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**Improving the color and aging aptitude of Syrah wines in warm climate by wood-
grape mix maceration**

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ABSTRACT

The amelioration of aging aptitude of Syrah red wines from warm climate regions by adding oak wood chips to the grape maceration/fermentation has been developed. Our attention was focused on the tristimulus colorimetry, differential colorimetry and phenolic composition. This technique was applied with an eye toward improving color stabilization of wines in a warm climate region where common loss of color normally occurs. On the basis of the results, a color enhancement (by an increment of chroma, C^*_{ab}) and significant ($p < 0.05$) higher amounts of total polyphenols, anthocyanins, flavan-3-ols and esters of hydroxycinnamic acids was observed in Syrah red wines, favoring a significantly ($p < 0.05$) higher percentage of copigmentation and, hence, color stabilization. Finally, it could be affirmed that the joint addition of oak wood chips (both during fermentation and aging) induced visually perceptible color differences ($\Delta E^*_{ab} > 3$), mainly quantitative ($\Delta^2 L^*_{ab}$ and $\Delta^2 C^*_{ab}$), from the early months of storage.

KEYWORDS

American oak wood chips, polyphenolic compounds, CIELAB, differential colorimetry, aging, warm climate.

INTRODUCTION

Obtaining high quality red wines in warm climate regions is difficult because of the usual color instability over time due to the high temperatures. This fact is produced because both phenolic and technologic maturities do not coincide at the moment of harvesting as occurred in colder viticulture zones [1]. As a consequence, seeds remain unripen and the low quantities of pigments and copigments occurred [2], hampering copigmentation phenomena (which contribute to color stabilization). As a result, it is common to notice a fall of color after some months of storage in either bottle or barrels, showing difficulties of ageing the red wines elaborated in warm climate regions.

In the search for color stabilization, phenolic compounds are the main contributors of the quality of the wines, taking into account the color and astringency. Phenolic compounds such as anthocyanins (mainly in skins) were not only the responsible for the final color of wines, but flavonols, flavan-3-ols and other compounds (normally colorless) act as copigments, improving the color stabilization of aged wines by means of copigmentation reactions [3]. Whereas some other reactions involving anthocyanins could produce loss of color (oxidation, hydration or adsorption), the formation of polymeric pigments take importance and contribute to the stabilization of the wines [4].

With the objective of increasing the quantities of pigment concentrations (tending to reach an optimal color stabilization), implementation with tannins have been recently developed in warm climate, both derived from grape and wood. That is the case of Gordillo et al. [5], who studied the stabilization of the color by adding Pedro Ximenez white pomace to Spanish Syrah red wines during fermentation, or Baiano et al. [6] in a study about the addition of French oak wood chips to two Italian grape varieties (Montepulciano and Aglianico), or the research of a native Greek variety (Agiorgitiko) by using oak chips from different sources [7]. However, despite of this practice, color

losses normally occurred after a short period of time in warm climate winemaking, so researches looking for new alternatives are of particular interest. These evidences led us to think that, probably, the implement with tannins would be carried out at the initial points of the vinification, in order to assure sufficient tannin concentration during vinification. Thus, in that sense, our research group studied the addition of oak wood chips during fermentation of red grapes [8], obtaining positive results in terms of color stabilization. However, it could be hesitated if wines elaborated in that way could be appropriate for long-lasting aging.

Thus, this research work sought to scrutinize the capacity of aging of Syrah wines, the more cultivated grape variety in D.O. Condado de Huelva (southwestern of Spain) and normally employed to elaborate young wine. To date, this research study is the first attempt to deeply scrutinize the effect of aging wines with an implement with tannins (by the addition of oak wood chips) in the early stages of vinification in warm climate regions. Our interest was focused on the study of phenolic composition (anthocyanins, benzoic acids, hydroxycinnamic acid derivatives, flavan-3-ols and flavonols), chromatic characteristics by applying differential colorimetry, copigmentation and polymerization.

