (Ruzicka *et al.*, 2003).

at even higher gas flow velocities, especially in vessels with diameters less than 0.3 m and highly viscous fluids (Deckwer & Schumpe, 1993). Slug flow can be characterised by large, irregularly shaped bubbles that often flow along the column walls or across its entire diameter (Kantarci *et al.*, 2005).

It is crucial to understand gas hold-up to understand the implications of operating within different regimes. Gas hold-up is a parameter used to assess flow regimes and is defined as the ratio of the volume of gas in the liquid to the total volume of gas and liquid in the bubble column. $k_L a$ is often correlated with gas hold-up, with a standard approximation being that $k_L a$ is proportional to gas hold-up raised to the power of 1.1 (Deckwer & Schumpe, 1993). This correlation is a broad approximation, suggesting that gas hold-up conditions will likely also influence $k_L a$. Operating in the homogeneous regime has the advantage of less variation in bubble sizes compared to other regimes, making it easier to predict how gas hold-ups and, consequently, $k_L a$ may increase with increasing superficial gas velocity. Within the heterogeneous and slug-flow regimes, the coalescence of bubbles is greater and much less predictable, leading to a broader range of bubble sizes. The relationship between gas hold-up and superficial gas velocity in these regimes depends more on vessel dimensions (Besagni *et al.*, 2018).

Under homogeneous conditions, gas hold-up typically increases linearly with superficial gas velocity. However, this effect often changes in the transition regime, where the gas hold-up can frequently remain stable or even drop with increases in superficial gas velocity (Besagni *et al.*, 2018). Once the heterogeneous regime is reached with further increases in superficial gas velocity, gas hold-up increases again, but at a slower rate than its increase in the homogeneous regime (Deckwer & Schumpe, 1993). This effect is illustrated in Figure 5.

The observed change in gas hold-up during the transition regime is attributed to a sudden increase in bubble coalescence, which increases the rate at which bubbles rise to the surface (Besagni *et al.*, 2019; Ruzicka *et al.*, 2003).

The homogenous regime is often the preferred operating regime, as it is more controlled and allows for more efficient mass transfer. The preferred regime within an industrial unit will depend on numerous factors, such as the required speed of operation, the necessary efficiency of operation in raw materials and energy input, and the unit's design.

3. Design factors

Design factors influencing the volumetric mass transfer coefficient (k_La) typically include the dimensions of the vessel, material, and pore size of the sparger (Besagni *et al.*, 2018). Bubble columns are generally designed to maximise gas–liquid contact, often operating with a height-to-diameter ratio of at least 5 (Kantarci *et al.*, 2005), which should be kept in mind by operators in wineries. Increasing this ratio increases the time bubbles travel through the liquid, enhancing mass transfer and operating in standard vessels that are relatively shorter and broader, resulting in lower bubble residence times, which can reduce k_La.

The diameter of the column does influence k_La, though generally only to a limited extent. According to Deckwer and Schumpe (1993), this effect occurs when the column diameter is less than 0.6 m. Akita and Yoshida (1973) suggest that this effect is prominent in columns with diameters under 0.15 m, likely due to wall effects on gas flow within the column.

Regarding column design, it is better to have a column that promotes homogeneous flow (Besagni *et al.*, 2018). The column must be sufficiently wide, typically exceeding 0.15 m in diameter (Besagni *et al.*, 2019). While tanks already present in a cellar can be repurposed for sparging operations, those with greater height are preferred. However, they may offer less mass transfer efficiency than a purpose-built bubble column.

4. Operating factors

Operating factors that affect the volumetric mass transfer coefficient (k_La) include temperature, gas flow rate, and system pressure. Temperature changes influence multiple liquid properties that affect mass transfer. An increase in temperature reduces the liquid's viscosity and surface tension, promoting the formation of smaller bubbles at the sparger and thereby increasing the interfacial area (a) (Sehabiague & Morsi, 2013). Additionally, gas diffusivity in the liquid increases with temperature, further enhancing the mass transfer rate. Crucially, a rise in temperature significantly reduces the solubility of oxygen in the liquid phase. This thermodynamic effect decreases the equilibrium concentration of dissolved oxygen (C^*), which increases the concentration gradient between the bulk liquid and the equilibrium state, thereby facilitating faster desorption. In wine systems, this contributes directly to improved oxygen removal during sparging at higher temperatures (Walls, 2020). Bewtra *et al.* (1970) developed a correlation to describe how k_La varies with temperature in water, expressed in Equation 6:

$$OTR \propto k_La = k_La_{20°C}(\theta^{(T-20)}) \qquad (6)$$

where $k_La_{20°C}$ is the k_La at 20 °C, and θ is a constant. For water, a θ value of 1.024 is commonly used, but values between 1.008 and 1.048 have been reported in the literature (Lee, 2017). A study by Walls *et al.* (2022) found that increasing the temperature of wine increases the oxygen removal rate.

