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Extraction of phenolic compounds in controlled macerations of Pedro Ximenez grapes

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Extraction des composés phénoliques dans des macérations controlées de raisins de la variété Pedro Ximenez

Résumé: Des raisins écrasés de la variété Pedro Ximenez ont été soumis à un processus de macération avec leur moût pendant 4, 16, 24 et 48 h aux températures de 10, 15 et 25 °C. Dans les moûts obtenus après le pressurage du raisin, 14 composés phénoliques correspondant aux fractions d'acides hydroxybenzoïques, d'acides hydroxycinnamiques, autres nonflavonoïdes, de flavan-3-ols et flavonols, se sont déterminés par HPLC. Par rapport aux conditions de macération, la température n'a pas une influence excessive dans l'extraction de composés phénoliques quand on ne dépasse pas 16 h de contact. Au-delà de ce temps, la température influe de manière bien marquée; c'est pourquoi il n'est pas recommendable de dépasser ce temps, si des pratiques d'oxydation postérieure à la macération ne suivent pas. Les résultats analytiques montrent les dérivés des flavan-3-ols, catéchine et epicatéchine, comme des composants extraits plus vite par rapport à la température de macération, suivis des acides hydroxybenzoïques, d'autres nonflavonoïdes, des acides hydroxycinnamiques et flavonols. La fraction des flavan-3-ols est aussi extraite en plus grandes proportions dans des différentes conditions expérimentales, bien qu'à des niveaux similaires à la fraction d'acides hydroxybenzoïques.

Key words: maceration, mash, must, temperature, time, extraction, phenol, flavour, analysis, white wine, Spain.

Introduction

In white wines, the maceration of the solid fraction of grapes with the must has been the subject of a number of investigations in the last years, with the purpose to increase its fruity character. The results obtained so far have proved to be rather dependent on the must maceration temperature and time (OSZMIANSKI *et al.* 1986; RAMEY *et al.* 1986; TEST *et al.* 1986; MARAIS and RAPP 1988). However, several authors have shown that the increase in the aroma fraction of the must depends significantly on the particular grape variety (ARNOLD and NOBLE 1979; SINGLETON *et al.* 1980; DUBOURDIEU *et al.* 1986; BAUMES *et al.* 1988, 1989 a, 1989 b). Likewise, under given maceration conditions it is the grape variety that determines the passage of a lesser or greater amount of phenolic compounds onto the must, inducing a colour increase and browning susceptibility of the wine (SINGLETON *et al.* 1980; RAMEY *et al.* 1986; CHEYNIER *et al.* 1989; LEE and JAWORSKI 1989). In order to avoid the extraction of too large amounts of phenolic compounds from grapes, maceration conditions should be as mild as possible, even at the expense of decreased extraction of aroma compounds.

A promising solution in this respect may be the induced oxidation of excessive phenolic compounds contained in the must (GUERZONI *et al.* 1981; NAGEL and GRABER 1988; CHEYNIER *et al.* 1989). In any case, a better knowledge of the amounts of phenolic compounds that are extracted and of their rate of extraction is important to establish

Statistical analysis

Statistical calculations were performed by using Statgraphics Statistical Computer Package. The F values for time and temperature were obtained with the aid of the subprogram 'multifactor analysis of variance' by using the data available for the different compounds and fractions. The data of the various polyphenol fractions obtained under the different maceration conditions were subjected to discriminant analysis by a direct method.

Results and discussion

Table 1 lists the average contents in phenolic compounds of the unmacerated must and those macerated under the different temperature and time conditions.

Table 1

Mean and standard deviation of the contents of phenolic compounds of the musts (mg/l)

Moyenne et écart-type des contenus des composés phénoliques dans les moûts (mg/l)

