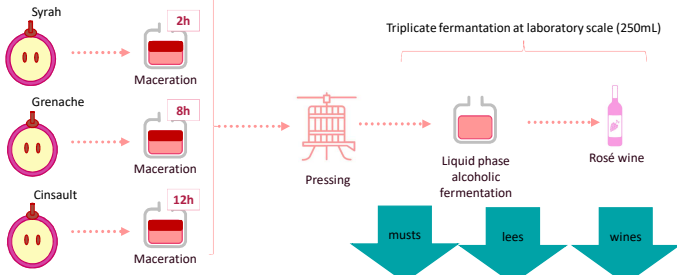


Characterization of rosé wine pigments using a combination of analytical methods. Impact of grape variety (Grenache, Cinsault, and Syrah) and of alcoholic fermentation

Introduction

Color is one of the key elements in the marketing of rosé wines as these wines are usually sold in clear glass bottles. Their broad color range is due to the presence of polyphenolic pigments, including anthocyanins, the pigments of pink and red grape berries, but also derived pigments formed from them through reactions with other phenolic compounds and with some yeast metabolites and brown pigments resulting from oxidation of hydroxycinnamic acids and /or flavanols [1]. In the present study, the link between pigment composition and color of rosé wines and the impact of changes taking place during alcoholic fermentation on these characteristics and the mechanisms involved were investigated for Grenache, Cinsault and Syrah, widely used for rosé winemaking.

Materials and Methods



Color and polyphenol analysis

Targeted analysis UPLC-ESI-QqQ-MS/MS MRM mode [2]

- benzoic acids
- hydroxycinnamic acids
- flavan-3-ols
- flavanols
- flavanonols
- stilbenes
- anthocyanins

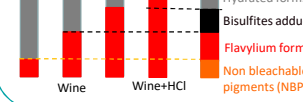


Color analysis UV-visible spectrophotometry

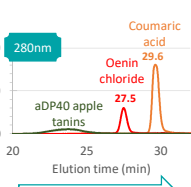
$$\text{Color Intensity} = A_{520} + A_{420} + A_{620}$$

$$\text{Hue} = \frac{A_{420}}{A_{520}}$$

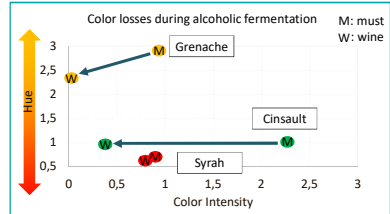
Absorbance at 520nm



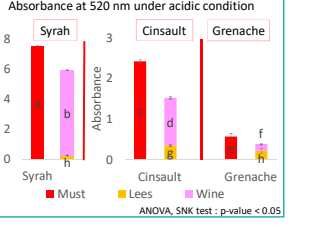
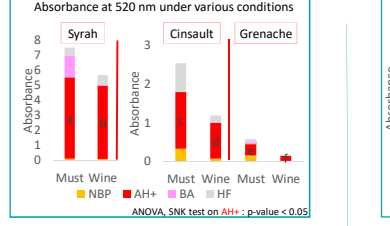
High Performance Size Exclusion Chromatography



Color analysis



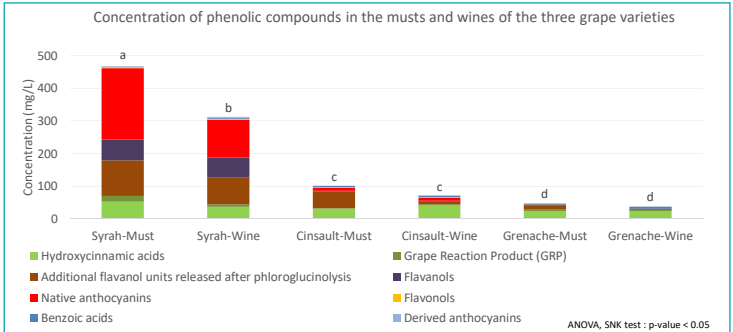
- Color of the three varieties
- Grenache and Cinsault exhibited a color loss during alcoholic fermentation
- Syrah alcoholic fermentation did not induce a major color loss



