Oxygen Permeation of BetterBottle® Carboys
– Direct Measurement –

Erich L. Gibbs, PhD
BetterBottle, a division of High-Q, Inc.
www.better-bottle.com
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INTRODUCTION

BetterBottle PET fermentation carboys were introduced to the home winemaking and brewing markets in the Spring of 2003, as a safe and easily handled alternative to glass carboys, and they have been widely used to ferment and bulk age excellent wines and beers ever since. The success of the BetterBottle PET fermentation carboys is empirical evidence that they have very low permeability for oxygen. However, until fairly recently, making direct permeation measurements of intact carboys has been a practical impossibility.

New instrumentation, based on the response of a phosphorescent platinum sensor material to a beam of laser light, accurately measures the concentration of oxygen in closed spaces.[1, 2] BetterBottle utilized this instrumentation to measure the extent to which oxygen permeates through, or leaks by, a variety of the closures and air locks that are used for home winemaking and brewing.[3] The present study utilizes the instrumentation and an extremely efficient *displacement* method for purging the oxygen from the large spaces within carboys in order to directly measure the oxygen permeation of BetterBottle PET fermentation carboys sealed with BetterBottle PET O-ring closures.

SUMMARY

Based on results obtained to date, oxygen permeates from air into BetterBottle fermentation carboys, which have been purged of oxygen and sealed with BetterBottle PET O-Ring closures, at a rate of about 0.05 ml of O_2 per contained liter per day. Put in perspective, this is less oxygen than is recommended for micro-oxygenation (MOX) and less oxygen than permeates through liquid-filled air locks and many types of closures, especially those made of materials such as silicone rubber, thermoplastic elastomer, or plasticized PVC.[3, 4, 5] It is also a miniscule fraction of the oxygen that enters a carboy when a closure is removed, even very briefly, to make an addition or to perform testing of a wine or beer. And the amount of oxygen that enters a wine or beer during *open racking* (siphoning from one open container to another open container) is off the charts by comparison.

METHODOLOGY

In order to measure the rate of oxygen permeation for a carboy with very low permeation in as short a period of time as possible, it is necessary to purge essentially all of the oxygen from the carboy. However, purging the oxygen from the relatively large volumes contained by carboys is not trivial. Assuming ideal mixing of the gases in a container and an oxygen-free purge gas, a basic purge equation can be used to make an approximate calculation of the amount of purge gas and time required to purge a container by *flushing* (flowing a purge gas through a space).[6]

$$\mathbf{T = [V/P] \bullet l_n [C_{inital} / C_{ending}]}$$

V = Volume of container in liters;
P = Rate of flow of the purge gas in liters/min;
$\mathbf{C_{inital}}$ = Initial concentration of oxygen in container;
$\mathbf{C_{ending}}$ = Final concentration of oxygen in container;
T = Minutes required to reach the purge level (C_{ending})

When:
V=22.7 liters
P = 10 liters per minute
$\mathbf{C_{inital}}$ = 21%(typical oxygen concentration of air)
$\mathbf{C_{ending}}$ = 0.01%
$\mathbf{l_n [C_{inital} / C_{ending}]}$ = 7.65

The time, **T**, will be 22/10 • 7.65 or 16.8 minutes
and
168 liters of purge gas will be required.

In practice, the *flushing* approach to purging carboys proved to be impractical. Perhaps as the result of our particular approach, it was not possible to reach 0.01% concentrations of oxygen in a 22.7 liter carboy even after 30 minutes of purging and about 300 liters of high-purity purge gas. Vacuum purging was not an option, because fermentation carboys are not designed to withstand vacuum. The use of an oxygen scavenger was also not an option, because there would have been no practical way to remove the scavenger from a sealed carboy, once the desired reduction of oxygen concentration had been achieved. Therefore, it was decided to use *displacement* purging, which involves replacement of the undesired gas by a purge gas without intermixing.

