Grape seed flavanols extraction and mechanical-acoustic properties as influenced by maceration time and ethanol content

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Introduction

Grape monomeric flavan-3-ols and their polymers are involved in mouth-related sensations and colour stability of wines. In particular, seeds flavanols are characterized by low molecular weight and high galloylation, correlated respectively to bitterness and astringency [1]. Maceration time and ethanol content are the main factors influencing their extraction in the first vinification stage [2].

In this study a descriptive evaluation of the maceration from seeds was done, focusing on the extracted flavanol content and compostion, and on the mechanical and acoustic parameters of the fresh and macerated seeds.

These latter instrumental parameters are correlated with hardness and crunchiness, and in turn with the flavanols extractability from seeds [3, 4, 5], and may help in understanding the influence of ethanol/time combination in the flavanol release during maceration.

Material & Methods

Three replicates of *Vitis vinifera* L. cv Pinot noir seeds from 40 g of berries were taken, cleaned, and inserted in 50 mL of the following buffered (pH 3.4, 5 g/L of tartaric acid) macerating solutions:

- 0% v/v ethanol
- 5% v/v ethanol
- 10% v/v ethanol
- 15 % v/v ethanol
- Simulated maceration (SimMac)

Sampling points:
3 days
7 days
10 days

SimMac samples were added with increasing ethanol concentration to simulate a fermentative maceration: at 48, 72, 96, 144, and 168 hours the alcohol levels were raised to 3, 6, 9, 12, and 15% v/v, respectively.

Chemical analysis

On macerating solutions

- Total phenolic index (IPT) [5]
- Condensed tannins Methyl-cellulose assay (MTC) [6]
- HPLC- UV analysis of monomers (Epicatechin and catechin) [5]
- HPLC-UV analysis of mean degree of polymerization (mDP) [5]

Texture analysis

On seeds before and after maceration

Compression test [4]:

Mechanical parameters:

Seed break force (F_s, N) and break energy (W_s, mJ), Young's modulus of elasticity (E_s, N/mm), deformation index (Di_s, %)

Acoustic emission (24 dB gain value):

average acoustic area (AV, dB), linear distance (LD), maximum acoustic pressure (Maximum, dB), number of acoustic peaks> 15 dB ($N_{pk>15dB}$), average acoustic pressure level of $N_{pK>15dB}$ ($AV_{pk>15dB}$, dB)

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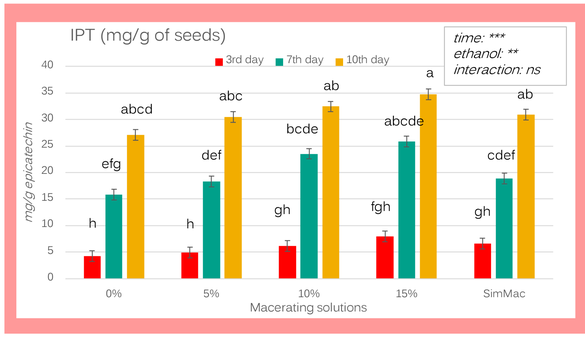
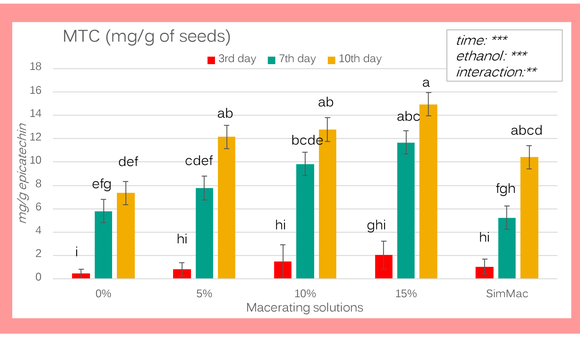
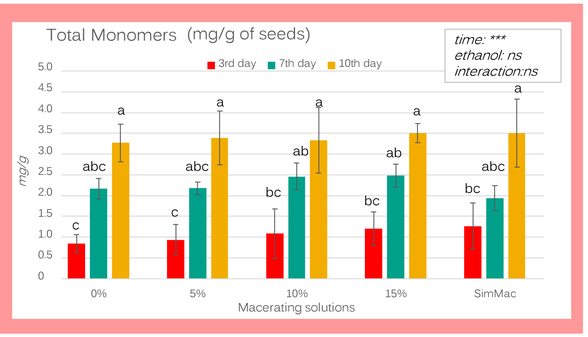
Results

