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Control of Oxygen Enrichment during Bottling in the Sparkling Winemaking Process

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ABSTRACT

A method useful for oxygen management during the last step (i.e. disgorging and bottling) in the production of sparkling wines is described here. This study evaluates the benefits of a new apparatus for bottling sparkling wines that is able to control the oxygen environment in the neck of a bottle. Data showed that when corking occurred under controlled atmosphere (O_2 = 6% and O_2 = 3.5%), oxygen intake by bottles was reduced by factors of, respectively, 6 and 10, as compared to bottling under non-controlled atmosphere (O_2 = 20.8%). Moreover, tasting experiments carried out on the wines following bottling (with or without any control of oxygen enrichment) clearly demonstrated that wines obtained under controlled atmosphere are less evolved and show less oxidation notes than do the ones obtained under non-controlled atmosphere.

Keywords: disgorging, oxygen management, sparkling wine industry, wines

INTRODUCTION

Oxygen is proven to play a very important role in red wines as well as in white and sparkling wines (Cheynier *et al*., 2003; Valade *et al*. 2007a, 2007b). However, it is generally accepted that exposure to air is harmful to the aroma and taste of white wine (Singleton and Esau 1969). Loss of discernible fruit varietal bouquet occurs, whereby colour is unfavourably affected (Catarelli 1967). Moreover, oxidation favours the production of aldehydes, ketones, lactones and esters (Baro and Quiros Carrasco 1977; Guichard *et al*. 1993; Rapp *et al*. 1993) which, by their low flavour thresholds, are able to affect aroma even at low concentrations.

Previous studies have revealed that bottling is one of the most critical points concerning wine oxygen enrichment during winemaking (Boulet *et al*. 2004).

For sparkling wines, recent experiments have shown that oxygen enters the bottle neck space during bottling (Tribaut-Sohier and Valade 2003; Valade *et al*. 2007a, 2007b). This oxygen intake depends on several factors such as the time interval between filling-up and corking, the operating principal of the filling machine, conditions that can cause de-sparkling, cork (elasticity, surface treatment), and corking process (interval, cork closing diameter, corking depth).

Until now, the traditional method to counteract oxygen intake during disgorging consists of sulphur dioxide (SO_2) addition either alone or coupled with ascorbic acid. SO_2 is a toxic compound for human health (Stevenson and Simon 1981), and precisely controlling the amount of oxygen entering the bottle during bottling, would help to reduce the SO_2 content without damaging wine sensory properties.

This work describes a new apparatus useful to control the oxygen enrichment during the disgorging step. The impact of oxygen management before bottling on wine organoleptic properties was also studied.

MATERIALS AND METHODS

Wines

Wines used in this study were produced at Ca' del Bosco (Franciacorta, North of Italy) according to the traditional method. Main characteristics of the wine were as follows: alcoholic content: 12.5°; total SO_2 content: 43 mg/L (free SO_2 = 8 mg/L); pH = 3.3, total acidity = 5.5 g H_2SO_4/L.

Description of the corking system

This study focused on a corking system that is able to control the residual oxygen environment in the neck of the bottle according to the European patent application no. 05425810.8 (Capelli and Bielli 2005). The system is composed of a corker, preceded by a carrousel aimed at injecting N_2 in the neck space of each bottle (**Fig. 1**).

Disgorging experiments under controlled or non-controlled atmosphere

The corking system enables to cork sparkling wines accurately controlling the residual O_2 quantity in the bottle neck space by modifying nitrogen concentration in the bottle environment. Two different experiment tests have started comparing different applications of the corking system: 1) corking in ambient atmosphere (oxygen = 20.8%), 2) corking with bottle neck and chamber saturation with nitrogen (residual oxygen percentage = 6%). In some cases, a residual oxygen percentage of 3.5% was also used.