A gas-tight *displacement* purging fixture, based on the design of the BetterBottle O-Ring Closure was developed (Figure 1). Solid PET was machined to make a purging adapter that had the same sealing dimensions as BetterBottle O-Ring Closures. This PET adapter and two stainless steel, double-ferrule, 1/8” NPT X 1/4” tubing adapters were sealed to an anodized 6061-T6 aluminum plate with ResinLab EP1305LV epoxy.[7] A stainless steel, 4-way, crossover flow path, 2500 PSIG (172 BAR) valve was attached to the aluminum plate and copper tubing was used to make connections for the flow of gas from one component to another.[8] Two additional valves, which were bubble tested at 50 PSI to confirm that they did not leak when closed, were attached in the purge gas line and vent line leading to and from the 4-way valve. The neck of a 91 cm premium latex rubber balloon was taped tightly to the PET adapter. A 1.5 mm ID polytetrafluoroethylene (Teflon) tube, long enough to reach the bottom of a carboy, was also taped to the adapter, outside the neck of the balloon and ending above the neck. The purpose of this tube was to prevent the expanding balloon from blocking the venting of gas outside the balloon and to permit the balloon to expand fully against the inside walls of the a carboy. With the 4-way valve in one position, the arrangement of the connections permits purge gas to flow into the balloon, while gases outside the balloon flow out the vent (Figure 1 A through D). Once the balloon is fully expanded, the 4-way valve is repositioned so that purge gas flows into the space outside the balloon, forcing the purge gas in the balloon out the vent until the balloon is completely collapsed again (Figure 1 D through A). This sequence completes one cycle of *displacement* purging.

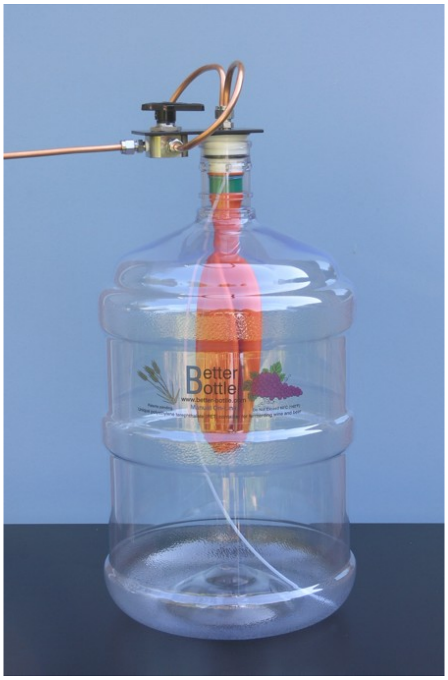


Figure 1A

Figure 1B

Figure 1C

Figure 1D

Only two cycles of *displacement* purging, requiring less than 5-6 minutes and an amount of purge gas equal to 4 times the volume of a carboy, were sufficient to purge carboys to oxygen concentrations of less than 0.01%. Once the oxygen concentration reached the desired level within a carboy, the purge gas was turned off and as soon as all flow from the vent had ceased, the 4 way valve was set to an off position, and the valves in the input and vent lines leading to and from the 4-way valve were closed. At this point, the pressure of Argon inside the carboy was equal to the external air pressure. The purge gas used for testing was ultra high purity, grade 5.0, argon (no oxygen).[9]

A Mocon OpTech O_2 instrument was used to make oxygen concentration measurements and Mocon organoplatinum targets were attached to the inside surface of the neck of test carboys (Figure 2).[10]

Figure 2

Care was taken to be certain the Mocon probe circuits were warmed up and stabilized. The distance of the Mocon probe to the targets was kept constant and calibration was preformed no more than 45 seconds prior to making a measurement. The Mocon calibration targets (zero O_2 and Air) were kept covered and close to the test carboys, so conditions would be the same for both. When this was done, measurements made several minutes apart varied by only a few percent (e.g., 0.116% vs. 0.119%). The oxygen concentration within test carboys was measured at 24 hour intervals for 25 days or until the levels reached approximately 0.22%, 1/100th the concentration of oxygen in air.