COMPOUNDS & FRACTIONS	WITHOUT MACERATION	MACERATIONS 10 °C 4 HOURS	16 HOURS	24 HOURS	48 HOURS
Gallic	0.11±.01	0.08±.02	0.11±.03	0.10±.02	0.13±.01
Protocatequic	0.10±.01	0.12±.03	0.22±.03	0.31±.09	0.37±.01
p-Hydroxybenzoic	0.48±.08	0.53±.12	1.23±.47	1.35±.31	1.57±.34
m-Hydroxybenzoic	0.10±.01	0.09±.01	0.61±.20	0.78±.02	0.87±.06
Vanillic	0.35±.05	0.34±.05	0.99±.13	1.09±.12	1.17±.17
HYDROXYBENZOIC	1.14	1.16	3.16	3.63	4.11
Caffeic	0.11±.01	0.15±.03	0.20±.09	0.21±.02	0.24±.01
Syringic	0.15±.02	0.18±.03	0.26±.06	0.27±.03	0.40±.01
p-Coumaric	0.10±.02	0.10±.01	0.10±.04	0.08±.02	0.11±.01
Ferulic	0.10±.01	0.11±.01	0.10±.05	0.10±.03	0.14±.03
HYDROXYCINNAMIC	0.46	0.54	0.66	0.66	0.89
Tyrosol	0.40±.01	0.47±.03	0.68±.26	1.03±.07	1.53±.09
Syringaldehyde	0.15±.02	0.18±.01	0.25±.03	0.37±.03	0.59±.01
OTHER NONFLAV.	0.55	0.65	0.93	1.40	2.12
NONFLAVONOIDS	2.15	2.35	4.75	5.69	7.12
Catechin	1.08±.04	1.06±.06	3.41±.22	3.50±.31	4.32±.44
Epicatechin	0.73±.17	0.75±.04	1.24±.27	1.15±.04	1.78±.14
FLAVAN-3-OLS	1.81	1.81	4.65	4.65	6.10
Quercetin	0.13±.03	0.12±.01	0.11±.01	0.18±.01	0.33±.05
FLAVONOLS	0.13	0.12	0.11	0.18	0.33
FLAVONOIDS	1.94	1.93	4.76	4.83	6.43

(continued overleaf)

the behaviour of different grape varieties in maceration processes or to study the behaviour of phenols as oxidation substrates. Little work on kinetic matters related to the phenolic fraction of grapes has so far been done compared with the aroma fraction and its passage onto the macerated must (RAMEY *et al.* 1986).

This work is part of a comprehensive study of the behaviour of Pedro Ximenez grapes in maceration processed with respect to the aromatic (MOYANO *et al.* 1990) and phenolic fractions of the musts. The main aim was to study the extraction of free phenolic compounds under different temperature and time conditions in order to obtain a more precise knowledge of this fraction with a view to subsequent studies on wine browning.

Materials and methods

Maceration

100 kg of grapes of cv. Pedro Ximenez were harvested in semi-ripe stage in the Montilla-Moriles region (Córdoba, southern Spain). Ripe grapes of this variety are customarily used for Sherry production, while semi-ripe grapes yield white table wines.

The grapes were divided into homogeneous batches that were crushed and macerated in triplicate at 10, 15 and 25 °C for 4, 16, 24 and 48 h. Every batch was supplied with 100 mg/l of SO_2 as potassium metabisulphite prior to processing. Once the treatment was finished, the grapes were pressed and the resulting musts were analysed. The average sugar content of the must from unmacerated grapes was 177 g/l (A.O.A.C. 1970), its total acidity 22 meq/l (O.I.V. 1978) and its pH 4.15.

Extraction of phenolic compounds

The unmacerated and macerated musts were subjected to extraction of free polyphenols according to RAMEY *et al.* (1986) by separating the acid and non-acid phenols after adjusting to pH to 2 and 7, respectively, using ethyl acetate as extractant in both cases. The extracts were then evaporated to dryness and redissolved in methanol for subsequent chromatographic analysis.

HPLC chromatographic analyses

Analyses were carried out on a C_{18} column (220 mm × 4.6 mm, 5 μm particle size) by using 2 % aqueous acetic acid and acetonitrile as mobile phases at a flow rate of 2 ml/min and detection at 280 nm.

Acid phenols: The elution phases for this fraction were as follows: gradient elution from 0.1 to 5 % CH_3CN in 5 min, isocratic elution for 10 min, gradient elution up to 15 % CH_3CN in 5 min, isocratic elution for 10 min, and gradient elution up to 100 % CH_3CN in 10 min. In this fraction the following compounds were quantified: gallic acid, protocatequic acid, p-hydroxybenzoic acid, m-hydroxybenzoic acid, vanillic acid, caffeic acid, syringic acid, p-coumaric acid and ferulic acid.