Higher operating pressures in a column have been observed to increase the k_La, as bubble sizes are decreased at higher pressures. In addition, higher pressure increases the driving force for mass transfer, further increasing the OTR (Besagni *et al.*, 2018). Increasing the gas flow rate also increases the mass transfer rate by increasing the volume of gas in contact with the liquid at any given time. However, as mentioned, the rate at which this occurs depends on the flow regime within the vessel (Besagni *et al.*, 2018).

STUDIES ON THE PHYSICO-CHEMICAL FACTORS THAT AFFECT OXYGEN MASS TRANSFER

In the previous section, various factors affecting gas–liquid mass transfer were discussed, with particular importance placed on the role of physico-chemical properties. Winemakers have noted that, under similar conditions, oxygen desorption occurs at different rates in different wines, suggesting that the unique physico-chemical properties of each wine significantly influence oxygen desorption.

This section reviews the literature on how different properties and compounds in wine affect the volumetric mass transfer coefficient (k_La) or its physico-chemical properties. Studies evaluating k_La in systems containing compounds similar to those found in wine are also presented. It is important to note that most research has focused on absorption k_La, with limited attention to desorption k_La.

1. Mass transfer studies of oxygen in wine

Few studies have been done on quantifying liquid–gas mass transfer of oxygen in wine, with most focusing on improving oxygen absorption during micro-oxygenation in fermentation (Chiciuc *et al.*, 2010; Devatine *et al.*, 2007; Moenne *et al.*, 2014). Devatine *et al.* (2007) investigated the effect of dissolved carbon dioxide on oxygen absorption during micro-oxygenation, evaluating both absorption and desorption k_La in model wine solutions (12 % *v/v* ethanol, 5 g/L tartaric acid, pH 3.5) and actual wines. Their results showed that in the absence of dissolved CO_2, desorption k_La was similar to absorption k_La. However, the presence of CO_2 introduced a significant deviation, with absorption k_La decreasing and desorption k_La increasing as CO_2 levels rose. Specifically, desorption k_La increased by approximately 60 % when the CO_2 concentration was raised from 0 to 0.4 g/L.

The increase in desorption k_La was attributed to CO_2 desorbing into gas bubbles, increasing bubble size and thus the interfacial area for oxygen desorption. Conversely, the decrease in absorption k_La was due to the decreasing partial pressure of oxygen within the bubble due to CO_2 desorption, which slows down oxygen transfer. Given that wines often contain dissolved CO_2 levels above 800 mg/L, using absorption rates to indicate desorption may lead to inaccurate conclusions. The significant effect of dissolved CO_2 on oxygen mass transfer highlights the need to control CO_2 levels, ideally by complete removal, to isolate the effects of other compounds.

Chiciuc *et al.* (2010) studied the influence of ethanol, sugar, sulfur dioxide, and polyphenols on oxygen absorption during micro-oxygenation. Their study used hydro-alcoholic solutions with tartaric acid at pH 3.5. They found that k_La values were relatively constant across a wide range of ethanol concentrations, with a notable increase only at deficient concentrations (0.05 % *v/v*), which was linked to a reduction in bubble size. Manjrekar (2016) suggests that this reduction in bubble size is due to decreased surface tension when ethanol is added to water. No significant effect on absorption k_La was observed when sulfur dioxide or phenolic compounds were added to model wine solutions. In addition, no significant differences were found between different types of wine (pink, white, red) and the model wine solution in terms of k_La.

Sutton *et al.* (2022) investigated the volumetric mass transfer coefficient in various wines and model wine solutions. Their study discovered that model wine solutions exhibited higher k_La values than three white wines. In addition, all model wine solutions showed greater k_La values than water, likely due to the presence of ethanol in the former. The addition of organic acids and the resulting pH reduction in the model wine solution did not influence the k_La. However, a significant decrease in k_La was noted when yeast extract was added to the model wine solution, suggesting that proteins might be inhibiting mass transfer. To further explore this, they evaluated k_La for a single wine divided into two batches, with one batch treated with bentonite to reduce protein content. The bentonite treatment resulted in a higher k_La value, indicating the potential role of proteins in hindering oxygen mass transfer in complex solutions. These results highlight the considerable variability of k_La values in such complex media.

