



Report

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Your project

Ellagitannins and low molecular weight polyphenol analysis on two wine bottles of Chablis 1er cru

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1 Introduction and purpose

The Procork company developed a membrane technology to control the rate of oxygen entering the wine bottles when closed with natural cork. This membrane made of 5 different layers allows selective permeation of oxygen to allow micro-aeration of grape and oak barrel tannins while blocking compounds passing from the cork to the wine.

In February and April 2020, the independent Bordeaux based wine critic Jean Marc Quarin conducted a professional tasting of two bottles of Chablis 1er cru, Fourchaume, ██████████ 2018: one closed with ██████████ technical cork and the second closed with a technical cork coated with the Procork membrane. The bottle closed with ██████████ cork has been described as having more tannic, acidic and bitter notes than the Procork bottle.

To further compare those two bottles, HPLC analyses have been performed to quantify the hydrolysable tannins present in the wine

This document summarizes the results obtained by HPLC and makes the link with the tasting conclusions of the wine critic.

2 Services summary

Title: Ellagitannins and LMW polyphenol analysis on two wine bottles of Chablis 1er cru		
Experimental Plan		
Number of samples	Two bottles of the same wine: <ul style="list-style-type: none"> - 1 closed with technical cork coated with the Procork membrane - 1 closed with [REDACTED] technical cork 	
Analyses		
Molecular analyses		
Parameters	Methodologies	Details
Hydrolysable tannins expressed as castalagin (no distinction)	External laboratory method	HPLC chromatography with UV-Vis or Fluorescence detection
Low molecular weight polyphenols (including Gallic, Protocatholic, p-Hydroxybenzoic, Vanillic, Caffeic, Syringic, p-Coumaric, Ferulic, Catechin, Epicatechin, Caftaric, Quercetin and Kaemferol)	External laboratory method	HPLC chromatography with UV-Vis or Fluorescence detection

3 Experimental

3.1 Sample preparation

The wine used for this study was a white wine: a Chablis 1er cru, Fourchaume, [REDACTED] 2018.

Two different bottles were used: one closed with [REDACTED] technical cork and the second closed with a technical cork coated with the Procork membrane. Those two wine bottles have been stored in the same conditions for six months.

The wine from each bottle has been sampled. To remove ethanol, each liquid sample has been first concentrated to dryness using a rotavapor and then redissolved.



3.2 HPLC analyses

For the analysis, high performance liquid chromatography (HPLC) method coupled with UV-Vis detection was used (HPLC Agilent 1200 detector DAD (UV/Vis)). The chromatographic separation was carried out in a C(18) column (Zorbax C18 150 mm x 4.6 mm) with water/phosphoric acid (solvent A) and methanol/phosphoric acid (solvent B) as the mobile phase.

A part of the fraction prepared has been directly used for the ellagic acid determination. The other part has been submitted to hydrolysis in an acid medium and a water bath in order to hydrolyze the ellagic tannins and release the ellagic acid. The difference between the released ellagic acid and the initial free acid indicated the amount of ellagic tannins. Results have been expressed as vesicalagin using the molecular weight of this compound.

4 Results and discussion

HPLC analysis has been performed on the wine samples collected from the two bottles. The bottles contained the same wine bottled with two different corks. The analysis was targeted to quantify hydrolysable tannins known to be found in all wine. The results are shown in the table below.

Compound	Results (ppm)	
	█ Bottle	Procork bottle
Organic tannins (expressed as vescalagin)	34.3	27.5
Gallic acid	1.6	1.3
Protocatholic acid	0.28	0.27
p-Hydroxybenzoic acid	0.30	0.25
Vanillic acid	<i>Not detected</i>	<i>Not detected</i>
Caffeic acid	1.7	1.6
Syringic acid	0.15	0.16
p-Coumaric acid	0.30	0.20
Ferulic acid	1.2	1.2
(+) - Catechin	0.68	0.60
(-) + Epicatechin	0.24	0.42
Caftaric acid	5.8	5.6
Quercetin	0.08	0.08
Kaemferol	0.03	0.05

4.1 HPLC results

- Tannin Extraction from █ compared to ProCork

The results show the █ bottle has approximately 25% more organic tannins expressed as vescalagin than the ProCork bottle. Both closures contain cork which is a material known to readily release tannins into water and alcohol mixtures. The lower level of tannins in the ProCork bottle demonstrates that the ProCork membrane would act as a barrier to the extraction of tannins from the cork into the wine.