Methods for determining dissolved oxygen in the wine

The oxygen dissolved in wine was determined using a dissolved O_2 analyser (Orbisphere, model 3650/113E), enabling the measurement of the permeating oxygen through a selective diaphragm. Oxygen is detected by an electrode made up of gold and silver with temperature compensation at 20°C. The instrument indicates

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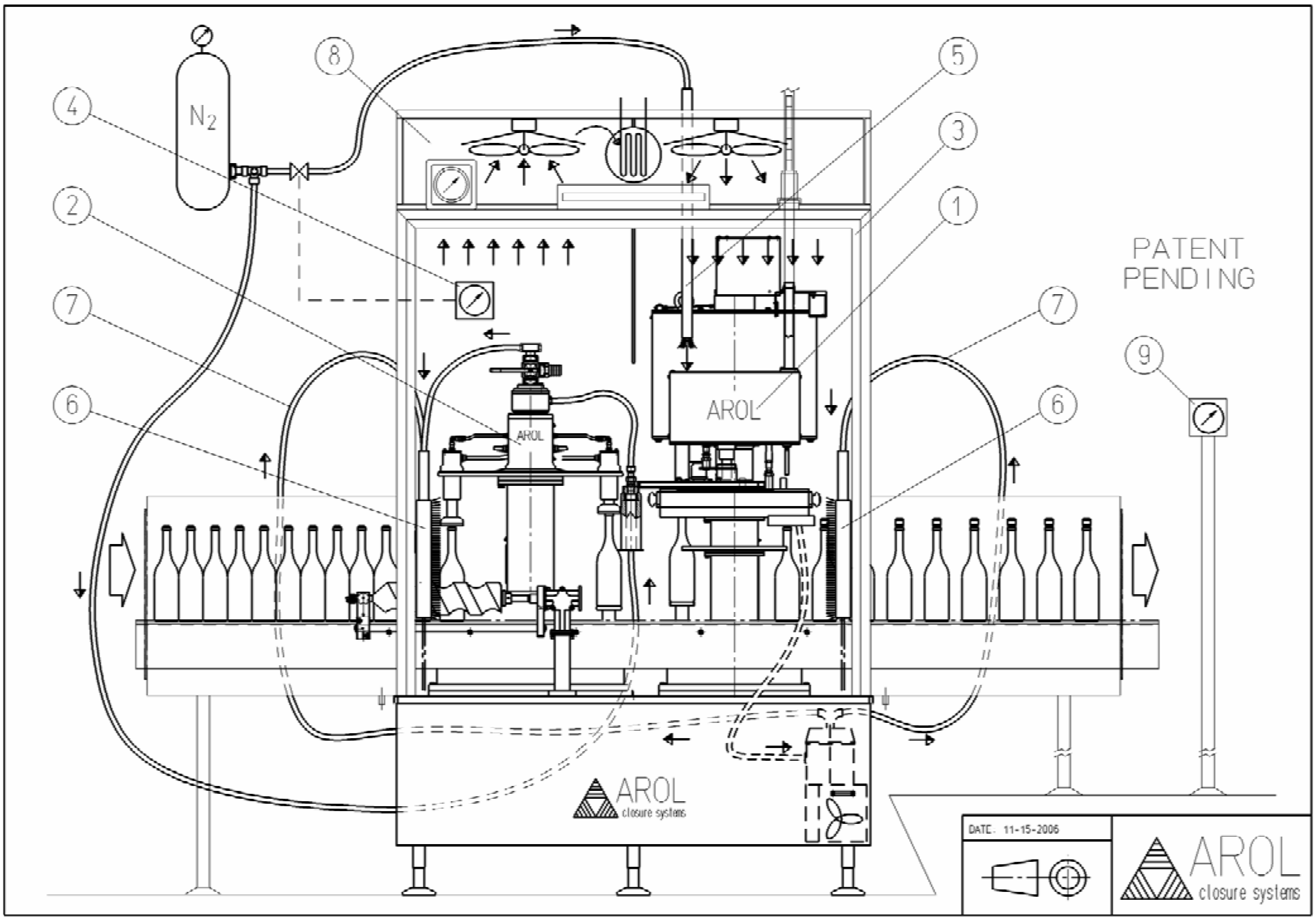

