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Standardisation of the chromatic characteristics of *sobretablas* wine macerates obtained by an accelerated ageing technique using heating and oak shavings

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Abstract

This paper presents the results of the selection and chromatic characterisation of macerates obtained from *sobretablas* wines and oak shavings, aged according to the method proposed and studied by our research group. This method allows considerable shortening of the time spent in barrels and represents a viable alternative to the oxidative ageing of *oloroso* wines from the "Jerez-Sherry" Origin Appellation (Spain). A detailed study was undertaken of the influence on chromatic characteristics of such factors as heating, maceration and exhaustion of the shavings (using water and hydroalcoholic mixtures). By using caramel colouring additive (authorised in the European Union as E-150), colour values entirely equivalent to those of commercial wines are achieved, which are not even apparent under sensorial analysis.

Keywords: Accelerated ageing; Wine; Jerez-Sherry; Colour; Caramel colouring.

1. Introduction

In Jerez, wine is aged by a unique process by which, unlike all the other wine-growing regions of the world, it is transferred from one barrel to another several times. This system, known as *criaderas* and *soleras* allows practically unlimited conservation of a particular type of wine with uniform characteristics (taste, bouquet, colour and sensory characteristics) that do not depend on factors such as the quality of the harvest, rate of consumption, etc. However, the one main disadvantage of the technique is its relative expense, which makes it impossible to lower the prices of these wines (Barbadillo, Peñin, Lopez & Vasserot, 1987).

Of the broad range of methods proposed for shortening the ageing period of alcoholic beverages, the most common are those involving the use of oak wood extracts obtained by decoction, infusion, leaching or maceration (Maga, 1989; Puech & Sarni, 1990) with or without prior treatment of the wood by physical methods such as ultrasound, high pressure or charring, or chemical methods such as the application of acids or alkalis (Bredemberg, Huska & Vuori, 1987; Puech, 1989; Tanahashi, Karina & Tamabuchi, 1989).

Our technique, based on the traditional charring process used in cooperage, causes thermodegradation of the wood and the formation of different families of compounds (phenolic acids, furanic and phenolic aldehydes, phenylketones, etc.) through thermal decay of the polyosides and lignin (Chatonnet, Boldrou & Dubourdieu, 1993; Puech & Moutounet, 1992; Sarni, Moutounet, Puech & Rabier, 1990).

The chromatic characteristics are one of the parameters requiring particular attention in this process. Optimisation of methods for colour measurement and analysis of the compounds responsible for colour is a widely studied subject in the field of wine (Ayala, Echavarri & Negueruela, 1997; Brouillard & Dangles, 1994; Gómez-Cordobés, Junquera & Estrella, 1995; Heredia & Guzmán Chozas, 1992; Heredia, Troncoso, Guzmán Chozas, 1997; Larrauri, Ruperez & Saura Calixto, 1997; Ortega, Hernandez-Agero, García de la Peña, Hidalgo Togores, Tienda Priego, Navarro Rozalén & Serran Cuadradillo, 1994).

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Taking, as a basis, previous studies on accelerated ageing, (Giménez, Martinez, Lopez, Villalon, Quesada & Lopez, 1996; Monedero, Olalla, Quesada, Lopez, Lopez & Carmen, 1998; Quesada, Granados, Villalon, Lopez & Lopez, 1996), we have examined the influence on chromatic characteristics of factors such as the heating, maceration and exhaustion of oak shavings (with hydroalcoholic mixtures and with water) in order to obtain chromatic parameters as close as possible to those of the commercial *oloroso* wines (subjected to oxidative ageing) of the Jerez-Sherry area (Spain).

After addition of caramel colouring (E-150), none of the chromatic parameters showed any statistically significant differences from those of the commercial *oloroso* wines. A study was therefore carried out using increasing amounts of the colouring agent and a linear regression equation was calculated to decide the amount required according to the initial colour of the macerate.

2. Materials and methods

2.1. Samples analysed

2.1.1. Commercial wines

In all, 18 dry *oloroso* wines from the Jerez-Xeres-Sherry region were analysed, all obtained from the market.