Moenne (2014) reported higher oxygen absorption k_La in sterile synthetic fermentation media compared to media containing biomass, attributing this to increased viscosity due to compositional changes, which increased the liquid film resistance to mass transfer.

2. Studies on viscosity changes in wine

Shehadeh *et al.* (2019) studied the effects of ethanol, glycerol, glucose, and tartaric acid on the viscosity of model wine solutions and actual wine samples, finding that all these components increased viscosity. Neto *et al.* (2014) also investigated ethanol, dry extract, and reducing sugars concerning the viscosity of red wines. They found a positive correlation between dry extract and viscosity, with a minor correlation between ethanol and viscosity, while reducing sugar content showed no correlation.

Yanniotis *et al.* (2007) identified ethanol and sugar concentration as the predominant factors affecting wine viscosity, with glycerol showing no significant effect. For every 1 % *v/v* increase in ethanol, the viscosity was about an order of magnitude greater than the viscosity increase for each additional 1 g/L of glycerol.

These studies suggest that varying concentrations of ethanol, glycerol, sugar, dry extract, and tartaric acid could potentially affect oxygen desorption by altering the viscosity of the solution. Sutton (2022) reported that ethanol and glycerol concentrations between 9 and 15 % *v/v* and 5 and 25 g/L, respectively, did not have a significant effect on k_La in model wine. Follow-up work showed no significant differences between 0.05 and 10 % *v/v* ethanol concentrations. However, a significant reduction in k_La was observed at lower ethanol concentrations (0.01 % *v/v* and water). A decrease in bubble size at relatively low ethanol levels was observed (> 0.05 % *v/v*), leading to an increase in bubble size and an increase in k_La. Different ethanol and glycerol concentrations, the concentrations normally found in wine, therefore, do not seem to explain differences in k_La between different wines. In addition, changes in viscosity may not directly correlate with changes in k_La, as other factors or compounds found in wine could simultaneously influence k_La.

METHODS FOR MEASURING THE VOLUMETRIC MASS TRANSFER COEFFICIENT AND THE INTERFACIAL AREA

1. Methods for measuring the volumetric mass transfer coefficient

The mass transfer rate can be measured using chemical or physical techniques (Tribe *et al.*, 1995). A widely used chemical method is the sodium sulphite oxidation method, which is based on the reaction of sodium sulphite with dissolved oxygen in the system. The k_La can be calculated from the change in sodium sulphite concentration over time. This technique is relatively easy to perform and can be performed within a bubble column. However, the addition of sodium sulphite, which produced sulphates, and the necessary catalyst can affect the physical properties of the solution, leading to inaccurate k_La values relative to the actual k_La (Garcia-Ochoa & Gomez, 2009). This method also requires higher ionic concentrations, which may affect k_La in some systems, making the technique potentially inaccurate and only representative of k_La in strongly ionic solutions (Van't Riet, 1979).

The typical physical methods involve a step change in the gas concentration within the solution. The most common physical method for measuring k_La is the gassing out procedure (GOP). This method measures the system's dissolved oxygen concentration response to a change in oxygen partial pressure in the gas sparged into the system, usually monitored *via* an oxygen probe (Clarke & Manyuchi, 2012).

An alternative approach is the pressure step procedure (PSP), which evaluates the response of dissolved oxygen concentration in the system to a pressure change. Although the PSP method is accurate, it is more mathematically and experimentally challenging than other physical methods, making the GOP method more commonly used (Clarke & Manyuchi, 2012; Zedníková *et al.*, 2018).

