

## CONTRIBUTION TO THE CHARACTERIZATION OF CORKS' USE-LIFE

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### Keywords:

**Abstract.** *Oxygen permeability data is relevant for selecting wine bottle closures, but the impact of wine contact, temperature, and time use has not yet been explained. Market available stoppers were analysed for oxygen ingress over time in stoppered bottles, under different temperatures, with and without contact between the cork and wine simulant. The Weibull model described well the oxygen ingress. Differences were found between cork types, in long-term oxygen pressure values and ingress rates. The temperature effect followed an Arrhenius behaviour, with statistical significance only for micro agglomerated corks. Micro agglomerated corks exhibited slower initial oxygen ingress but higher long-term oxygen ingress than natural corks. Principal Component Analysis (PCA) showed that factors related to the bottleneck-cork interface contributed more to the variance of the system than the cork type. Liquid contact reduces oxygen ingress rate around five times. The temperature impact in the oxygen ingress was lower for natural corks than for micro agglomerated corks.*

## 1 INTRODUCTION

Despite corks being used as a closure for bottled wine for a long time, they still lack confirmation for the reasons of their effectiveness and the changes in their performance during the use period.

Much of the research regarding cork performance has been conducted in the sense of bottled wine ageing [2], [16], [17]. Other research work has been conducted in the context of determining the permeability under different test conditions [9], [10] or analysing the applicable mass transfer models [1], [3], [8]. Reviews have been presented to describe the complexity of the wine bottles' closure system [5], [6].

The rationale of the current study considers that significant improvements in the reliability of closure performance can only be achieved by closer attention to the parameters that affect cork closure performance. The ongoing project aims to investigate the preservation of cork properties by analysing their mechanical behaviour; permeability to gases; wine-cork interactions, and fatigue analysis.

## 2 BACKGROUND

The research focuses on cork behaviour regarding mechanical properties and permeability to oxygen, considering both wine-cork interactions, bottleneck, and temperature.

Market available stoppers, natural and micro agglomerated (technical) corks, were analysed, under different temperatures (8, 23, and 40 °C), with and without contact between cork and wine simulant.

Compression pattern differs from natural to micro agglomerated corks. The compression behaviour has been reported with properties of flexible cellular materials exhibiting compression curves (stress–strain) with three regions [12]. Recovery tests aim at bringing insights into elastic properties decay and show different mechanical contours of natural and micro agglomerated corks.

The empirical Weibull model describing the oxygen ingress in the bottle over time (equation (1)) described well the observed data. This model has been used to describe various processes in food processing, quality, and safety, including the mass transfer of additives from plastics into food [14].

Differences were found between cork types in long-term oxygen pressure ( $P_o$ ) and ingress rates ( $\tau$ ). Micro agglomerated corks exhibited slower initial oxygen ingress but higher long-term oxygen ingress than natural corks. Liquid contact reduces oxygen ingress around five times. The temperature impact in the oxygen ingress was lower for natural corks. Principal Component Analysis (PCA) showed that factors related to the bottleneck-cork interface contributed more to the variance of the system than the cork type.

The data obtained for oxygen ingresses allows modelling the effect of temperature in the range of 8 to 40 °C for predicting the behaviour under varying conditions of storage or transportation.