2.1.2. Macerate preparation

The macerates were prepared using American oak (*Quercus alba*) shavings in 2% proportion in *sobretablas* wine provided by a winemaker from the region. Shaving size was 3 to 5 mm which, according to Giménez et al. (1996), is the ideal size for optimum extraction of phenolic compounds from the wood using hydroalcohol mixtures. The shavings were heated to 180°C for 3, 6 and 15 h. An exhaustion treatment was also tested using water and 35 and 60% hydroalcohol dissolutions, in order to lower the high levels of phenolic compounds (e.g. gallic acid, syringaldehyde or synapaldehyde) in some of the macerates in comparison with commercial *oloroso* wines (Monedero, 1998). Twelve macerates were obtained (Table 1) and in all cases maceration in *sobretablas* wine lasted 3 months.

2.2. Chromatic characteristics

CIE tristimulus coordinates method (C illuminant): abcissa (x), ordinate (y), luminosity (Y), purity and dominant wavelength (λ_d); colour intensity (CI), tonality, Saudraud index, Glories (420, 520 and 620%) and *CIELAB space indices*: Lightness (L), coordinates a^* and b^*, Chroma (C^*) and Hue (H^*) (Amerine & Ough, 1976; EU, 1990; Glories, 1984; OIV, 1990; Ribereau-Gayon, Peynaud, Sudraud & Ribereau-Gayon, 1980).

Table 1
Characteristics of the macerates obtained by the accelerated ageing process

Macerate no.	Charring time	Wood treatment
1	3	—
2	6	—
3	15	—
4	3	Exhaustion 60% alcohol
5	6	Exhaustion 60% alcohol
6	15	Exhaustion 60% alcohol
7	3	Exhaustion 35% alcohol
8	6	Exhaustion 35% alcohol
9	15	Exhaustion 35% alcohol
10	3	Exhaustion water washed
11	6	Exhaustion water washed
12	15	Exhaustion water washed

2.3. Analytic method

Direct spectrophotometric measurement, using a Perkin–Elmer 551s UV/VIS spectrophotometer and quartz cuvets 1 and 0.5 cm thick prior to centrifuging of the sample, was employed.

3. Results and discussion

3.1. Chromatic characterisation

Table 2 shows the statistical summary of the results obtained after calculating the main chromatic values of the wines subjected to traditional oxidative ageing and the *sobretablas* wine macerates with previously heated oak shavings.

Study of these data shows that there are clear chromatic differences between the two groups. The *oloroso* wines from the Jerez-Sherry region have a mean dominant wavelength of 578.6 nm (minimum 572.5 nm and maximum 589.06 nm), which places them in the yellow zone of the Spectrum Locus diagram according to the zonal denominations proposed by Kelly (Lozano, 1979). On the other hand, the macerates with a dominant mean wavelength of 548.5 nm (in a range of 517.0 to 563.6 nm) would be represented by the yellowish-green zone of the same chromaticity diagram.

As regards the luminosity values, i.e. the area included within the curve in the dominant wavelength zone, the commercial wines have clearly lower mean values than the macerates (49.1 as against 78.2), meaning that the latter are much more transparent. Differences in percentage purity (height of curve in the zone of dominant tone or wavelength) values are even more outstanding, with mean values of 18.9 in macerates as against 71.74 in commercial *oloroso* wines (minimum 54.71, maximum 96.6).

Table 2
Statistical summary of the chosen chr omatic parameters in samples of commercial *oloroso* wines and *osobretablas* macerates