TEST RESULTS

The test data are presented in graphic format, so they can be easily understood. The blue bars in the figures represent the reading for a given day. The slopes of the red lines represent the average transfer rate (TR) of oxygen from the start of testing to the last measurement and the numerical values of the slopes lines are given in milliliters of oxygen per day and milliliters of oxygen per day per liter of carboy volume. The fact that the values of the daily measurements closely match the average slopes is noteworthy, because this confirms the stability of the test fixture and analytical equipment as well as the conditions during testing. It is reasonable to assume that the results represent the TR for the carboys and that the test fixture is essentially gas tight, because previous testing of BetterBottle O-ring closures and the other components of the test fixture demonstrated that they were gas tight at the low differential pressures involved.[3]

Figure 3

Oxygen Permeation

22.7 Liter, 680 gram BetterBottle® Carboy

Conditions: Temp: 21-22 °C   Barometric Pressure 735-750 mmHg

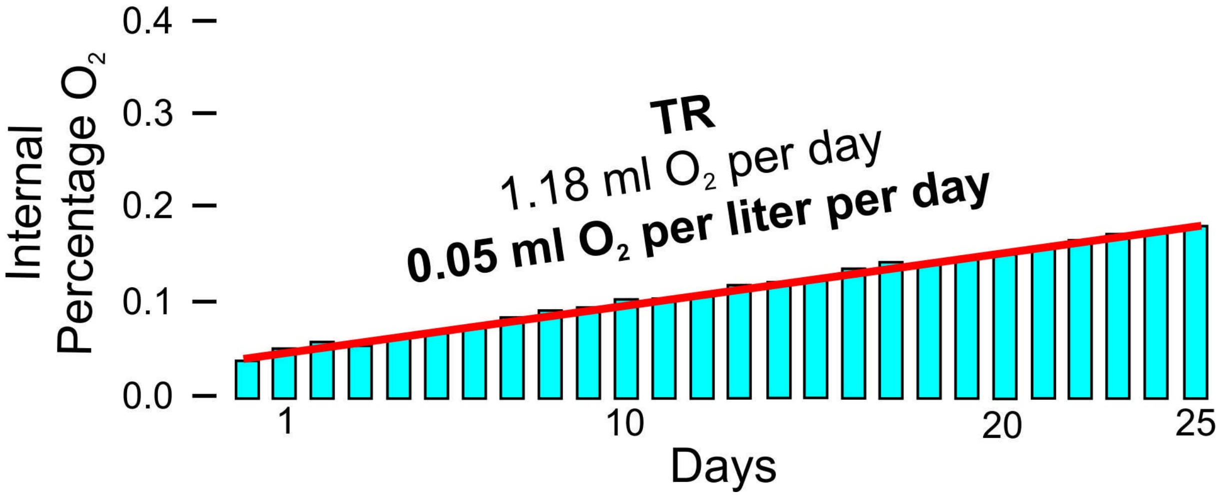


Figure 4

Oxygen Permeation

18.9 Liter, 680 gram BetterBottle® Carboy

Conditions: Temp: 21-22 °C   Barometric Pressure 735-750 mmHg

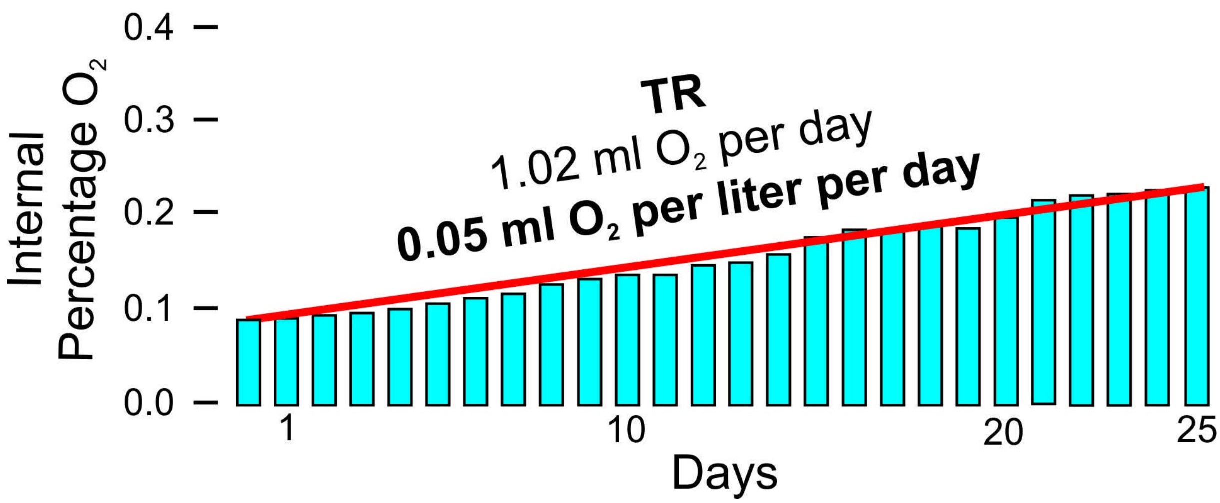
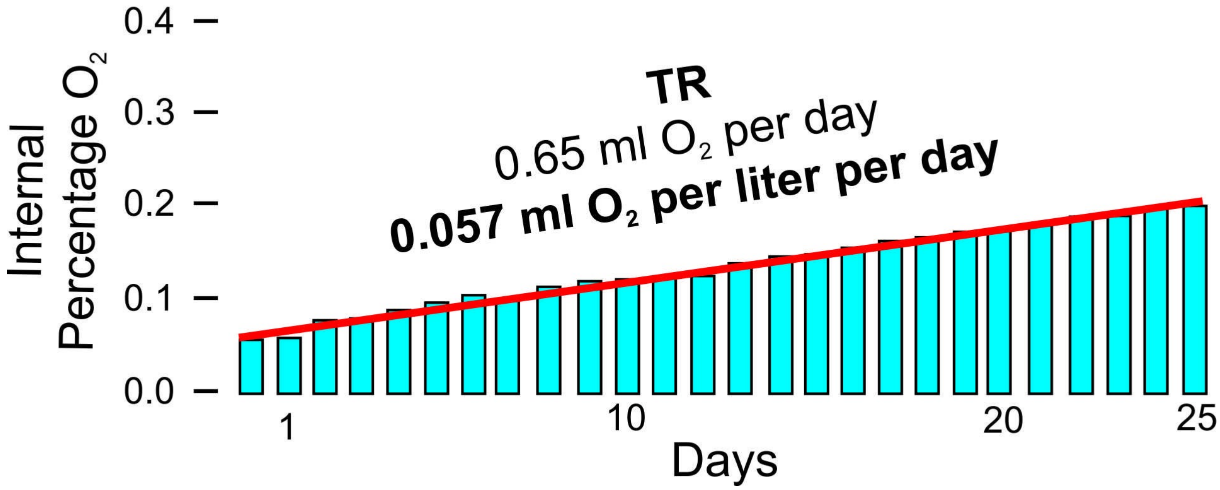


Figure 5

Oxygen Permeation

11.4 Liter, 680 gram BetterBottle® Carboy

Conditions: Temp: 18.1-19.6 °C Barometric Pressure 741-754 mmHg



CONCLUSIONS