Tannin content and composition





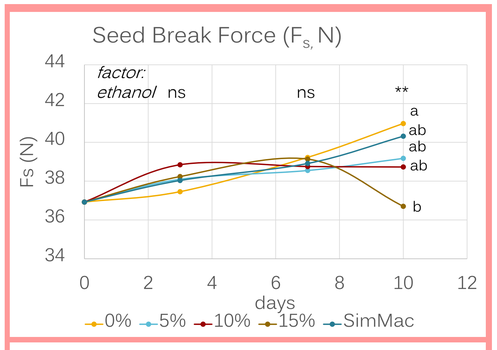


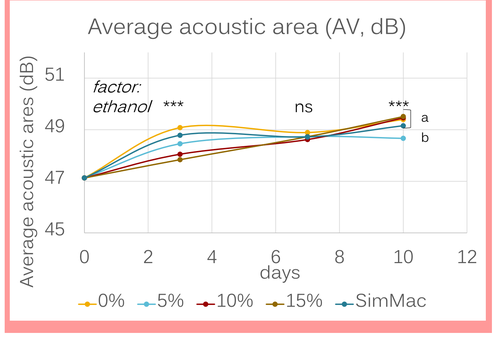
- Time and ethanol influenced the extraction of total phenols (IPT), in particular condensed tannins (MTC).
- Monomeric flavanols are extracted depending on the maceration length.

	Ethanol	Total mDP		Total G%		Terminal units (%) Ct		ECt		ECGt		Extension units (%) Cext		ECext		ECGext	
3rd day	0%	1.47 ± 0.12	e	6.79 ± 1.77	f	63.41 ± 1.69		35.41 ± 1.32		1.18 ± 0.37	f	6.08 ± 0.92	a	90.06 ± 0.16	abc	3.87 ± 0.77	i
	5%	1.77 ± 0.29	de	10.53 ± 2.76	def	64.94 ± 0.92		32.94 ± 1.07		2.11 ± 0.80	def	5.01 ± 1.34	abc	89.40 ± 0.86	abc	5.59 ± 0.61	gh
	10%	1.82 ± 0.43	de	11.85 ± 4.23	cdef	63.76 ± 1.99		33.79 ± 1.24		2.44 ± 1.48	def	3.77 ± 1.29	abc	89.39 ± 1.20	abc	6.84 ± 0.10	def
	15%	2.62 ± 0.44	abc	17.63 ± 2.13	ab	65.91 ± 3.93		29.27 ± 5.23		4.82 ± 1.32	abc	3.59 ± 1.16	abc	88.46 ± 0.98	c	7.95 ± 0.41	abcd
	SimMac	1.53 ± 0.13	e	8.99 ± 2.31	ef	62.70 ± 0.73		35.66 ± 1.26		1.64 ± 0.55	ef	5.56 ± 2.71	ab	89.89 ± 2.38	abc	4.55 ± 0.38	hi
7th day	0%	2.09 ± 0.13	cde	11.75 ± 0.84	cdef	61.87 ± 1.23		35.56 ± 1.53		2.56 ± 0.31	def	2.53 ± 0.70	c	91.39 ± 0.57	a	6.09± 0.27	fg
	5%	2.39 ± 0.10	abcd	14.79 ± 0.72	abcd	62.18 ± 0.64		34.26 ± 0.92		3.56 ± 0.28	bcde	2.31 ± 0.30	c	90.21 ± 0.59	abc	7.48± 0.31	bcde
	10%	2.84 ± 0.30	ab	17.26 ± 0.55	ab	64.54 ± 10.95		30.57 ± 11.47		4.89 ± 0.61	abc	2.41 ± 0.10	c	89.52 ± 0.16	abc	8.07± 0.20	abc
	15%	2.86 ± 0.10	ab	18.13 ± 0.22	ab	60.46 ± 1.58		34.43 ± 1.66		5.11 ± 0.11	abc	2.23 ± 0.08	c	89.16 ± 0.12	abc	8.61± 0.14	ab
	SimMac	1.83 ± 0.14	de	11.44 ± 1.40	cdef	61.22 ± 0.63		36.52 ± 1.07		2.25 ± 0.44	def	3.19 ± 0.63	abc	90.47 ± 0.38	abc	6.35± 0.25	efg
10th day	0%	2.29 ± 0.27	abcde	13.34 ± 0.62	bcde	62.89 ± 1.61		33.84 ± 2.09		3.26 ± 0.49	cde	2.93 ± 0.21	bc	91.01 ± 0.27	ab	6.06± 0.09	fg
	5%	2.37 ± 0.04	abcde	16.09 ± 0.60	abc	62.99 ± 2.45		33.03 ± 2.40		3.98 ± 0.14	abcd	2.58 ± 0.08	bc	89.97 ± 0.41	abc	7.44± 0.33	cde
	10%	2.97 ± 0.27	a	18.16 ± 0.95	ab	61.61 ± 4.77		33.05 ± 4.48		5.34 ± 0.53	ab	2.47 ± 0.11	c	89.11 ± 0.36	abc	8.42± 0.35	abc
	15%	2.97 ± 0.08	a	19.52 ± 0.38	a	62.26 ± 1.08		31.98 ± 1.27		5.76 ± 0.20	a	2.27 ± 0.03	c	88.78 ± 0.46	abc	8.95± 0.45	a
	SimMac	2.24 ± 0.03	bcd	15.48 ± 0.92	abcd	63.92 ± 0.28		32.42 ± 0.33		3.66 ± 0.25	bcd	2.53 ± 0.19	c	90.15 ± 0.47	abc	7.31± 0.39	cde
factor:time		***		***		ns		ns		***		***		ns		***	
factor: ethanol		***		***		ns		ns		***		ns		***		***	
interaction		ns		ns		ns		ns		ns		ns		ns		*	