	Olorosos wines					*Sobretablas* macerates				
	Count	Average	S.D.	Variance	Range	Count	Average	S.D.	Variance	Range
Abcissa	18	0.4498	0.0309	9.50E-4	0.0974	12	0.3540	0.0122	1.50E-4	0.0343
Ordinate	18	0.4488	0.0154	2.37E-4	0.0580	12	0.3804	0.0175	3.08E-4	0.0614
Luminosity	18	49.0953	9.0886	82.6019	28.8060	12	78.2043	5.3216	28.3193	19.1842
C.I.	18	2.2523	0.6400	0.4096	2.1660	12	0.7430	0.1700	0.0289	0.526
Sudraud	18	3.8023	0.2912	0.0848	1.0870	12	4.2174	0.7833	0.6136	2.2168
Tonality	18	−52.1156	8.1082	65.7437	28.7312	12	−24.0481	5.5788	31.1232	16.1068
C′.I.	18	2.4115	0.6888	0.4744	2.3310	12	0.8117	0.1860	0.0346	0.5550
λd	18	578.5780	3.3862	11.4665	16.5600	12	548.5480	14.0969	198.7240	46.6100
Purity	18	71.7433	12.4033	153.8410	41.8900	12	18.9058	6.5284	42.6203	22.0000
420(%)	18	74.1112	1.8703	3.4979	6.6140	12	73.7516	5.7855	33.4726	16.3683
520 (%)	18	19.5663	1.0489	1.1002	3.9220	12	17.9030	2.4497	6.0010	7.0468
620 (%)	18	6.3225	0.9372	0.8784	3.4250	12	8.3454	3.3621	11.3036	9.6498
L	18	75.1674	5.7336	32.8743	18.9597	12	90.8156	2.4496	6.0006	8.8324
*a**	18	0.5810	1.1478	1.3174	3.6659	12	−1.6889	0.4978	0.2478	1.9526
*b**	18	14.6846	2.9113	8.4757	12.0819	12	6.3754	1.6770	2.8122	5.4396
*C**	18	14.7316	2.9367	8.6241	1.21146	12	6.6026	1.7185	2.9532	5.7755
*H**	18	88.4517	4.2542	18.0986	13.5700	12	109.1117	5.4475	29.6749	8.3600

Relative absorbance, proposed by Glories (1984), showed that, in both groups of samples, yellow is the predominant colour (absorbance at 420 nm), which is even higher than the sum of red and blue (520 and 620%).

These differences are even more noticeable in the CIELAB space. According to the hue values (*H**), the *oloroso* wines made by the traditional technique are light brown (values below 90°) and the macerates greenish yellow (values above 100°). Moreover, on the basis of the colour shading classification of white wines published by Ortega, Hernandez-Agero (1994), we found that, in this space, the macerates with negative *a** values (green), positive *b** values and *L* (lightness) values above 90% had a greenish straw-coloured hue. On the other hand, the commercial wines with predominance of red (positive *a** values), positive *b** values and *L* values above 50% were chromatically characterised as wines with an old gold hue.

In order to determine the statistical significance of these differences, the results were subjected to a variance analysis using the Statgraphics® (U.6.0.) statistics programme.

After application of the Kolmogorov–Smirnov test (normal or abnormal distribution of samples) and the Bartlett test (homogeneity of variances), we applied a parametric or non-parametric method, such as the Kruskall–Wallis test, if one or both of the postulates failed (significant results $P < 0.05$). Table 3 shows that, except for three variables (420%, 620% and Sudraud), the difference is statistically significant.

3.2. Caramel addition

In order to obtain macerates with similar colour parameters to those of commercial *oloroso* wines, we added caramel (E-150 colouring agent) in authorised amounts.

E-150 is soluble in water and hydroalcoholic mixtures and, according to the Spanish Ministry for Health and Social Security (Ministerio de Sanidad y Seguridad Social, 1981), its use is authorised in many food products intended for human consumption, such as soft drinks and beer, as well as sweetstuffs and many other foods. According the Annex VI of the EU (1987) Directive on admissible winemaking practices and treatments, caramel addition is permitted. Moreover, in the EU (1997) Directive, this addition is also permitted with the aim of darkening the colour of *liqueur wines* and *vlcprd*.

In order to establish the amount of additive for each macerate and taking one of them as reference, increasing amounts of caramel (1, 2, 3, 4 5 and 7 g/l) were added and the absorbances of the macerates measured at different wavelengths. A chart was then drawn up of the increases in absorbance between the original macerate and the macerate with the different concentrations of added caramel, giving the straight line calibrations shown in Table 4, all of which have correlation coefficients higher than 0.999.