MATERIAL AND METHODS

Winemaking

Around 600 Kg of *Vitis vinifera* Syrah grapes were harvested in “Condado de Huelva” Designation of Origin, in the southwestern Spain, in a good maturity (12.4 °Brix). After destemming and crushing, the must were distributed in three stainless steel tanks of 220 L for skin maceration. 3 g/L of American oak (*Quercus alba*) wood low-toasted chips of 1 cm² average size (Tonelería Martín y Vázquez, Logroño, Spain) were added to the must, together with skins. Fermentation caps were punched down at each tank once a day during the on-skin maceration period, which lasted four days (fermentative

alcoholic fermentation). Alcoholic fermentation, and the subsequent malolactic fermentation, were correctly and spontaneously developed, guided by assays of density and enzymatic measurements of malic and lactic acid contents, respectively. Then, wines were racked to stainless steel tanks of 50 L and were kept for stabilization. After around four months (130 days), wines were racked again. Subsequently, two types of experiences were carried out: (i) three tanks were submitted to the additional treatment with 3 g/L of American oak wood medium-toasted chips during 40 days (CCp), and (ii) other three tanks contained wine with the only addition of oak wood chips during fermentation (C). Experiments were carried out in triplicate.

Oenological parameters

The official methods established by European Union were used to obtain the conventional oenological parameters such as pH, total and volatile acidity, reducing sugars, and free and total SO₂ [9].

Spectrophotometric color measurement

A Hewlett-Packard UV-vis HP8452 spectrophotometer (Palo Alto, CA) was used to determine the whole visible spectrum (380-770 nm) at constant intervals ($\Delta\lambda=2$ nm), using 2 mm path length glass cells and distilled water as reference. The CIELAB color parameters (L^* , a^* , b^* , C^*_{ab} , and h_{ab}) were determined by using the original software CromaLab© [10], following the Commission Internationale de l'Éclairage's, CIE, recommendations [11]: the CIE 1964 10° Standard Observer and the CIE Standard Illuminant D65. The L^* value (vertical axis) defines the lightness (property according to which each color can be considered as equivalent to a member of the gray scale, between black and white, with values ranging between 0 and 100, respectively). The parameters a^* and b^* are the colorimetric coordinates, which represent opponent red-green and blue-yellow axes. The parameters hue (h_{ab}) and chroma (C^*_{ab}) are defined

from L^* , a^* and b^* . Hue angle (h_{ab}) is the attribute according to which colors have been traditionally defined as red, yellow, etc. Besides, the chroma (C^*_{ab}) allows each hue to be determined by its degree of difference in comparison to a gray color with the same lightness. Moreover, these colorimetric parameters indicate a quantitative (L^* and C^*_{ab}) or qualitative (h_{ab}) contribution to color [12]. Euclidean distance between two points in the three-dimensional space define by L^* , a^* , and b^* were used for calculating color differences (ΔE^*_{ab}): $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

Previous adjustment to pH 3.6 using 1 M NaOH and HCl, the percentage contributions of copigmented anthocyanins (% copigmented pigments) to the total wine color and the degree of anthocyanin polymerization (% polymeric pigments) were determined [13]. Folin-Ciocalteu reagent was used for the analysis of total phenolics [14].

HPLC-DAD analysis of phenolic compounds in wines

An Agilent 1200 chromatographic system, equipped with a quaternary pump, and UV-vis diode-array detector, an automatic injector, and ChemStation software (Palo Alto, CA), was used to the HPLC separation, identification and quantification of phenolic compounds. Prior direct injection, the samples were filtered through a 0.45 μm Nylon filter (E0034, Análisis Vínicos, Tomelloso, Spain). All analyses were made in triplicate. The method proposed by Heredia et al. [15] was used for the anthocyanin identification. Acetonitrile-formic acid-water (3:10:87) as solvent A and acetonitrile-formic acid-water (50:10:40) as solvent B were used. The elution profile was as follows: 0-10 min 94% A - 6% B; 10-15 min 70% A - 30% B; 15-25 min 60% A - 40% B; 25-35 min 55% A - 45% B; 35-40 min 50% A - 50% B; 40-42 min 40% A - 60% B; 42-43 min 94% A - 6% B. The flow rate was 0.8 mL/min, and the injection volume was 50 μl . A reversed-phase column Zorbax C18 (250 x 4.6 mm, 5 μm particle size), thermostatted at 38 $^\circ\text{C}$, were used. UV-Vis spectra were recorded from 200 to 800 nm with a bandwidth of 2.0 nm.

