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The influence of different prefermentative maceration processes and tartaric stabilization treatments on the color, cation content and other physico-chemical parameters of 'Băbească neagră' rosé wines

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Summary

This study enhances knowledge in the physicochemical and color parameters of rosé wines produced by the prefermentative maceration of native Romanian 'Băbească neagră' (Vitis vinifera L.) grape varieties, both before and after tartaric stabilization treatments by the contact procedure in an MK70 ultra-refrigerator. One variety was prepared without maceration as a control sample and the other was subjected to the maceration process for one of the following seven periods of time: 3.5 hours, 7 hours, 10.5 hours, 14 hours, 17.5 hours, 21 hours and 24.5 hours. The prefermentative maceration process exerts a significant influence on the amount of volatile acids, on the pH, conductivity and the K⁺ cation. The phenolic compound content, the chromatic parameters and the cation content of wines are significantly influenced by both prefermentative maceration and tartaric stabilization. The multifactor ANOVA tests prove that there is an interaction for all parameters except for total acidity.

K e y w o r d s : tartaric stabilization, rosé wine, prefermentative maceration, chromatic parameters, cation content.

A b b r e v i a t i o n s: ANOVA analysis of variance; VA volatile acidity; TA total acidity; χ conductivity; TSW tartaric stabilized wine; TPC total phenolic compounds; ANTH anthocyanins; L clarity; a red-green color component; b yellow-blue color component; C chroma; H° tone; WCI wine color intensity; WCH wine color hue; CIEDE color difference; CCS computerized color simulation.

Introduction

The ultimate challenge for a winemaker is to provide a quality wine that consumers will enjoy. Each step in the winemaking process is carefully planned and executed to maximize the quality of the wine. Control of the prefermentative maceration period and tartaric stabilization in must and wine are two of the fundamental steps used in winemaking to enhance and maintain color quality from year to year. Red wine color evolves during the maceration process through the conditioning phase; the process continues during the storage period (Auw *et al.* 1996, SARNI-MANCHADO *et al.* 1995). In the rosé winemaking process, red berries are usually pressed and the juice is then fermented. If no maceration step is involved, only some of the anthocyanins are extracted from the grape skin and other phenolic compounds, mainly those with a nonflavonoid structure, are extracted from the pulp. However, these compounds do not form stable anthocyanin-phenolic complexes (CHEYNIER 2001). Instead, the free monomeric anthocyanins, which are known to be unstable in wine and produce an unstable color, dominate (RIBÉREAU-GAYON *et al.* 2000).

Prefermentative maceration at low temperatures in white winemaking has been widely used to achieve better color stability (RAMEY *et al.* 1986). The technique may be improved by adding enzymatic preparations capable of degrading the polyosidic structure of the skin cell membranes, thereby increasing the release of polyphenols (CA-NAL-LLAUBÉRES 1993, GERBAUX *et al.* 2002, PARDO *et al.* 1999).

Among the grapes used for rosé and red wines, the 'Băbească neagră' grape variety is ranked first among native Romanian varieties in terms of cultivated area. Several appellations in south-eastern Romania make their wines from this grape variety. 'Băbească neagră' rosé wines have a bright red hue after fermentation that evolves to a salmon tone during the conditioning process.

Tartaric acid and tartrate play an important role in the stability of wines. The precipitation of potassium bitartrate (KHT) appears in both acidified and non-acidified wines (RATSIMBA et al. 1989). Several methods can reduce the tartaric acid content, such as the use of crystallization inhibitors, the modification of the wine pH and the linking of tartaric acid in soluble complexes. Among these procedures, the best known are refrigeration, the contact procedure, the reverse osmosis technique, the ion exchange method, electrodialysis and deacidification (RHEIN and NERADT 1979, CRACHERAU et al. 2001, MOUTOUNET et al. 1997). Cold tartrate stabilization is traditionally used to prevent the precipitation problem. This process involves cooling the wine to -4 °C over several days or the addition of crystallization germs (potassium tartrate crystalline powder) in a dose of 4 g·L⁻¹ prior to maintaining the wine at -4 °C for 3 h, the so-called contact method, in order to generate KHT precipitation prior to bottling (FEUILLAT 1980, BLOUIN 1982, MAUJEAN 1994). This technique can cause the simultaneous precipitation of polyphenols and can affect wine quality (VERNHET et al. 1999). The pigments of red and rosé wines are often involved in complexes with tartaric acid. As wine oxidizes and pigment polymerization occurs, the holding capacity for tartaric acid diminishes, often resulting in the

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delayed precipitation of potassium bitartrate (BALAKIAN and BERG 1968).