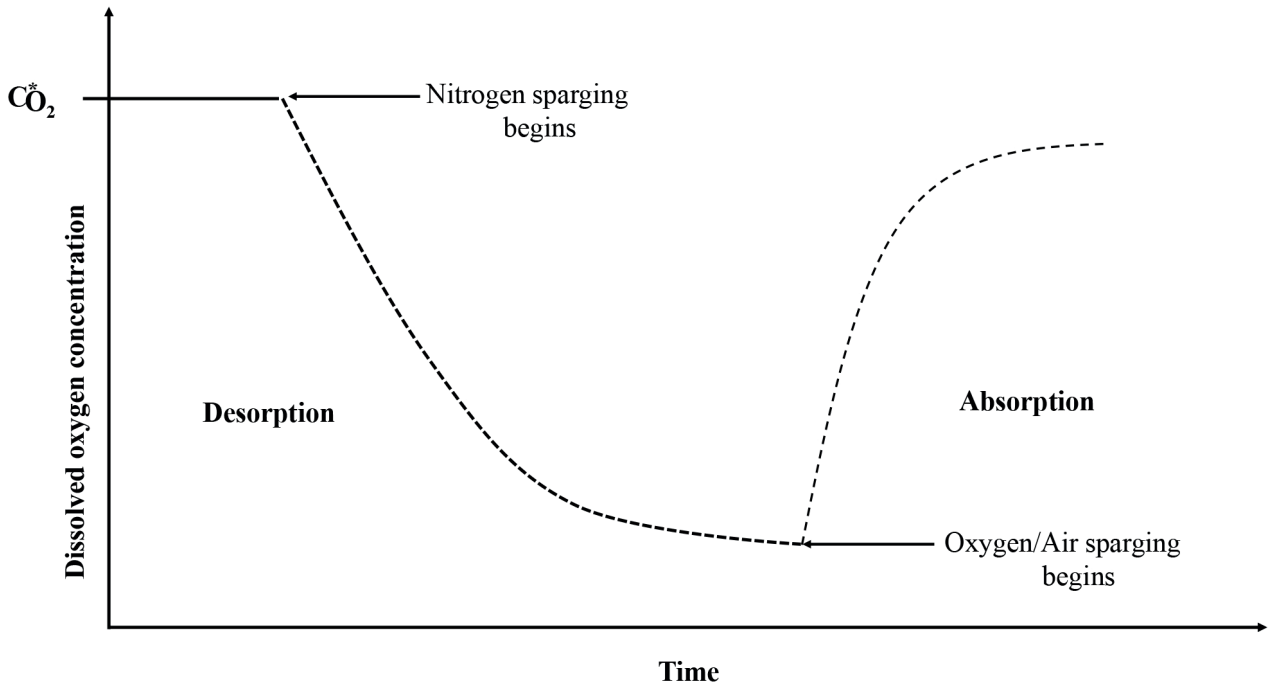


FIGURE 6. An example of the expected change in oxygen concentration using a typical dynamic method for determining the volumetric mass transfer coefficient, modified from Garcia-Ochoa and Gomez (2009).

For measuring oxygen desorption $k_L a$, the GOP procedure involves sparging nitrogen through the system solution to remove all other dissolved gases, such as carbon dioxide. Subsequently, air or pure oxygen is sparged into the solution until it reaches saturation. A second step of nitrogen sparging is then conducted, during which the probe measures the dissolved oxygen response until the oxygen level is reduced to zero or near zero (Garcia-Ochoa & Gomez, 2009). The desorption $k_L a$ can be determined for each time point using Equation 7, where C_{L0} is the initial dissolved oxygen concentration after oxygen sparging, and $C_{L,t}$ is the concentration of dissolved oxygen at time t.

$$\ln\left(\frac{C_{LO}}{C_{L,t}}\right) = k_L a \,.\, t \qquad (7)$$

Figure 6 illustrates the expected changes in dissolved oxygen concentration during desorption (nitrogen input) and absorption (oxygen input) for an aqueous solution. Each experiment involving desorption and absorption generates a graph like the one shown, from which $k_L a$ values for both processes can be obtained.

Inaccuracies in this method may occur if the probe response lag (τP) is not considered. The probe output obtained during measurement may not directly reflect the instantaneous value of the system due to this lag, which depends on the type of oxygen probe used and the system's properties (Van't Riet, 1979). To ensure accurate $k_L a$ measurements, it may be necessary to determine the probe response lag, especially if the probe response time is significant relative to the oxygen transfer rate. Van't Riet (1979) describes the probe response time as the time required for the probe to register a 63 % stepwise change in gas input. The accuracy of the probe measurement improves as τP decreases relative to the mass transfer response time ($1/k_L a$) (Tribe *et al.*, 1995; Van't Riet, 1979). Generally, the probe response may be neglected if $1/k_L a$ is greater than $10\tau P$ (Garcia-Ochoa & Gomez, 2009). According to Van't Riet (1979), the error is less than 6 % for probe responses less than $10/k_L a$, although this is rarely the case in bubble columns (Garcia-Ochoa & Gomez, 2009). If the lag response time is like $1/k_L a$, a second-order model is required for accurate k_L determination.