Fig. 1 Description of the corking system. The system is composed of a corker **(1)**, preceded by a carrousel **(2)** aimed at injecting N_2 in the neck space of each bottle, to replace the existing air, collect it and bleed it outside. Both turrets are fitted in a sealed chamber **(3)**, to keep the concentration of O_2 at a steady value; such concentration is controlled by the detector **(4)** and adjusted by feeding N_2 through the ducts **(5)** and **(7)**. To reduce the flow of the gas mixture between inside and outside as much as possible, gas curtains **(6)** are placed at the entry and exit of the chamber and use the flow exiting the injection carrousel. **(8)** and **(9)**: Nitrogen detectors.

the value in mg/L of dissolved O_2; readings have been made at 1/3 and half of the bottle analysed, then averaged. The dissolved oxygen is assessed after having positioned the bottle on the rotary shaker (ASAL, model 709/R), set at 100 rotations/min for 30 min. Later on, the wire-hood and the cork are drilled by a suitable drill, till the height of the second cork level, leaving the first one intact to prevent de-sparkling. The bottle is immediately placed under the Orbisphere bottle sampler (model 29972); the cork is completely drilled by fitting the sampling needle. An overpressure of about 8 bars is applied with nitrogen on the sampler, then the detector escape valve is adjusted so that the wine can flow without creating excessive pressure drops. The oxygen measurement starts after draining the reading cell and detecting the flow stability (about 150 mL/min); it is completed after about 300 mL have flowed. Results are expressed in mg/L of dissolved oxygen.

Quantitation of SO_2

The total SO_2 content of wine was determined according to the EEC Regulation 2676/90.

Tasting experiments

Some bottles of every test (wines bottled under controlled or non-controlled atmospheres) were presented to the panel of judges, consisting of ten trained Enologists and Technicians responsible for the winemaking process, in a randomized order for a blind tasting study. Each judge had to taste the same number of bottles, that is, 10 bottles (with 5 bottles disgorged under non-controlled atmosphere and 5 bottles disgorged under controlled atmosphere). The panel was composed of the same judges over the time course experiments. Each panelist had to describe the features of evolution of the colour and scent of every sample tasted, according to a pre-defined scale of values, and had to judge, according to a similar scale of values, the presence or absence of a bitter taste. Series of tests were planned at regular time intervals of 4 months during one year (one tasting session per interval). Colour was scored against a colour standard according to visual variations in the green/yellow colour intensity. Colour was assessed as follows: from greenish/pale yellow (note 0), marking the less evolved wine, to the Antique gold yellow (note 7), characterizing the most evolved wine. Different sensory attributes were also used to characterize the smell of the wine: from closed, fresh (mineral notes, citrus, fresh fruit and floral aromas) (note 0) to evolved, oxidized (dried fruit, toasty, smoky, ripe fruit, cooked fruit, nutty and honey aroma) (note 7) showing more oxidation notes. Finally, judges had to evaluate bitterness: absent (note 0) or evident bitter taste (note 5) since a correlation was observed between oxygen intake and bitterness.

Statistical analysis

Statistical analysis of variance (ANOVA) (Snedecor and Cochran 1980) was only performed on data concerning tasting experiments in order to evaluate the significance of the differences obtained (at $p = 0.01$) between wines bottled under controlled atmosphere and non-controlled atmosphere, for colour, smell or bitterness.

Calculations for total oxygen assessment in the bottle

To determine the total oxygen content in the bottle, the quantity of oxygen dissolved in wine must be added to the oxygen content of the neck space (Pampuro and Hale 1988). The following equation was used to calculate the total quantity of oxygen (Q) in a corked bottle under balanced conditions:

$$Q_{[mg]} = C \times \left[\frac{V + (32000 \times v)}{S \times R \times T} \right]$$