## 3 FIGURES

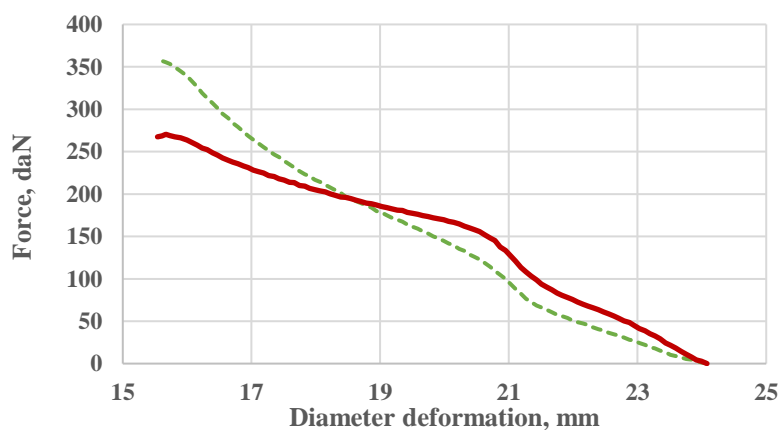


Figure 1. Compression profile of natural (—) and technical (---) corks

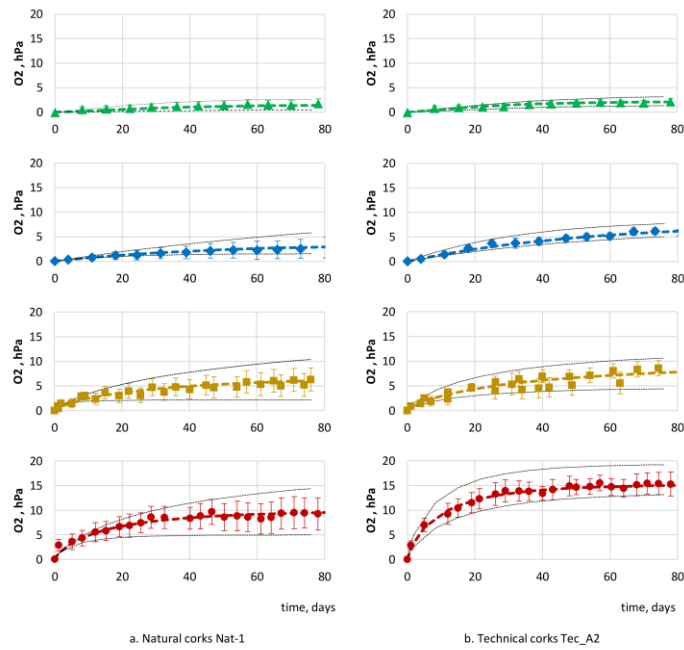


Figure 2. Monitoring of oxygen ingress along time (dots for average, bars for standard deviation). Test with wine simulant contact at 23 °C (▲); Test in dry condition at 8 °C (◆), 23 °C (■), 40 °C (●). - - - - model fit. - - - - upper and lower curves represent the replicates with the higher deviation from the average curve.

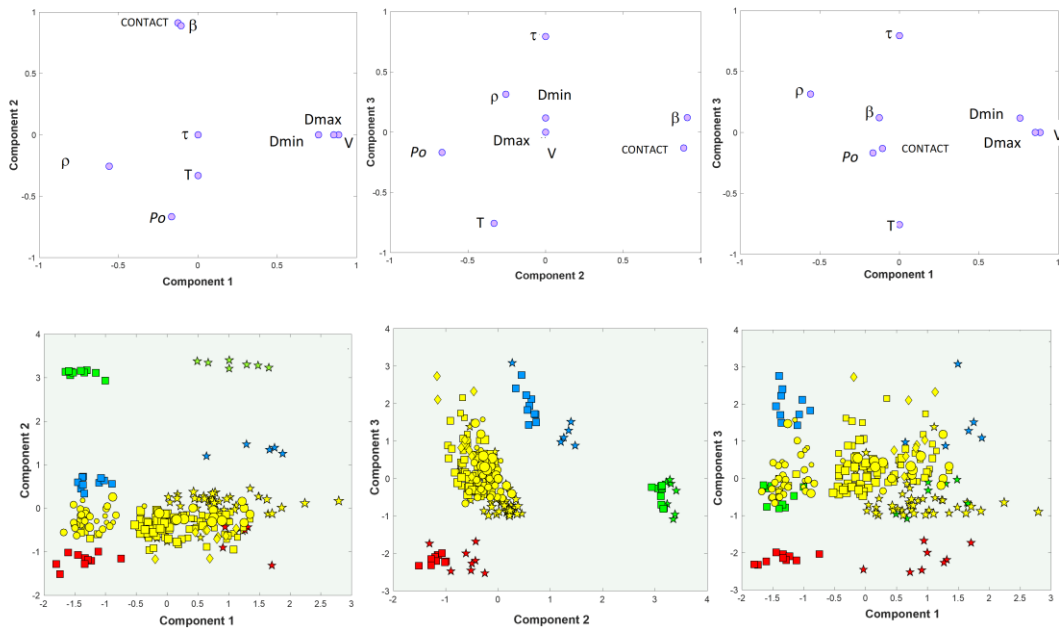


Figure 3: PCA loadings and score plots with all variables. Natural (☆), Tec\_A (□), Tec\_B (○), Tec\_C (◇). Symbol colour for temperatures: blue 8 °C, yellow 23 °C, red 40 °C and green for 23 °C under wet condition. Symbol sizes for different closures lengths.

## 4 EQUATIONS

The Weibull model is an empirical model that considers two parameters  $\tau$  and  $\beta$ .

$$p(t) = p_0 \left( 1 - \exp \left( -\frac{t}{\tau} \right)^\beta \right) \quad (1)$$

where  $p(t)$  is the oxygen partial pressure inside the bottle changing with time  $t$ ,  $p_0$  is the pressure at equilibrium, and the initial pressure is considered zero.

$\tau$  is the system time constant associated with the process rate and has been found to depend on the temperature following an Arrhenius-type behaviour. The parameter  $\beta$  and the corresponding shape of the curve at earlier times was found to relate to different mechanisms controlling the mass transfer.

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