The direct measurement of the oxygen permeability for BetterBottle fermentation carboys confirms the extensive empirical evidence that these carboys are well suited for home winemaking and brewing. Furthermore, the linearity of the results over substantial periods of time indicates that testing for shorter periods can be meaningful. This knowledge, taken together with the efficiency and effectiveness of *displacement* purging should make it possible to measure the oxygen permeability of a variety of the different types of containers offered to the home winemaking and brewing markets.

Significantly less oxygen permeates through the walls of BetterBottle carboys than permeates through liquid-filled air locks and many types of closures, especially closures made of silicone rubber or plasticized PVC.[3] The oxygen permeating into BetterBottle carboys also represents a miniscule fraction of the oxygen that enters any type of carboy when the closure is removed, even very briefly, to make an addition or to perform testing of a wine or beer. And the amount of oxygen that enters a wine or beer during *open racking* (siphoning from one open container to another open container) is off the charts by comparison to the permeability of BetterBottle carboys.

The very low oxygen permeation rates of BetterBottle carboys are on the low side of what is recommended for micro-oxygenation (MOX), which means that MOX can be controlled and adjusted by the winemaker or brewer.[4, 5]

References

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7 Ellsworth Adhesives, W129 N11687 Morese Drive, Germantown, WI 53022

8 Swagelok SS-43YF2-125 – Chicago Fluid System Technologies, 360 Windy Point Drive, Glendale Heights, IL 60139

9 The BOC Group, Inc., *575 Mountain Avenue. Murray Hill, NJ 07974*

10 Mocon, Inc., 7500 Mendelssohn Ave. N., Minneapolis. MN 55428