On the basis of these straight-line calibrations and taking, as reference, the mean value of the A_{420} of the commercial samples, we proceeded to add the corresponding

Table 3
Variance analysis (P values) applied to the two groups of samples studied before and after addition of caramel colouring to the *sobretablas* macerates

	Before addition of colouring			After addition of colouring		
Variable	Anova	Kruskall–Wallis	Significance	Anova	Kruskall–Wallis	Significance
Abcissa	—	4.83×10^{-6}	SI	—	0.1500	NO
Ordinate	0.000	—	SI	—	0.1755	NO
Tonality	0.000	—	SI	—	0.9325	NO
λ_d	—	4.80×10^{-6}	SI	—	0.2706	NO
Purity	—	4.83×10^{-6}	SI	—	0.0105	NO
Luminosity	0.000	—	SI	—	0.0940	NO
C′.I.	—	4.83×10^{-6}	SI	—	0.1755	NO
Sudraud	—	0.01989	NO	—	0.2710	NO
420 (%)	—	0.2358	NO	—	0.0110	NO
520 (%)	—	7.65×10^{-3}	NO	—	0.2195	NO
620 (%)	—	0.075	NO	—	0.1172	NO
L	—	4.83×10^{-6}	SI	—	0.2896	NO
a^*	—	4.83×10^{-6}	SI	—	0.1384	NO
b^*	0.000	—	SI	—	0.1384	NO
C^*	0.000	—	SI	—	0.1993	NO
H^*	—	0.000138	SI	—	0.3093	NO

Table 4
Regression equations obtained from the increase in absorbance at different wavelengths after addition of increasing amounts of caramel colouring

	Media	Minimum	Maximum	S.D.	R.D. (%)	95% Confidence	99% Confidence	a	b	r
ΔA_{420}	0.8898	0.315	1.431	0.465695	0.208265	0.4082	0.537324	−0.008794	0.2927	0.999842
ΔA_{445}	0.7801	0.222	1.529	0.470413	0.192945	0.376409	0.495477	−0.00708	0.2087	0.99995
ΔA_{495}	0.4011	0.114	0.786	0.242174	0.098867	0.193779	0.225077	−0.004108	0.1070	0.99995
ΔA_{520}	0.291	0.082	0.571	0.176129	0.071905	0.140933	0.185514	−0.003746	0.07781	0.99992
ΔA_{550}	0.2026	0.057	0.397	0.122696	0.050091	0.098178	0.129234	−0.002708	0.0542	0.99982
ΔA_{620}	0.089	0.024	0.176	0.0548	0.022373	0.04385	0.057721	−0.002687	0.0242	0.99968
ΔA_{625}	0.0858	0.023	0.171	0.05302	0.02164	0.042427	0.055847	−0.002857	0.02341	0.99945

amount of caramel. Absorbance at 420 nm was chosen as it has the most effect on the colour of these wines, although the results would scarcely differ if another wavelength were chosen.

The statistical summary of the chromatic parameters obtained after addition of caramel colouring is shown in Table 5. It can easily be seen that values much closer to those of the commercial *oloroso* wines from Jerez were obtained.

Figs. 1 and 2, which represent the three groups of samples in the CIE and CIELAB spaces studied in this paper, also show that the macerates with added caramel colouring shift towards the zone of the commercial *oloroso* wines aged by traditional methods, becoming indistinguishable from them.

Table 3 shows the results of another variance analysis undertaken in the same conditions as the previous one in order to determine whether the influence of the addition of the colouring agent was statistically significant. These results show, statistically, that the addition of caramel produces macerates whose chromatic parameters have no significant difference from those of the commercial samples, although these differences were present in most parameters prior to the addition.

Similarly, a discriminant analysis (Table 6) was applied to both groups of samples using the basic coordinates of the CIELAB space (L^*, a^* and b^*) as discrimination variables. It was found that an equation could not be established, with these variables, that classified the entire group of macerates after addition of caramel (Fig. 3). Discrimination was possible before addition of the colouring agent.

3.3. Sensory analysis

In order to make a sensory characterisation of the chromaticity of the *oloroso* wines and the macerates prepared by our method of accelerated ageing and to verify the lack of visual differences after the addition of caramel colouring, we selected four of the commercial samples and four macerates. In the case of the former, selection was based on the mean values of the physical–chemical

Table 5
Statistical summary of the chosen chromatic parameters in macerates after addition of caramel colouring