The quantification was made at 525 nm by comparing the areas and the retention times with the malvidin 3-glucoside standard.

For the identification of the phenolic compounds (flavan-3-ols, flavonols, hydroxycinnamic acid derivatives and other low molecular weight phenolic compounds), the method developed by Gordillo et al. [8] was used. A volume of 50 μ l was injected in triplicate onto a Zorbax C18 column (250 x 4.6mm, 5 μ m particle size), maintained at 40 °C, with a flow rate of 0.63 mL/min. Acetonitrile-formic acid-water (3:10:87) as solvent A and acetonitrile-formic acid-water (50:10:40) as solvent B were used. The elution profile was as follows: 0 min 94% A - 6% B; 5 min 89% A - 11% B; 10 min 89% A - 11% B; 15 min 80% A - 20% B; 20 min 77% A - 23% B; 25 min 74% A - 26% B; 30 min 60% A - 40% B; 35 min 50% A - 50% B; 38 min 40% A - 60% B; 46 min 94% A - 6% B. UV-Vis spectra were recorded from 200 to 800 nm with a bandwidth of 2.0 nm. The quantification was made at 280, 320 and 360 nm by comparing the areas and the retention times with the gallic acid, caffeic acid, and quercetin standards, respectively. Total anthocyanins, flavonols and flavan-3-ols were calculated as sum of individual phenolic compounds identified by HPLC.

Statistical Analysis

All statistical analyses were performed using Statistica v.8.0 software [16]. Univariate analysis of variance (ANOVA) was applied using the general linear model program to establish whether mean values of the sample data differed significantly each other. The means values of each set of samples ($n = 3$) were compared by the Tukey test at a significance level of $p < 0.05$. Moreover, multivariate analysis of data (linear discriminant analysis, LDA) was performed in order to classify wine samples according to phenolic compounds and color parameters. This method was applied to the set of data

consisting of 70 rows (wine samples) and 28 columns (individual phenolic compounds and colorimetric variables).

RESULTS AND DISCUSSION

Climate conditions

The “Condado de Huelva” Designation of Origin is a typical warm climate region. According to the Köppen climatic classification [17], the southwestern of Spain has a “Csa” assignation, according to the classification of the main climates and precipitation and temperature conditions. Thus, “C” refers to “warm temperature”, “s” to “summer dry” and “a” to “hot summer”, respectively.

Fig. 1 shows the maximum, minimum and average temperature, relative humidity and rainfall of the summer (from 21st June to 21st September) of the area of “Condado de Huelva” Designation of Origin recorded for the last ten years. On the one hand, the values of temperature and relative humidity was almost constant over the last ten years. The average temperature was around 25 °C, although it ranged between 14 and 41 °C. Moreover, relative humidity ranged between 8 and 97 %, establishing around 50 % the medium value. Although some high peaks of rainfall were registered depending on the year, the average rainfall was established under 1 mm/day. These data have been provided by Instituto de Investigación y Formación Agraria y Pesquera (IFAPA), Junta de Andalucía, Spain.

Enological parameters

The results of analytical parameters of Syrah red wines revealed the correct development of the alcoholic fermentation (reducing sugars < 2.0 g/L), considering them as dry wines. pH values and total acidity were similar (around 3.4 units and 5.0 as tartaric acid, respectively) and volatile acidity values were below the limit established

by EU (1.2 g/L). Correct values of free and total SO₂ were also obtained (around 20 and 46 mg/L, respectively).

Identification of Polyphenolic Compounds in wines

Several types of polyphenolic compounds have been identified in this research study, belonged to monomeric anthocyanins, benzoic acids, hydroxycinnamic acid derivatives, flavan-3-ols and flavonols. Benzoic acids (gallic acid), hydroxycinnamic acid derivatives (GRP, *trans*-caftaric acid, *trans*-coutaric acid and *p*-coumaric acid) and flavan-3-ols ((+)-catechin) were the expected, well-known, compounds normally occurred in wines [18]. Among flavonols, myricetin and quercetin were identified as their 3-glucuronide and glucoside forms and only the last one for the rest of flavonols (kaempferol, isorhamnetin and syringetin) [19]. No aglycons were identified. Native grape anthocyanins were detected, including non-acylated, acetylated and *p*-coumaroylated derivatives of the five expected anthocyanidins (delphinidin, cyaniding, petunidin, peonidin and malvidin) [20, 21].