The aim of this study is to reveal the evolution of the physico-chemical and color parameters of rosé wines obtained by the prefermentative maceration of 'Băbească neagră' (*Vitis vinifera* L.) grapes, before and after tartaric stabilization by the chilling and contact methods and to evaluate these effects.

Material and Methods

Wines: A total quantity of 1200 kg of 'Băbească neagră' (*Vitis vinifera* L.) autochthonous grape varieties harvested on September 29, 2011 from Iași vineyard, Bucium wine center were used for this study. Harvesting was performed manually in plastic crates with an 18 kg capacity.

At the time of the harvest, the grapes had a sugar concentration of 240 g·L⁻¹. The grapes were crushed and destemmed, the two phases were immediatly separated (free run must and solid parts) using a Bucher Vaslin XPro 5 press (500 L), only by gravitational force. The two phases were weighed and quantitatively distributed in eight tanks, without addition of enological extraction enzymes or sulfur dioxide. 'Băbească neagră' 0 h (BN-0 h). In this variant, the must was immediately separated from the grape mass through gravitational drainage without pressing the pulp. The unclarified free run must was passed quantitatively into glass containers (50 L demijohns), and 30 g·hL⁻¹ Nutristart fermentation activators and 25 g·hL⁻¹ Laffort® Saccharomyces cerevisiae RX 60 yeast were added according to the protocols prescribed by the manufacturers. 'Băbească neagră' - the other seven variants (BN-3¹/₂ h; BN-7 h; BN-10¹/₂ h; BN-14 h; BN-17¹/₂ h; BN-21 h and BN-24¹/₂ h). These seven variants of unpressed grape marc underwent a prefermentative maceration process for the following periods of time: 3.5, 7, 10.5, 14, 17.5, 21 and 24.5 hours. The glass containers were vigorously homogenized for five minutes every hour throughout the prefermentative maceration process. The unclarified free run must was subjected to the same wine-making experimental protocol applied to variant 1 (BN-0 h). After the alcoholic fermentation was complete, the wines were removed from the yeast deposit, were not sulfited, were filtered with 0.45 µm sterile membrane filters and were bottled in 0.75 L glass bottles, using Nomacork Select 100 SeriesTM closures (0.37 mg O₂ after 3 months). The prefermentative maceration process and alcoholic fermentation were conducted at 10 °C in a room with controlled temperature. Wine bottles were stored in a cellar at 12 °C. After two weeks, the main physico-chemical characteristics, total phenolic compound content (TPC), total anthocyanin content (ANTH), chromatic parameters L, a, b, C, H° using the CIE-Lab 76 method, wine color intensity (WCI) and wine color hue (WCH) were measured. A sample was taken from each wine and was subjected to tartaric stabilization by the contact procedure to investigate the effect on the color of rosé wines. The tartaric stabilization technique was performed with an MK70 ultra-refrigerator by the following technique: 4 g·L⁻¹ crystallization germs (crystalline powder of acid potassium tartrate) was added, the wine was chilled to 4 °C with constant mechanical stirring for 3 hours at the same temperature and tartaric crystals were formed. The parameters analyzed before the tartaric stabilization were again measured.

S t a n d a r d a n a l y s i s: The main physico-chemical parameters analyzed were alcoholic strength, reducing sugars, total acidity (TA), volatile acidity (VA), real acidity (pH) and conductivity (X) (O.I.V., 2012).