Non-acid phenols: The elution phases for this fraction were as follows: gradient elution from 0.1 to 15 % CH_3CN in 5 min, isocratic elution for 5 min, gradient elution up to 20 % CH_3CN in 5 min, gradient elution up to 30 % CH_3CN in 5 min, gradient elution up to 100 % CH_3CN in 10 min. In this fraction the following compounds were quantified: tyrosol, syringaldehyde, catechin, epicatechin and quercetin.

Catechin and epicatechin were the two most abundant phenolic compounds in the unmacerated must. Their extracted levels increased by a factor of 8—10 on maceration at the most energetic conditions (48 h and 25 °C). However, this does not mean that these compounds were extracted in the highest proportions in relation to their contents in the unmacerated must; in fact, other compounds such as m-hydroxybenzoic acid increased up to 20 times of the initial amounts.

In order to determine the influence of maceration time and temperature on the extraction of the different compounds, variance analysis has been carried out (Table 2). For all of them significant differences ($P < 0.01$) in the two variables have been found. When plotting the extracted concentrations of the different compounds against time and temperature, it is seen that as a rule the influence of the temperature becomes more marked as the time increases, with the exception of m-hydroxybenzoic acid, for which the opposite is true. Likewise, the influence of maceration time is higher when temperature increases.

The parameters of the concentration-maceration time regression lines are listed in Table 3 for all compounds and fractions at each extraction temperature (10, 15 and

Table 1 (cont.)

COMPOUNDS & FRACTIONS	MACERATIONS 15 °C			
	4 HOURS	16 HOURS	24 HOURS	48 HOURS
Gallic	0.11±.01	0.16±.04	0.16±.02	0.18±.04
Protocatequic	0.13±.01	0.26±.03	0.32±.06	0.63±.04
p-Hydroxybenzoic	0.46±.05	1.87±.01	1.88±.01	1.88±.07
m-Hydroxybenzoic	0.40±.02	1.29±.07	1.38±.03	1.52±.07
Vanillic	0.49±.06	1.13±.06	1.11±.11	1.24±.08
HYDROXYBENZOIC	1.59	4.71	4.85	5.45
Caffeic	0.22±.06	0.32±.01	0.33±.01	0.39±.03
Syringic	0.24±.03	0.31±.03	0.34±.10	0.48±.02
p-Coumaric	0.11±.01	0.21±.09	0.29±.02	0.30±.01
Ferulic	0.11±.01	0.14±.04	0.12±.01	0.14±.02
HYDROXYCINNAMIC	0.68	0.98	1.08	1.31
Tyrosol	0.57±.03	0.79±.12	1.37±.05	2.33±.16
Syringaldehyde	0.20±.01	0.24±.10	1.08±.04	1.43±.08
OTHER NONFLAV.	0.77	1.03	2.45	3.76
NONFLAVONOIDS	3.04	6.72	8.38	10.52
Catechin	1.05±.04	3.69±.29	4.40±.24	4.48±.76
Epicatechin	0.71±.13	0.70±.05	1.46±.10	2.80±.05
FLAVAN-3-OLS	1.76	4.39	5.86	7.28
Quercetin	0.11±.01	0.14±.01	0.40±.01	0.50±.17
FLAVONOLS	0.11	0.14	0.40	0.50
FLAVONOIDS	1.87	4.53	6.26	7.78

25 °C). The correlation coefficients obtained were significant ($P < 0.05$) at the three temperatures for protocatequic acid, vanillic acid, caffeic acid, syringic acid, tyrosol, syringaldehyde, catechin, epicatechin, quercetin, hydroxybenzoic acids, hydroxycinnamic acids, other non-flavonoids, flavan-3-ols and flavonols, while those of other compounds (gallic acid, p-hydroxybenzoic acid and m-hydroxybenzoic acid) were only significant at one or two temperatures, and those of p-coumaric acid and ferulic acid were not significant at any temperature.

Fig. 1 shows a plot of the slopes of the above mentioned regression lines (extraction rates; Table 3, a) against maceration temperature. On comparing the extraction rates of compounds from the same fraction, for each temperature it is seen that, among hydroxybenzoic acids, they decrease in the following order: p-hydroxybenzoic acid > m-hydroxybenzoic acid > vanillic acid > protocatequic acid > gallic acid. However, some of the slopes in this group correspond to lines with not significant correlation coefficients (e.g. gallic acid at 10 °C, p-hydroxybenzoic acid at 15 °C and m-hydroxybenzoic acid at 25 °C). Within the hydroxycinnamic acids fraction, the extraction rate was higher for syringic acid than for caffeic acid at 10 and 15 °C, no comparison can be

Table 1 (cont.)