- Impact of tannin

Tannins are naturally found in wine due to their extraction from the grape skins, seeds, and stalks by the water and alcohol in the wine. Further tannins are added to the wine when tannins are also extracted from the oak wood in the barrel and the oak bark in the cork. Over-extraction of tannin is a wine fault which reduces wine quality and particular sources of tannin (such as stalk tannin) can be worse than others.

Tannins are known to impact the sensory response to wine due to their interaction with both taste receptors and mechanoreceptors. Tannins induce a tactile astringency response due to the mechanoreceptors in the mouth as well as a gustatory acidity and bitterness response due to the taste receptors.

The astringency response gives sensations of dryness and roughness in the mouth as well as puckering felt in the muscles of the face and it is triggered at a threshold of approximately 1 ppm. Astringency develops and dissipates slowly, taking more than 15 seconds for perception to fully develop.

4.2 Comparison of HPLC results to a professional tasting

Prior to the HPLC analysis conducted here, a professional tasting of the wine was conducted in February and April 2020 by Jean-Marc Quarin, an independent Bordeaux based wine critic. His summary is shown below and his identification of more tannic, acidic and bitter notes leading to a loss of silky mouth feel were confirmed by the HPLC analysis. It indicates the measured 25% difference due to tannins extracted from the cork had a significant and negative impact on the wine tasting.

Jean-Marc Quarin
Chablis 1er cru, Fourchaume, [REDACTED] 2018
Comparative tasting with Procork technical cork stopper versus [REDACTED] stopper

Conclusion and hypotheses on the first tasting made on February 16 2020 and continued on April 3, 2020

- 1) *In this first series of open bottles, the [REDACTED] corking seems more tannic, which removes the silky mouth feel from the wine, especially between the middle and the finish where I have spotted both more acidic and more bitter notes.*
- 2) *The Procork corking induces a more regular stimulation of the wine between the entry in the mouth and the finish (no loss of silky mouth feel). The balance of sensations is better respected.*
- 3) *[REDACTED] is regular in the presence of freshness on the nose.*
- 4) *Procork is regular in the presence of more ripe fruit and silky mouth feel on the palate. To note that this maturity does not take away from the freshness.*
- 5) *There are 2 different styles.*

Conclusion and hypotheses of the second tasting of April 3 and 6, 2020

- 1) *[REDACTED] shows a difference from one bottle to another. Presence of more acidity marked the first time versus the second and a bitter shade the second time.*
- 2) *[REDACTED] is regular in the presence of freshness on the nose.*
- 3) *Procork is regular in the presence of more ripe fruit and silky feel on the palate.*
- 4) *In the 2 tests Procork presents a wine very well constructed on the palate. It respects the 3 best stimulation times: presence on attack, presence in the middle, presence in the finish. In addition, the aromatic part reveals regularity in the presence of ripe fruit ("exotic fruit") or sometimes creamy. It is very stable.*
- 5) *One might think that the wine corked by Procork will keep its qualities over time. After three days of opening, it picked up tension in the body while keeping the silky mouth feel mentioned at the start while the [REDACTED] wine loses its qualities.*

5 Conclusion

HPLC analyses have been conducted on two bottles of the same wine six months after bottling (Chablis 1er cru, Fourchaume, ██████████ 2018): a bottle closed with a technical cork coated with the Procork membrane and a control bottle closed with ██████████ technical cork. The objective was to compare the tannins present in the two bottles.

The HPLC analysis showed more tannins in the ██████████ bottle (34.3 ppm) compared to the Procork bottle (27.5 ppm). A professional tasting of these bottles had previously identified in the ██████████ bottle more tannin, acidity and bitterness disrupting the silky mouth sensation. The HPLC analysis confirmed the taster's perception of more tannin.

The results showed that such variation of only 25% in tannins may have a negative impact on the wine taste. It also indicated that extra tannin can be extracted from the cork and alter the taste of the wine in a negative way.