	Count	Average	S.D.	Variance	Range
Abcissa	12	0.4353	0.0045	2.03E-5	0.0149
Ordinate	12	0.4441	0.0010	9.87E-7	0.0034
Luminosity	12	51.9293	0.4713	0.2222	1.5550
C.I.	12	2.0075	0.0230	5.29E-4	0.0800
Sudraud	12	4.0220	0.1400	0.01961	0.5606
Tonalidad	12	−51.1464	0.2169	0.0470	0.6494
C′.I.	12	2.1525	0.0216	0.0005	0.0757
λ_d	12	577.6440	0.4268	0.1822	1.0300
Purity	12	59.9042	2.0970	4.3973	6.1300
420(%)	12	74.9849	0.5798	0.3362	2.3699
520(%)	12	18.6596	0.4799	0.2303	1.8988
520(%)	12	6.3728	0.2243	0.0503	0.6720
L^*	12	77.1773	0.3799	0.1443	1.3250
a^*	12	−0.0378	0.2805	0.0787	1.0300
b^*	12	13.4065	0.294	0.0862	0.9000
C^*	12	13.4093	0.2936	0.0887	0.9205
H^*	12	90.0790	1.1727	1.3752	4.2691

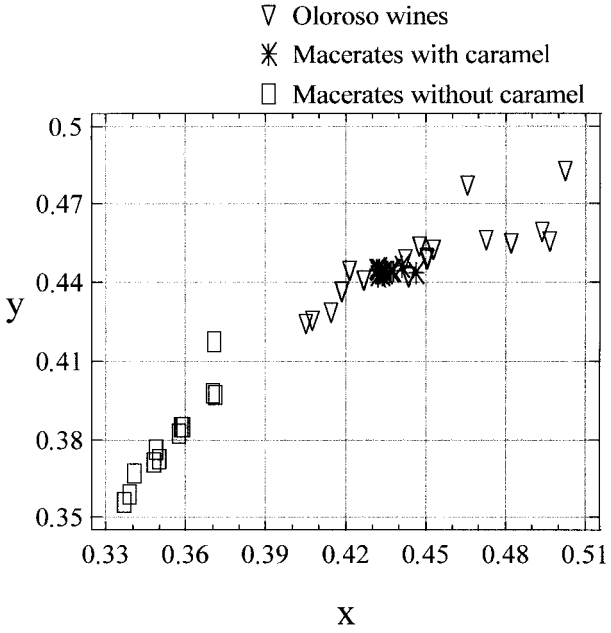

Fig. 1. Representation of commercial *oloroso* wines and macerates before and after addition of caramel colouring on the *x* and *y* coordinates of the CIE chromaticity diagram.

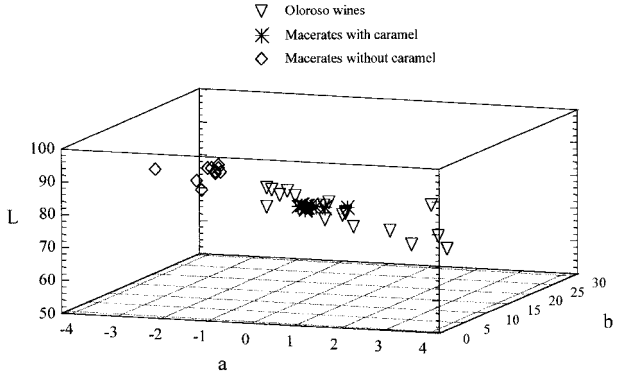

Fig. 2. Representation of samples analysed in the tridimensional space of the Lab parameters of the CIELAB space.

parameters analysed in previous studies and also on criteria of cost/quality, while the four macerates (numbers 1, 4, 7 and 10) were those most similar in polyphenol content and physical–chemical characteristics to the selected commercial samples (Monedero, Olalla, Martín-Lagos, Lopez Garcia, Lopez & Carmen, 1999; Monedero et al., 1998).

Table 7 shows the hedonistic scores for the chosen samples, all of which, both commercial and macerates, were classified higher than Correct and, in some cases, close to Good for Fluidity, Tonality and Vivacity. Moreover, variance analysis showed that there were no differences in the values of the three visual parameters between the macerates and the commercial wines that could have distinguished one sample group from the other.

3.4. Sensory profile

Using the same tasting questionnaire, a sensory profile of the samples was built up (Fig. 4). Eleven descriptors were used for the visual analysis, six for Hue (tawny brown, light brown, old gold, straw-coloured, golden yellow and pale yellow) and five for Appearance (turbid, matt, transparent, bright and very bright), all chosen from previous studies on this type of wine.