COMPOUNDS & FRACTIONS	MACERATIONS 25 °C			
	4 HOURS	16 HOURS	24 HOURS	48 HOURS
Gallic	0.10±.01	0.16±.04	0.19±.01	0.20±.02
Protocatequic	0.17±.04	0.37±.14	0.47±.13	1.22±.04
p-Hydroxybenzoic	0.36±.05	1.39±.06	1.89±.03	1.98±.03
m-Hydroxybenzoic	1.20±.01	1.66±.06	1.73±.06	1.87±.18
Vanillic	0.55±.29	1.34±.05	1.43±.01	1.49±.24
HYDROXYBENZOIC	2.38	4.92	5.71	6.76
Caffeic	0.33±.16	0.31±.05	0.40±.01	0.75±.01
Syringic	0.36±.06	0.40±.02	0.38±.10	0.58±.05
p-Coumaric	0.11±.01	0.13±.03	0.25±.01	0.43±.03
Ferulic	0.13±.01	0.18±.01	0.27±.01	0.38±.01
HYDROXYCINNAMIC	0.93	1.02	1.30	2.14
Tyrosol	0.67±.09	0.85±.02	1.61±.03	3.93±.05
Syringaldehyde	0.21±.05	0.48±.08	1.29±.04	1.83±.02
OTHER NONFLAV.	0.88	1.33	2.90	5.76
NONFLAVONOIDS	4.19	7.27	9.91	14.66
Catechin	1.35±.44	2.87±.30	4.43±.25	8.81±.28
Epicatechin	1.52±.06	2.64±.39	2.03±.30	7.26±.87
FLAVAN-3-OLS	2.87	5.51	6.46	16.07
Quercetin	0.11±.03	0.13±.04	0.30±.09	0.71±.03
FLAVONOLS	0.11	0.13	0.30	0.71
FLAVONOIDS	2.98	5.64	6.76	16.78

from 15 to 25 °C. This strong dependency on the temperature is consistent with the findings of other authors (PALLOTTA and CANTARELLI 1979).

Table 4 lists the results obtained by discriminant analysis using the direct method for the different phenolic groups under the different temperature and time conditions. Fig. 2 shows the first two discriminant functions, which jointly account for 98,52 % of the total variance. The discriminant function 1 accounts for over 95 % of the variance, so excerting a main discriminant power. As can be seen, the different maceration conditions can be classified into various groups according to function 1.

Group 1 includes the samples macerated for 4 h at the different temperatures and the unmacerated must (T), the centroid of which lies very close to that corresponding to the mildest maceration conditions; this indicates that the amount of polyphenols extracted after maceration at 10 °C for 4 h was minimal.

Group 2 is made with the samples macerated for 16 h at the three temperatures. Note that, according to discriminant function 1, this group includes the samples macerated at 10 °C for 24 h, which indicates that the amount of polyphenols extracted under

Table 3

Correlation between concentration and maceration time for the phenolic compounds and fraction ($y = a x + b$)

Corrélation entre concentration et durée de la macération pour les composés phénoliques et leurs fractions ($y = a x + b$)