Where Q is the total quantity of oxygen in the bottle (mg), C is the concentration of oxygen dissolved in the wine (mg/L), V is the liquid volume (L), v is the volume of the neck space (L), S is the solubility of oxygen in water (mg/L/bar) , that is, 43.86 mg/L/bar at 20°C, (Pampuro and Hale 1988), R is the gas constant (R = 8.21 x 10^{-2} m^3 x bar/mol x K, i.e. 0.0821 L x bar/mol x K) (Weast 1988), 32,000 is the oxygen molecular weight (mg/mole) and T, the absolute temperature at 20°C, 293.15 K). The first equation term indicates the oxygen dissolved in the wine, the second term indicates the oxygen in the neck space. Tables (data not shown) have been created to calculate Q in mg/L for each type of bottle and size. There, a linear regression equation has been obtained, depending on the liquid volume and the neck space volume, for values of C measured from 0.1 to 6.0 mg/L. A constant of 30.315 is applied to simplify Q calculation in the tables. This constant derives from the following calculation:

$$\text{Constant} = \frac{32000}{S \times R \times T}$$

RESULTS AND DISCUSSION

Determination of the total oxygen content (mg/L) in the bottle (dissolved + neck space) in relation to different corking conditions

Concentrations of total oxygen in the bottle under non-controlled or controlled atmospheres during disgorging are given in **Table 1**. Tests were made on 0.75 L *champenoise* bottles (volume V), with filling level at 72 mm from the top of the bottle and a neck space volume of 0.0182 L (volume *v*) after corking:

$$Q_{[mg/L]} = C \times \left[\frac{V + (30.135 \times v)}{V + v} \right]$$

Thus, $Q_{[mg/L]} = C \times 1.695$

Data showed that when corking occurred under ambient conditions (traditional corking, O_2 = 20.8%), the total oxygen quantity in the bottle was 5.507 mg/L (3.249 mg/L dissolved in wine plus 2.258 mg/L in the neck space). Under controlled atmosphere (O_2 = 6% and O_2 = 3.5%), the total oxygen quantity absorbed by bottles was reduced by factors of, respectively, 6 and 10. Standard deviations (SD) of the oxygen measurements (**Table 1**) are relatively high, as the number of bottles analysed is rather low. It is noteworthy that, before bottling, the oxygen level of the wine was very low (0.005 ± 0.003 mg/L). N_2 injection into the bottle neck did not cause any significant loss of CO_2, that is, 0.48 bar (under controlled atmosphere) *vs* 0.43 (traditional disgorging).

Analyses of oxygen consumption and SO_2 loss after disgorging

At the disgorging step, 15 mg/L SO_2 together with 50 mg/L ascorbic acid were added. **Fig. 2** presents changes in the oxygen and SO_2 contents experienced by a 750 mL bottle 12 months after disgorging in relation to corking conditions. Data showed that the oxygen content of a bottle corked under non-controlled atmosphere is high at the time of disgorging (4.92 mg/L) and then dramatically decreases within four months following disgorging (with a loss of 4.56 mg/L); the oxygen content reached 0 within 12 months. A decrease in O_2 concentration appears to be linked with a relatively high reduction in the total SO_2 content, the concentration of which decreases from 54 mg/L (disgorging step) to 41 mg/L after four months and 39 mg/L after 12 months. Under controlled atmosphere (O_2 = 6%), there is a loss of 0.85 mg/L O_2 after four months, the oxygen content at the time of disgorging being low (0.89 mg/L). Simultaneously, there is no significant decrease in the total SO_2 content after 4 and 12 months, respectively, ca 1 mg/L and 2 mg/L. This last observation is of considerable importance as it shows that the less the O_2 content at the time of disgorging is, the less addition of SO_2 is necessary.

It is well known that a continuous oxygen exposure occurs during wine ageing in the bottle due to oxygen permeation through the closure. Interestingly, screw cap closures have recently become a popular alternative for some white wines. Wines under the screw cap retains high concentrations of SO_2 and ascorbic acid and show lowest degrees of browning together with low levels of oxidized aroma characters, suggesting that such a type of closure is less permeable to oxygen than natural corks. Unfortunately, this closure produces a rubber-like flavour/aroma in wine after 18 months, likely as a consequence of a lack of oxygen in the bottle. This deleterious consequence of a lack of oxygen in the bottle explains why we have chosen to carry out disgorging experiments using low – but not nil – oxygen concentrations, in this study.

Tasting experiments

Results of the two first tasting tests carried out 4 and 8 months, respectively, after bottling did not show any difference between wines corked under controlled or non-controlled atmosphere (data not presented). This is linked to the antioxidant activity of the SO_2 and the ascorbic acid added before bottling. Results of the third test, that is, 12 months after bottling, showed the different evolutions of the wines

Table 1 Oxygen concentration in the bottle in relation to different corking conditions (tests made on 0.75 L *champenoise* bottles).