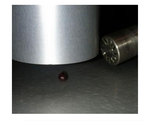
- Extracted tannins mDP is increasing during maceration. Ethanol helped in the solubilisation of longer structures: after 3 days of maceration the mDP of 15% ethanol solution was almost as high as that found after 10 days of maceration.
- Modification in terminal and extension units depended on the beginning of galloylated units release: both ethanol and time increased the ratio of G%.
- The **simulated maceration (SimMac)** extracted similarly to the 0% ethanol solution for condensed tannins and monomers. Therefore the **hydration is the main driver of their extraction in fermentative maceration condition**. In contrast, the galloylated units extraction was influenced by the increasing ethanol, particularly from the 7th day of maceration (when 15% ethanol level was reached).

Texture analysis: acoustic and mechanical parameters





Acoustic/ Mechanical	Texture parameters	Phenolic parameters	R Pearson
A	Maximum (dB)	mDP	* 0.590
A	$N_{pk>15dB}$		-0.540 *
A	$AV_{pk>15dB}$		* 0.626
A	Maximum (dB)	G (%)	** 0.706
A	$N_{pk>15dB}$		-0.565 *
A	$AV_{pk>15dB}$		** 0.729
A	Average acoustic area (dB)	Ct (%)	* -0.527
A	Maximum (dB)	ECGt (%)	* 0.625
A	$AV_{pk>15dB}$		** 0.646
A	$N_{pk>15dB}$	ECt (%)	* 0.603
M	W_s, seed break energy (mJ)	Cext (%)	-0.524 *
A	Maximum (dB)		-0.713 **
A	$AV_{pk>15dB}$		-0.770 ***
M	F_s seed break force (N)	ECext (%)	* 0.555
M	$AV_{pk>15dB}$		* 0.576
A	Maximum (dB)	ECGext (%)	** 0.716
A	$N_{pk>15dB}$		-0.519 *
A	$AV_{pk>15dB}$		** 0.736
A	Maximum (dB)	Total polyphenols-	** 0.642
A	Average acoustic area (dB)	IPT (mg/g)	** 0.643
A	$AV_{pk>15dB}$		** 0.691
A	Maximum (dB)	Condensed tannins-	* 0.623
A	Average acoustic area (dB)	MTC (mg/g)	* 0.569
A	$AV_{pk>15dB}$		** 0.666
M	W_s, seed break energy (mJ)	Total monomers	* 0.596
A	Maximum (dB)	(mg/g)	** 0.645
A	Average acoustic area (dB)		** 0.661
A	$AV_{pk>15dB}$		** 0.702



- Seed break force (F_s) showed different trends depending on the ethanol level of macerating solutions: 0%, 5% v/v, and SimMac showed a constant hardness increase. In the strongest medium (15% v/v ethanol) after 7 days an evident softening was found, this trend (althought not significant) was reported also for 10% v/v solution.
- **Simulated maceration** followed the same trend of 0% v/v ethanol level, confirming the hydration-driven behavior.
- Several correlations between texture and flavanols parameters were found confirming the feasibility of this instrumental technique to study flavanols extractability patterns during maceration.

References

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