Pigment Evolution

Fig. 2 showed the evolution of the content of polyphenols (by Folin Ciocalteu) in different stages of the vinification process: wines with the addition of oak wood chips during fermentation (C) and those with the additional aging with oak wood chips (at 130 days) (CCp). The evolution of each wine were conducted during 365 days. Moreover, in order to know the significant differences between both kinds of wines, the mean values of all parameters was calculated and Tukey test was applied (Table 1).

A diminution of polyphenolic content was observed over time during the first 130 days of winemaking (Fig. 2). From this moment (the beginning of aging), both wines greatly differed on the evolution of polyphenols content. The aging of wines that contained chips during fermentation (CCp) made the content of polyphenols remain almost

constant in Syrah red wines, contrarily to the continue decrease observed in wines with the only addition of oak wood chips during fermentation (C). That is it, the common decrease of total polyphenolics in wines elaborated warm climate was avoided when Syrah red wines were submitted to aging with chips. In fact, significant ($p < 0.05$) higher content of all chemical compounds (both individual and total amounts) was experimented in CCp Syrah wines (Table 1). Differences were significant ($p < 0.05$) for total anthocyanins (belonged to the three kind of compounds, glycosylated, acetylated and *p*-coumaroylated), and flavan-3-ol ((+)-catechin) contents. With regard to the individual compounds, significant higher contents were observed in CCp Syrah wines in glucosilated anthocyanins (3-glucosides of delphinidin, cyaniding, petunidin, peonidin and malvidin) and in 3-acetyl-glucosides and 3-*p*-coumaroyl-glucosides of peonidin and malvidin. Hydroxycinnamic acid derivatives also evolved similarly, with significant ($p < 0.05$) higher values of *trans*-caftaric acid, *trans-p*-coutaric acid and *p*-coumaric acid, and (+)-catechin in CCp Syrah wines. These increases could be probably due to their release from wood, as other authors previously demonstrated [22, 23]. However, any significant difference on flavonols were observed because of the addition of oak wood chips in the aging stage. The higher levels of anthocyanins, hydroxycinnamic acid derivatives and (+)-catechin, and also the higher degree of copigmentation in CCp Syrah wines (Table 1) could be maybe consequence of the ellagitannins protective effect because of the presence of the wood [24]. The higher contents of anthocyanins and copigments (flavan-3-ols and hydroxycinnamic acid esters) could be related to the higher percentage of copigmentation observed in CCp Syrah wines (Table 1) (better pigment/copigment ratio), fact that could positively influence on a greater chemical stabilization of the wines [25]. That fact occurred at least during the first seven months

of winemaking (215 days), although the content of both kind of wines tended to be similar after one year of storage.

Chromatic evolution

The evolution of CIELAB color parameters (L^* , C^*_{ab} and h_{ab}) during vinification for wines with the addition of oak wood chips during fermentation (C) and those with the additionally aging with oak wood chips (CCp) were evaluated (Fig. 3).

Until 130 days of treatment (before aging), C Syrah red wines suffered a decrease in the values of lightness (L^*) and an upward trend of hue (h_{ab}) (10 and 70 %, respectively) (Fig. 3A and 3C). As affirmed Gordillo et al. [8], these behavior in color characteristics could be related with the conversion of copigmentation complexes into polymeric pigments.

The joint addition of chips during fermentation and aging (CCp) exerted a positive effect on the CIELAB color parameters of Syrah red wines. An immediate (and significant ($p < 0.05$)) darker colored wines (lower values of lightness, L^* , Fig. 3A) with a higher chromatic intensity (higher values of chroma, C^*_{ab} , Fig. 3B), together with a slightly higher yellowish tonalities (higher values of hue, h_{ab} , Fig. 3C), were observed in CCp Syrah wines. Similar results were found by several authors [26-28], who demonstrated a higher contribution of the yellow color (b^*) in the first months of aging with chips. This tonality could be related to the higher amounts of flavan-3-ols and total phenolics found in CCp Syrah wines, that could form direct condensations anthocyanins-tannin (red-orange colorations) and favor the color stabilization [29]. Therefore, the aging of Syrah wines with the addition of oak wood chips during fermentation provoked a desirable effect, not only on the color density (C^*_{ab} , Fig. 3B) but also on the color stability. The improvement of the quality of Syrah red wines could

be due to the transfer of some compounds from the wood to the wines (especially ellagitannins) [24].