Sample	Oxygen level (%)	Oxygen concentration (mg/L) Dissolved	Neck space	Total	Number of samples	SD	SD (%)
Traditional Corking	20.8	3.249	2.258	5.507	6	0.843	15.3
Inert Corking	6	0.550	0.383	0.933	6	0.095	10.2
Inert Corking	3.5	0.322	0.223	0.545	6	0.065	11.9

Table 2 Sensory characteristics of wines bottled under non controlled or controlled atmospheres 12 months after disgorging.

Parameters tested	Sensory characteristics of wines bottled under controlled atmosphere (O_2= 6%)		Sensory characteristics of wines bottled under Non-controlled atmosphere	
	Judges Average Notes	SD[4]	Judges Average Notes	SD[4]
Colour[1]	1.9 a[5]	0.45	5.8b[5]	0.2
Smell[2]	2.2 c[5]	0.5	5.6d[5]	0.33
Bitterness[3]	1.4 e[5]	0.32	3.8f[5]	0.44

[1]from greenish/pale yellow (note 0) to Antique gold yellow (note 7)
[2]from closed, fresh (mineral notes, citrus, fresh fruit and floral aromas) (note 0) to evolved, oxidized (dried fruit, toasty, smoky, ripe fruit, Cooked fruit, nutty and honey aroma) (note 7)
[3]absent (note 0) or evident bitter taste (note 5)
[4]Standard Deviations
[5]Within a same line, Judges average notes followed by different letters are significantly different at p= 0.01 using an ANOVA test.

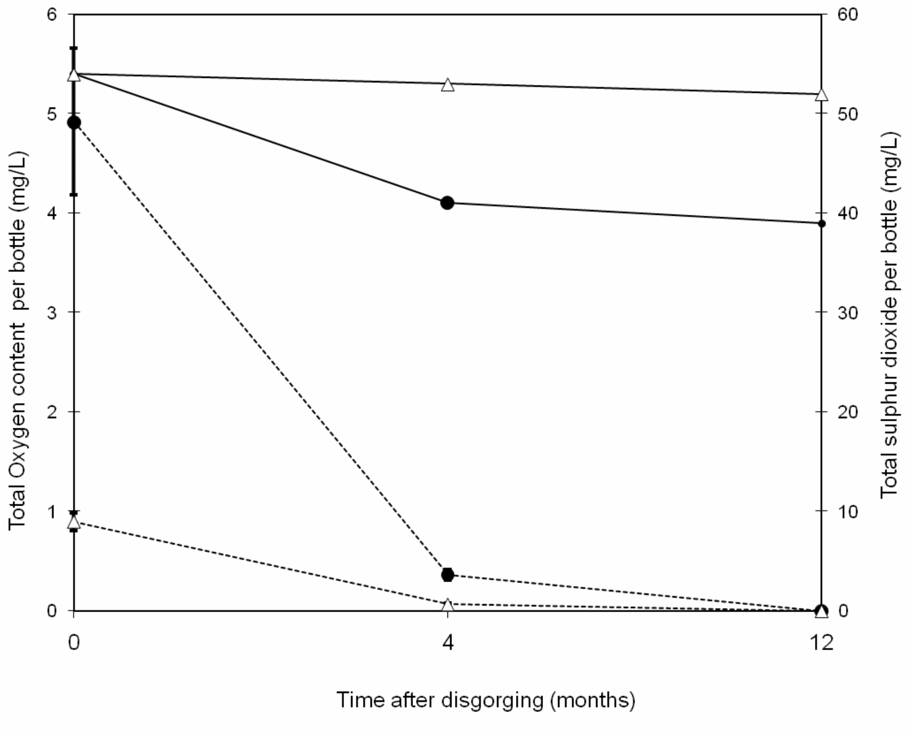

Fig. 2 Oxygen consumption (---) and SO_2 loss (—) after disgorging in two different corking conditions: Traditional corking (●) and inert corking (O_2 = 6%) (Δ). Error bars are provided when they are higher than the symbols used.