2. Measuring the interfacial area

To understand the effects of changing factors on k_L and the interfacial area, one of these parameters must be measured separately from $k_L a$. The interfacial area is typically easier to calculate and can be determined by measuring the gas hold-up and the Sauter mean bubble diameter (Sehabiague & Morsi, 2013). The Sauter mean diameter represents the mean bubble size during sparging. While there are other measures of mean bubble size, the Sauter mean diameter is almost exclusively used in mass transfer studies (Azzopardi, 2011). This preference is because the Sauter mean diameter provides a more accurate mean diameter when the goal is to measure the average surface area of a particle (Rawle, 2003).

The Sauter mean diameter is typically measured using high-speed photography, which allows for determining bubble sizes based on the area occupied by pixels in the image, correlated with a ratio of distance units to pixels. Once the interfacial area has been determined along with $k_L a$, k_L can be calculated. Collecting data for each parameter across different systems will enable a more comprehensive understanding of how systems respond to applied changes.

Sutton *et al.* (2022) investigated the Sauter mean diameter, gas hold-up, and interfacial area in various wines and model wine solutions. The study found no significant differences in mean bubble sizes across the different types of wine, including rosé wine with 70 g/L sugar and dry wines with less than 5 g/L sugar. Similarly, the Sauter mean diameter (D_{32}) remained consistent across different model wine solutions and processed wines. Adding organic acids, subsequent pH reductions, and yeast extract in the model wine solutions did not influence bubble size. However, the study did find that bubble sizes in wines and model wine solutions were significantly smaller than in water.

One key finding was that ethanol emerged as the primary factor affecting bubble size in wine. However, variations in ethanol concentrations among the wines, which ranged from 7 % in sweet rosé to 13 % in dry red wine, did not lead to significant differences in bubble size or interfacial area. This lack of variation is explained by ethanol's effect on bubble size, plateauing once a concentration threshold of around 1 % is reached.

The study observed no significant variation in gas hold-up during desorption among the different wines or model wine solutions. In contrast, gas hold-up in water was notably lower due to the larger bubbles rising more rapidly to the surface, resulting in shorter residence times. Similarly, the interfacial area did not significantly vary among the wines and model wine solutions, while water exhibited a substantially reduced interfacial area. This reduction was attributed to the larger bubble size and lower gas hold-up observed in water. Specifically, the Sauter mean bubble diameter in water was approximately 0.55 mm, compared to about 0.25 mm in each of the three wines studied.

Sutton *et al.* (2022) also studied the mass transfer coefficient (k_L) for different wines and model wine solutions. Their findings revealed that the k_L value for water ranged between 0.13 and 0.16 mm/s, whereas for wines, it varied between 0.025 and 0.055 mm/s. These results align with the general understanding that for bubble sizes less than 1.5 mm, the k_L typically falls around 0.1 mm/s.

Interestingly, the study noted that the mass transfer coefficients for model wine solutions did not differ significantly. However, when yeast extract was added to the model wine solution, a reduction in k_L was observed, which was also found in protein-unstable wine. This reduction might be attributed to proteins from the yeast extract or wine proteins adsorbing onto the gas bubbles as they ascended through the bubble column. This adsorption likely increased the resistance to mass transfer, consequently lowering the desorption rate.

Further observations indicated that all white wines exhibited lower mass transfer coefficients than the model wine solutions. This decrease in k_L is likely due to the additional components in the wines that adsorb to the gas–liquid interface.

CONCLUSION

The review highlights the critical role of various compounds influencing wine's oxygen desorption rate constant (k_La). Ethanol and glycerol affect k_La in opposing ways: while ethanol lowers surface tension and promotes smaller bubbles (enhancing interfacial area), both ethanol and glycerol increase viscosity, thereby reducing oxygen diffusivity and lowering k_L. Proteins and polysaccharides, acting as surfactants, accumulate at the gas–liquid interface, forming a barrier that impedes mass transfer and decreases k_L. Temperature significantly affects desorption by modifying liquid properties and gas solubility. Higher temperatures reduce viscosity and surface tension, promote smaller bubble formation, and increase gas diffusivity, all of which enhance k_La. The solubility of oxygen decreases with increasing temperature, raising the driving force for desorption and further improving oxygen removal.

These effects vary across wine types due to differences in wine composition and matrix complexity. The gassing-out method, combined with high-speed imaging to estimate interfacial area, provides a reliable approach for assessing how physicochemical and operational factors affect k_La in wine, but further studies are required to fully elucidate the exact mechanisms and factors affecting oxygen mass transfer in wine.

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