COMPOUNDS & FRACTIONS	10 °C a $x10^{-4}$	10 °C b $x10^{-2}$	10 °C r	15 °C a $x10^{-4}$	15 °C b $x10^{-2}$	15 °C r	25 °C a $x10^{-4}$	25 °C b $x10^{-2}$	25 °C r
GALLIC	6.6	9.3	.698	15.2	11.5	.910	21.4	11.2	.902
PROTOCATEQUIC	58.8	11.5	.960	110.3	8.4	.996	230.0	4.2	.983
p-HYDROXYBENZOIC	237.2	59.5	.912	316.8	73.1	.785	355.2	56.6	.887
m-HYDROXYBENZOIC	174.8	16.8	.897	287.9	40.8	.857	287.3	78.3	.760
VANILLIC	185.1	44.7	.864	183.0	52.7	.850	236.6	59.6	.839
HYDROXYBENZOIC Ac.	662.8	142.0	.904	913.5	186.7	.865	1130.8	210.1	.920
CAFFEIC	24.7	13.6	.916	50.9	18.0	.883	115.9	16.6	.950
SYRINGIC	50.5	15.9	.992	62.7	18.8	.977	71.2	24.3	.900
p-COUMARIC	1.2	9.5	.220	45.0	11.9	.904	71.2	7.3	.972
FERULIC	7.1	9.6	.786	7.2	10.8	.773	59.0	10.3	.990
HYDROXYCINNAMIC Ac.	83.7	48.8	.984	165.9	59.6	.946	317.4	58.6	.977
TYROSOL	241.8	37.6	.993	405.5	34.5	.989	728.4	15.1	.970
SYRINGALDEHYDE	93.0	13.6	.994	288.9	8.8	.930	371.3	10.4	.965
OTHER NONFLAVONOID	334.9	51.3	.994	694.4	43.4	.972	1101.6	25.7	.982
CATECHIN	713.7	136.0	.905	777.1	151.0	.854	1640.5	68.9	.994
EPICATECHIN	218.7	72.7	.972	450.3	45.1	.946	1265.1	50.8	.940
FLAVAN-3-OL	932.5	208.8	.931	1230.5	194.9	.958	2905.6	119.7	.982
QUERCETIN	43.8	9.3	.917	87.2	9.5	.921	126.1	4.3	.947
FLAVONOL	43.8	9.3	.917	87.2	9.5	.921	126.1	4.3	.947

r significant at 0.05 ..... 0.811
r significant at 0.01 ..... 0.917
r significant at 0.001 ..... 0.974

made with p-coumaric acid and ferulic acid as the correlation coefficients of their regression lines were not significant at any temperature. As far as the other-nonflavonoids fraction is concerned, tyrosol was extracted at a higher rate than syringaldehyde. Finally, catechin was extracted more rapidly than epicatechin in the flavan-3-ols fraction at all three temperatures.

Fig. 1 also shows plots of extraction rates of the phenol fractions against maceration temperature. As to be seen, again for each temperature, the extraction rates of these fractions decrease in the following order: flavan-3-ols > hydroxybenzoic acids > other nonflavonoids > hydroxycinnamic acids > flavonols.

On comparing the extraction rates at different temperatures, it is found that the extraction rate of all compounds increases with maceration temperature. However, such an increase is linear for some and exponential for others. Thus, flavan-3-ols (catechin and epicatechin) undergo a marked increase in their extraction rate in passing

Table 2

Variance analysis of the phenolic compounds and fractions · F-values calculated

Analyse de variance des composés phénoliques et leurs fractions · F calculée

COMPOUNDS & FRACTIONS	SOURCE OF VARIATION	
	TIME	TEMPERATURE
Gallic acid	8.53	9.48
Protocatequic acid	97.94	39.41
p-Hydroxybenzoic acid	82.02	6.13
m-Hydroxybenzoic acid	280.88	248.84
Vanillic acid	72.60	9.53
HYDROXYBENZOIC ACIDS	282.35	76.86
Caffeic acid	31.19	31.44
Syringic acid	33.82	13.60
p-Coumaric acid	31.01	34.19
Ferulic acid	25.30	56.54
HYDROXYCINNAMIC ACIDS	81.89	70.30
Tyrosol	483.85	115.42
Syringaldehyde	589.33	236.58
OTHER NONFLAVONOIDS	995.22	287.27
Catechin	249.33	28.90
Epicatechin	117.64	106.84
FLAVAN-3-OLS	323.38	112.84
Quercetin	52.46	9.05
FLAVONOLS	52.46	9.05
Degrees of freedom	4/30	2/30
Significant at 0.05	2.69	3.32
Significant at 0.01	4.02	5.39

these conditions was virtually the same as that obtained by maceration at 15 °C for 16 h. Just the former conditions were those yielding the largest amount of extracted terpenes in must from *Vitis vinifera* Pedro Ximenez (MOYANO *et al.* 1990).