in a decisive and undisputable way. Data clearly indicate that all of the 10 judges rated the bottles obtained after disgorging under controlled atmosphere as less evolved and less bitter. Judgements about colour and smell both received on average notes of 1.9 and 2.2, respectively, while the bitter taste received a note of 1.4 (**Table 2**). In contrast, bottles disgorged traditionally were characterized as more evolved and oxidized. Judgements about colour and smell both received on average notes of 5.8 and 5.6, respectively, while the bitter taste received a note of 3.8 (**Table 2**). Data were analysed using an ANOVA test and all means (disgorging under controlled atmosphere *vs* traditional disgorging) were significantly different for p value of 0.01.

CONCLUSION

This work has evaluated the benefits of a new apparatus for disgorging and corking sparkling wine that is able to control the oxygen environment in the neck of the bottle with a positive influence on the colour and sensory properties of the resulting sparkling wine. This apparatus seems to have potential in the sparkling wine industry and addresses an industry need to reduce oxygen intake during disgorging and corking.

REFERENCES

Baro ALJ, Quiros Carrasco JA (1977) Les conditions de formation des aldéhydes dans les vins. Relation et importance en rapport avec les phénomènes d'oxydation et les caractéristiques organoleptiques. *Bulletin de l'Office International de la Vigne et du Vin* **50**, 253-267

Boulet JC, Moutounet M, Vidal JC (2004) Les apports d'oxygène au cours des traitements des vins. Bilan des observations sur site. 3ème partie. *Revue Française d'Oenologie* **205**, 25-33

Capelli S, Bielli P (2005) European Patent Office Application N° 05425810.8.

Catarelli C (1967) Etude de la madérisation et de sa prévention dans les vins blancs. Fermentation et Vinification. *2ème Symposium International d'Oenologie*, Bordeaux-Cognac, 13-17 June

Cheynier V, Atanasova V, Fulcrand H, Mazauric JP, Moutounet M (2003) Oxygen in wine and its role in phenolic reactions during aging. *Australian Journal of Grape and Wine Research* **9**, 23-27

Guichard E, Phamt T, Etiévant P (1993) Mise au point d'une méthode de dosage rapide du sotolon, molécule-clé pour l'arôme des vins Jaunes du Jura. Connaissance aromatique des cépages et qualité des vins. *Actes du Symposium International*, Montpellier, France, pp 360-365

Pampuro P, Hale HJ (1988) Misure di ossigeno disciolto nelle bevande [Measurements of oxygen dissolved in beverages]. *Industrie delle Bevande* **17**, 150-153

Rapp A, Versini G, Ullemeyer H (1993) 2-Aminoacetophenon Versursachende Komponente der "untypischen Alterungsnote" bei Wein. *Vitis* **32**, 61-62

Singleton VL, Esau P (1969) *Phenolic Substances in Grapes and Wine and their Significance,* Academic Press, NY, 600 pp

Snedecor GW, Cochran WG (1980) *Statistical Methods* (7th Edn), The Iowa State University Press, Ames, Iowa, pp 215-269

Stevenson DD, Simon RA (1981) Sensitivity to ingested metabisulfites in asthmatic subjects. *Journal of Allergy and Clinical Immunology* **68**, 26-32

Tribaut-Sohier I, Valade M (2003) Le rôle de l'oxygène lors du dégorgement. *Le Vigneron Champenois* **11**, 2-14

Valade M, Tribaut-Sohier I, Bunner D, Pierlot C, Moncomble D, Tusseau D (2007a) Les apports d'oxygène en vinification et leurs impacts sur les vins. Le cas particulier du Champagne (1ère partie). *Revue Française d'Oenologie* **221**, 1-8

Valade M, Tribaut-Sohier I, Bunner D, Laurent M, Moncomble D, Tusseau D (2007b) Les apports d'oxygène en vinification et leurs impacts sur les vins. Le cas particulier du Champagne (2ème partie). *Revue Française d'Oenologie* **222**, 17-28

Weast RC (Ed) (1988) *Handbook of Chemistry and Physics* (1st Student Edn), CRC Press, Boca Raton, 2536 pp