- Syrah wine and must were the most colored at 520 nm (red)
- Conversion of bisulfites ions to pigments in Syrah must explains the absence of color loss during fermentation
- Non bleachable pigments are found in larger proportions in Cinsault and Grenache modalities
- Pigments lost during fermentation was recovered from lees
- Pigments recovered from Grenache and Cinsault lees represented respectively 40% and 15% of the must color
- Pigments recovered from Syrah lees represented less than 5% of the must color

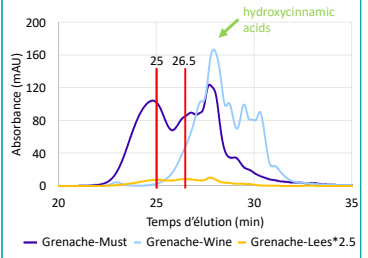
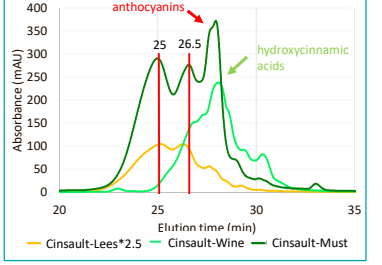
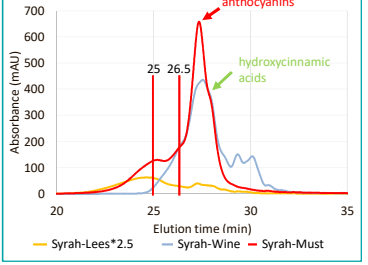
Polyphenol analysis

Targeted analysis (UPLC-QqQ-ESI-MS in the Multiple Reaction Monitoring (MRM) mode)



- Native anthocyanins, flavanols and hydroxycinnamic acids are the main compounds detected in the musts and wines of all three grape varieties
- Native anthocyanins are the major compounds observed in Syrah must and wine whereas mainly hydroxycinnamic acids were detected in Grenache and Cinsault
- Losses of the total targeted polyphenol concentrations during Syrah fermentation (but no color change)
- No significant drop of the total targeted polyphenol concentrations for Grenache and Cinsault, despite large color loss
- Missing compounds in the targeted method

High Performance Size Exclusion Chromatography



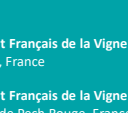
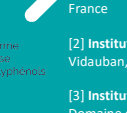
- Mainly anthocyanins in Syrah versus hydroxycinnamic acids in Grenache/Cinsault
- Populations eluted at 25 min and 26.5 min present in must, absent in wine and recovered from the lees
- Populations eluted at 320 nm, 420 nm, 520 nm in Grenache/Cinsault
- presence of high molecular weight pigments not included in the targeted method

Conclusion

Combining color analysis with polyphenol analysis using targeted MS analysis and size exclusion chromatography, the present study confirmed that light and dark rosé wines have different phenolic compositions. These differences exist from the must stage: Syrah musts exhibited high levels of anthocyanins and tannins whereas Grenache and Cinsault contained larger proportions of hydroxycinnamic acid derivatives, including higher molecular weight compounds and pigments likely resulting from enzymatic oxidation. These specific must compositions impacted changes taking place during fermentation. Syrah anthocyanins were partially converted into derived pigments, with limited impact on color while the large color loss observed during fermentation of Grenache and Cinsault was attributed to pigment adsorption on lees.

[1] Leborgne, C., Lambert, M., Ducasse, M.-A., Meudec, E., Verbaere, A., Sommerer, N., Boulet, J.-C., Masson, G., Mouret, J.-R., & Cheynier, V. (2022). Elucidating the Color of Rosé Wines Using Polyphenol-Targeted Metabolomics. *Molecules*, 27(4), 1359. <https://doi.org/10.3390/molecules27041359>
[2] Lambert, M., Meudec, E., Verbaere, A., Mazerolles, G., Wirth, J., Masson, G., Cheynier, V., and Sommerer, N. (2015). A High-Throughput UHPLC-QqQ-MS Method for Polyphenol Profiling in Rosé Wines. *Molecules* 20, 7890–7914.

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