With the aim of evaluating the colorimetric implications of the aging of wines with an addition of oak wood chips during fermentation, the mean color differences (ΔE^*_{ab}) among both kind of wines in each studied point of winemaking process were calculated. Taking into account that ΔE^*_{ab} of up to three CIELAB units indicates color differences appreciable to the human eyes [30], it could be affirmed that the addition of oak wood chips in aging led to color differences visually perceptible between both Syrah wines (ΔE^*_{ab} among 5 and 12) (data not shown). Once calculating the role of each color attribute respect $\Delta^2 E^*_{ab}$ (as percentage of the quadratic increases of lightness, chroma and hue), the visually appreciable differences were mainly quantitative ($\Delta^2 L^*_{ab}$ and $\Delta^2 C^*_{ab}$).

LDA

Linear discriminant analysis was applied with the objective to establish the variables which discriminating among samples, and carrying out an explanatory tool to uncover unknown trends in the data. A forward stepwise LDA was performed to differentiate the two sample groups (wines with the addition of oak wood chips during fermentation (C) and wines the additional aging with oak wood chips (CCp)). This analysis was carried out based on the phenolic composition and color characteristics, according to the Wilks' λ statistics to choose the descriptors that best distinguished the different wines. An F statistic is computed from the partial λ values, leading to a p level.

According to p -levels and F -values, the variables that discriminate between both groups of samples (C and CCp) with high level of significance ($p < 0.001$) were petunidin-3-glucoside and (+)-catechin and the colorimetric characteristic of chroma (C^*_{ab}). According to the classification functions, C and CCp wines were clearly differentiated

(Fig. 4), and 59% of the samples were correctly assigned. Moreover, the percentage of prediction was 60% and 58% for C and CCp samples, respectively.

CONCLUSIONS

The improvement of aging aptitude of Syrah wines with the addition of oak wood chips in the early stages of vinification has been demonstrated, in the light of the increase of the concentration of several phenolic compounds (anthocyanins) and copigments (flavan-3-ols and hydroxycinnamic acid esters), and intermolecular copigmentation reactions. As a result, it could be asserted that the common losses of color in warm climate winemaking could be avoided by using this joint tannin implementation. Besides, the combined use of both techniques (oak chips addition both during fermentation and subsequently aging) could be a viable oenological alternative to potentiate the ageing of Syrah red wines, commonly elaborated as young wines in warm climate. This study could be another step forward to improve the color stability of the red wines and produce high-quality wines elaborated in warm climate regions.

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Compliance with ethics standards

Conflict of interest. None.

Human and animal rights statement. This article does not contain any studies with human or animal subjects.

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FIGURE CAPTIONS

Fig. 1. Maximum (max), minimum (min) and average (averg) temperature (A), relative humidity (RH, B) and rainfall (RF, C) of the summers of the area of “Condado de Huelva” Designation of Origin recorded for the last ten years.

Fig. 2. Total polyphenols content (mg/L) evolution during vinification of Syrah red wines with the addition of oak wood chips during fermentation (C) and with the additional aging with oak wood chips (CCp). For each day, asterisks denote significant differences according to Tukey test ($p < 0.05$) between both kind of wines.

Fig. 3. Evolution of CIELAB parameters (L^* (A), C^*_{ab} (B) and h_{ab} (C)) during vinification of Syrah red wines with the addition of oak wood chips during fermentation (C) and with the additional aging with oak wood chips (CCp). For each day, asterisks denote significant differences according to Tukey test ($p < 0.05$) between both kind of wines.

Fig. 4. Scatterplot of the canonical variate obtained by LDA: Syrah red wines with the addition of oak wood chips during fermentation (C) and with the additional aging with oak wood chips (CCp).