The predominant descriptors were obtained from this profile, following simplification criteria proposed by authors such as Cantagrel and Lavergne (1989) or Damasio and Costell (1991), based on frequency of mention. Descriptors that were not mentioned by at least four of the 12 judges in more than half of the samples tasted (more than four) were discarded.

Table 6
Discriminant analysis applied to the groups under study using the *L*, *a* and *b* coordinates of the CIELAB space as classification variables[a]

Discriminant function	Eigenvalue	Relative percentage	Canonical correlation	
1	3.98789	98.65	0.89416	
2	0.054566	1.35	0.22747	
Functions derived	**Wiks lambda**	**Chi-square**	**DF**	***P*-value**
1	0.190112	63.0854	6	0.0000
2	0.948257	2.0189	2	0.3644
Classification function coefficients				
	Oloroso Wine	*Macerate*[b]	*Macerate*[c]	
a	36.4536	35.6399	37.3844	
b	10.776	10.5824	9.56274	
L	13.4058	13.3501	14.1607	
Constant	−593.685	−586.521	−643.016	
		Predicted type		
Type	Group size	Oloroso wine	Macerate[b]	Macerate[c]
Oloroso wine	18	11 (61.11%)	7 (38.89%)	0 (0.00%)
Macerate[b]	12	1 (8.33%)	11 (91.67%)	0 (0.00%)
Macerate[c]	12	0 (0.00%)	0 (0.00%)	12 (100.00%)

[a] Percent of cases correctly classified: 80.95%.
[b] Macerate with caramel colouring.
[c] Macerate without caramel colouring.

The samples analysed were found to have a colour hue ranging from golden yellow to tawny brown. In comparison with the colour values obtained in commercial samples, after application of different chromatic parameters, and specifically the CIELAB space, we obtained mean values of these parameters that characterised wines as having a dark golden hue, which is very similar to that obtained in almost all the commercial samples after sensory analysis (old gold–light brown).

The colour hues of the macerates ranged from golden yellow to tawny brown, although the predominant hue was also old gold.

Table 7
Results of the application of sensory analysis (visual examination) to the chosen samples of commercial wines (C) and macerates (M)

Sample	Fluidity	Tone	Vivacity
C1	7.16	7.83	6.91
C2	7.25	7.50	7.16
C3	7.25	7.75	7.58
C4	7.33	7.91	7.83
M1	7.25	7.25	6.66
M2	7.66	7.75	7.25
M3	6.75	7.25	6.75
M4	7.50	7.58	7.41
Average	7.26	7.60	7.19
S.D.	0.26	0.25	0.40
V.C.(%)	3.65	3.33	5.67
Range	0.75	0.66	1.17

Regarding the appearance of the sample analysed, we can conclude that almost all the samples were bright and transparent.

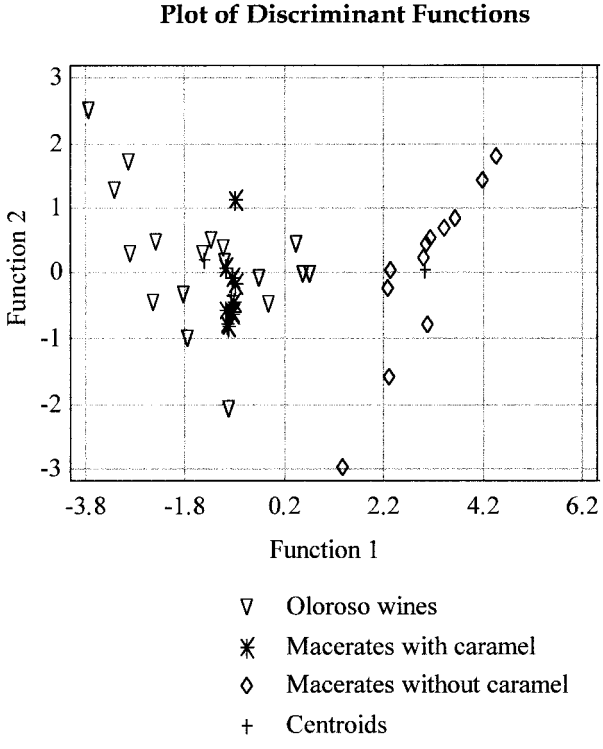

Fig. 3. Discriminant analysis applied to the groups under study using the *L*, *a** and *b** coordinates as classification variables.