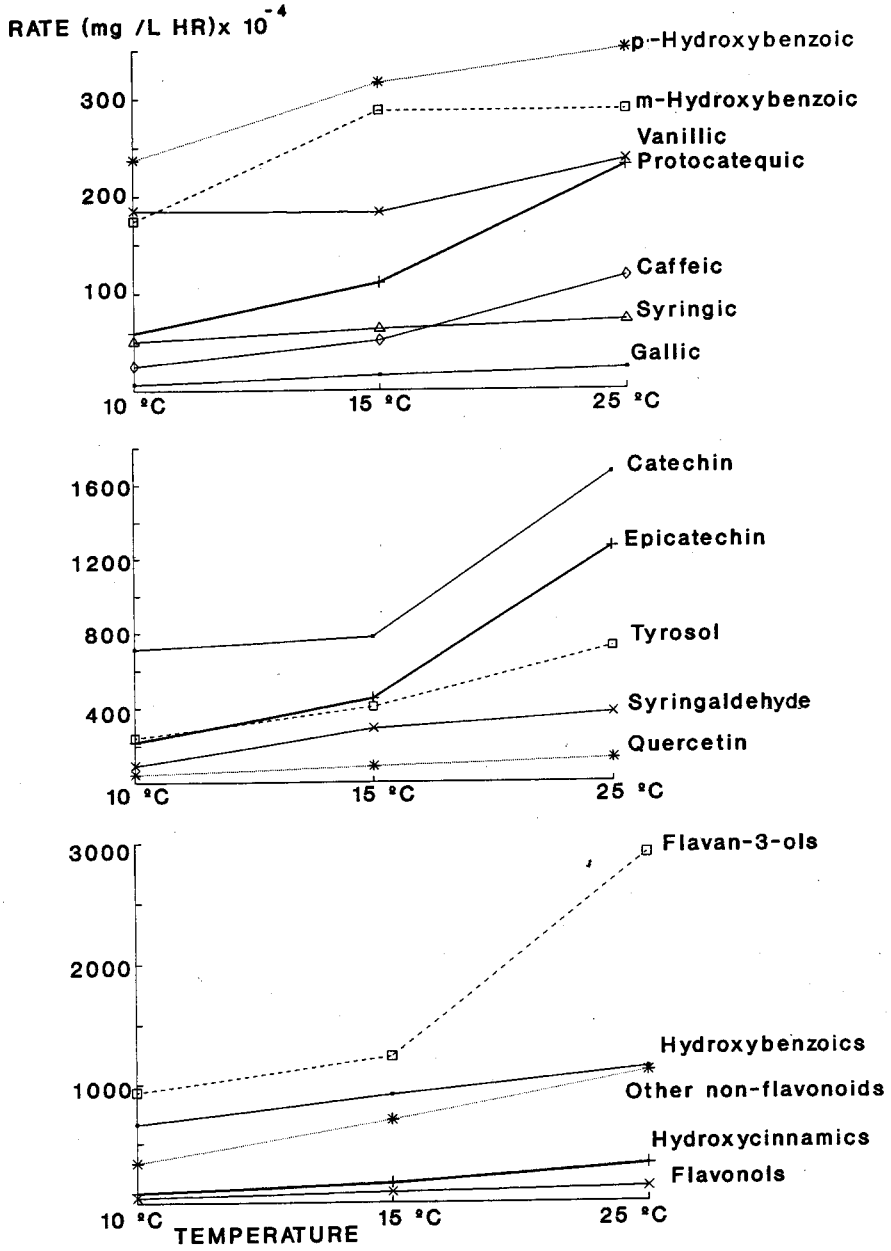

Fig. 1: Changes in the extraction rates of phenolic compounds and fractions as a function of temperature.

Variation de la vitesse d'extraction des composés phénoliques et leurs fractions en fonction de la température.

Table 4

Discriminant power and standardized discriminant function coefficients obtained

Pouvoir discriminant et coéfficients des fonctions discriminantes standardisées obtenues

DISCRIMINANT FUNCTION	EIGENVALUE	RELATIVE PERCENTAGE	CANONICAL CORRELATION
1	565.18	95.75	0.999
2	16.34	2.77	0.971

FUNCTIONS DERIVED	WILKS LAMBDA	CHI-SQUARE	DF	SIG. LEVEL
0	4.4×10^{-6}	419.25	70	0.000
1	2.4×10^{-3}	203.72	52	0.000

VARIABLE	FUNCTION 1	FUNCTION 2
HYDROXYBENZOIC ACIDS	0.644	-0.410
HYDROXYCINNAMIC ACIDS	0.230	-0.508
OTHER NON FLAVONOIDS	1.069	0.645
FLAVAN-3-OLS	0.430	-0.899
FLAVONOLS	0.515	1.043

The next group includes the most energetic maceration conditions. As can be seen, after 24 h of maceration the temperature exerts more influence on the extracted amount of polyphenols in the must, which was greater in maceration at 15 °C for 24 h than at 10 °C for 48 h. This greater influence of the temperature results in a marked separation between the most extreme maceration conditions (48 h at 15 °C and 48 h at 25 °C).

In conclusion, as temperature does not seem to be too influential at maceration times of 4 and 16 h, the latter time appears to be the advisable limit for the maceration time unless the must is subjected to subsequent oxidation. At these maceration times, the precise temperature will be dictated by economic reasons.

Flavan-3-ols (catechin and epicatechin) were the compounds extracted in the highest proportions and at the highest rates under all of the assayed conditions. However, the concentrations of other fractions (e.g. hydroxybenzoic acids) were similar of those of the flavan-3-ols in many cases and, although these are reknown active browning compounds, other phenol fractions may equally contribute to some extent to browning. It would be interesting to investigate more deeply the activity of the different phenol fractions as potential browning agents for white wines.

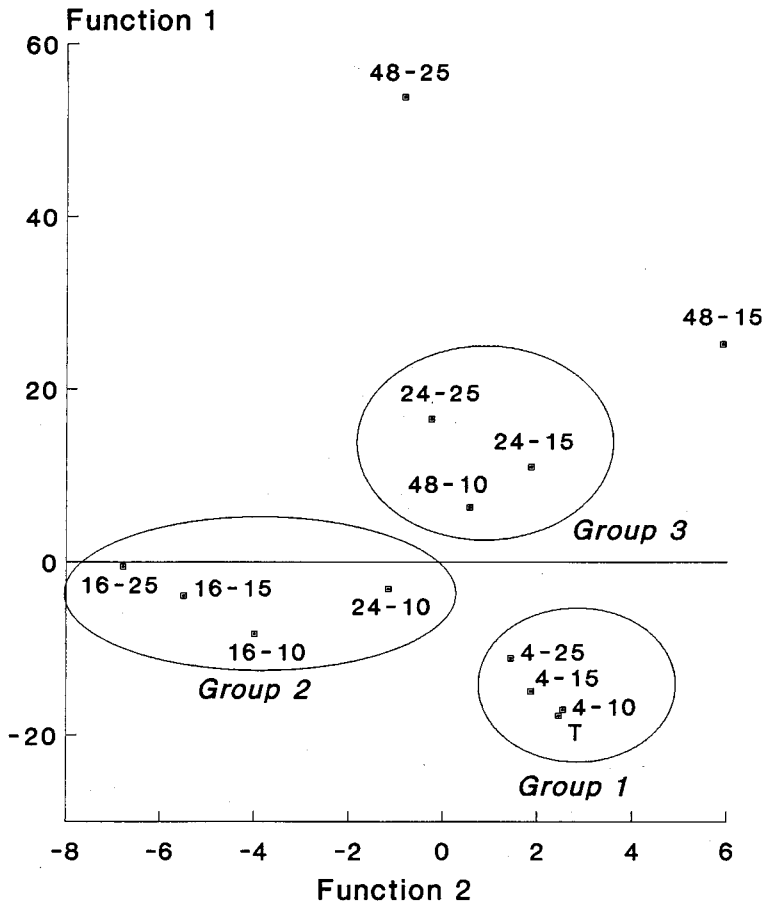

Fig. 2: Graphical representation of maceration conditions (time-temperature) in the discriminant plane.

Représentation graphique des conditions de macération (temps-température) sur le plan discriminant.

Summary

Crushed grapes of cv. Pedro Ximenez were macerated together with their must for 4, 16, 24 and 48 h at temperatures of 10, 15 and 25 °C. The musts obtained after pressing were used for the determination of 14 phenolic compounds from the following fractions: hydroxybenzoic acids, hydroxycinnamic acids, other nonflavonoids, flavan-3-ols and flavonols. The temperature was found not to exert a marked influence on the extraction of the phenolic compounds in the first 16 h of maceration. After that time, however, it had a significant effect, so maceration times longer than 16 h are inadvisable unless subsequent oxidation is applied. The analytical results obtained showed that the flavan-3-ols, catechin and epicatechin were extracted at the highest rates, related with maceration temperature, followed by hydroxybenzoic acids, other nonflavonoids, hydroxycinnamic acids and flavonols. The flavan-3-ol fraction was also extracted in higher proportions under the different conditions assayed, though very closely followed by that of hydroxybenzoic acids.

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