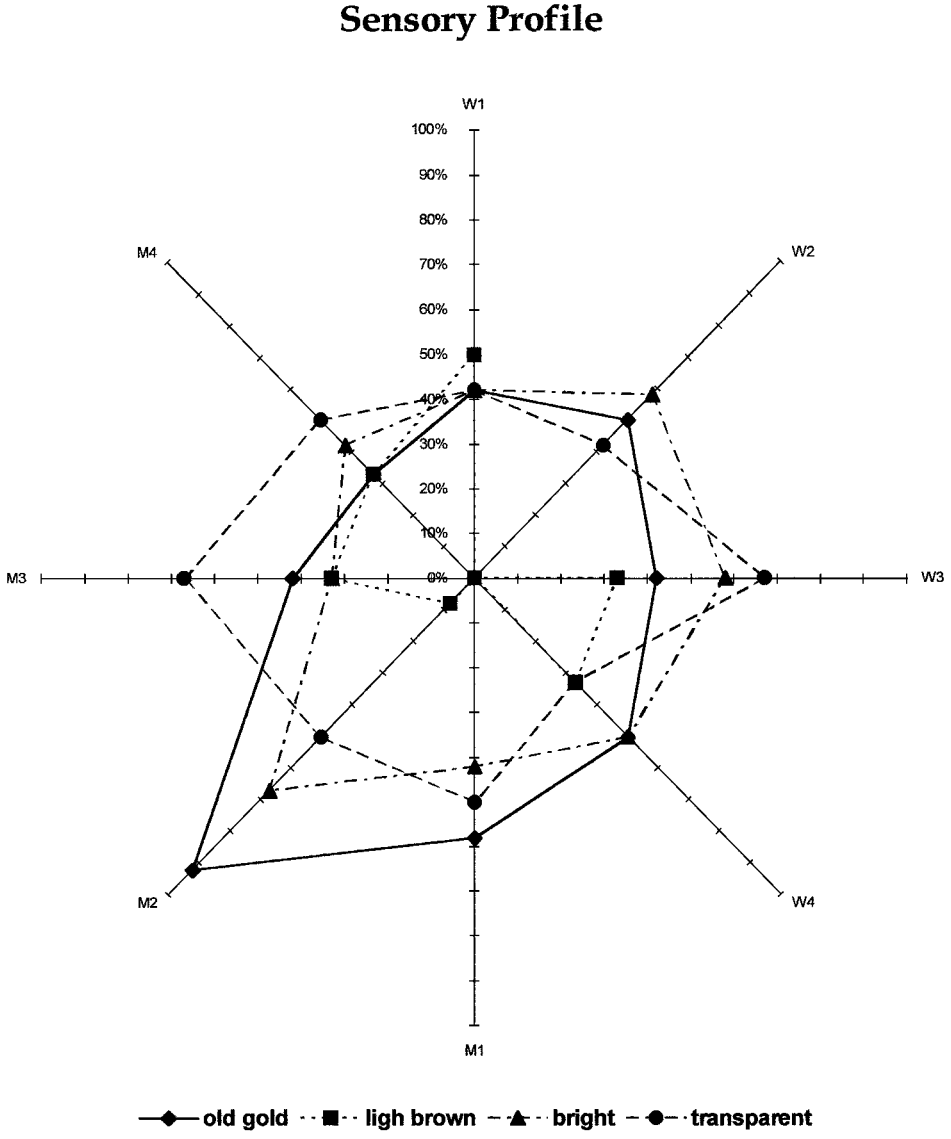

Fig. 4. Sensory profile of the visual analysis applied to the samples.

4. Conclusions

In our study we conclude that, by the addition of caramel colouring additive (authorised in the European Union as E-150) in quantities calculated from absorbance, chromatic values statistically similar to those of commercial Oloroso wines from the "Jerez-Sherry" Origin Appellation (subjected to oxidative ageing) can be achieved in macerates obtained from sobretablas wines and oak shavings previously charred, values which are not even apparent under sensorial analysis.

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