Phenolic compound analysis: The total phenolic compounds (TPC) contained in the wines were oxidized by the Folin-Ciocalteu reagent (SOMERS and ZIM-METIS 1985). The resulting blue coloration has a maximum absorption at approximately 750 nm that is proportional to the total quantity of phenolic compounds originally present. To determine the TPC, we used a calibration curve obtained for different concentrations of gallic acid. The absorbances of the wines were measured at 750 nm according to the Folin-Ciocalteu method OIV-MA-AS2-10 using an Analytik Jena Specord S200 UV-VIS spectrophotometer with a 10 mm optical path length cuvette (Hellma® made of Quartz SUPRASIL®). The calibration curve equation was the following (x = A750 nm, y = TPC g·L⁻¹ gallic acid, r = 0.9881): y = 1.3238x-0.0014 (1).

Total anthocyanin content determination: In an acidic environment, there is a balance between the color and colorless forms of anthocyanins. This balance is dependent on the pH value. The variation of the color intensity between pH = 0.6 (HCl 2 % (v/v)) and pH =3.5 (citrate-phosphate buffer) in the presence of an alcoholic solution (0.1 % HCl) is proportional to the anthocyanin content. Phenolic function is not affected by changing the anthocyanin content, and other phenolic compounds (tannins) do not interfere in the anthocyanin determination. The absorbances at 520 nm were measured in a 10 mm optical path length glass cuvette (Hellma® made of Quartz SU-PRASIL®) using a UV-VIS Analytik Jena Specord S200 spectrophotometer. The anthocyanin content was calculated using the calibration curve (SOMERS and ZIMMETIS 1985). The equation is (x = A520 nm sample-A520 nm blank, y)=ANTH, mg/L gallic acid, r = 0.978): y=386.15x-1.3713 (2). Where x = A520 nm sample - A520 nm blank and y is ANTH in g·L⁻¹ gallic acid, r = 0.9787.

Color analyses: Wine color was assessed by the CIE-Lab tristimulus method established by the international organization Commission Internationale de l'Eclairage in 1976 and recommended for the wine industry by the Office International de la Vigne et du Vin since 1978 for the commercial description of wine color (O.I.V., 2012). Wine color measurements of each variant were taken at room temperature in triplicate. Wines were analyzed spectrophotometrically using an Analytik Jena Specord S200 UV-VIS spectrophotometer. An automated registration and classification of the absorption spectra were copied to a file using Aspect Plus[®] V1.7 software (Zeiss, Germany). Culoare software was used for the CIE-Lab values (L, a, b, C, H°), the wine color intensity (WCI) and the wine color hue (WCH). The wines were scanned from 360-830 nm in a 10 mm quartz cuvette (Hellma® made of Quartz SU-PRASIL®) at the D65 daylight illuminant with 10 degree

observer angle and bidistilled water as the blank. Wine color intensity (WCI) is a basic measure of how dark a wine is that is calculated using a summation of the absorbance measurements in the violet, green and red areas of the visible spectrum: WCI = A420 nm + A520 nm + A620 nm ($A\lambda$ represents the absorbance at wavelength λ). Wine color hue (WCH) is a simplistic measure of the appearance of the color, a ratio of the absorbance in the violet to the absorbance in the green: WCH = A420 nm/A520 nm. The color differences were also calculated with the CIEDE2000 formula. For values of CIEDE2000 smaller than unity, the colors of two wines are seen as identical; they cannot be sensorially differentiated.

Cation analyses: Potassium, calcium and sodium cation content were analyzed using an atomic absorption spectrophotometer Shimadzu AA6300 with auto sampler ASC-6100 and hollow cathode lamps self reverse SR. The calibration of the spectrophotometer was done with Merck standard solutions.

Potassium and sodium are determined directly in diluted wine by atomic absorption spectrophotometry after the addition of cesium chloride (CsCl solution containing 5 % cesium) to suppress ionization of potassium (OIV-MA-AS322-02A - K, OIV-MA-AS322-03A - Na) (OIV 2012). The equation of the calibration curve for K⁺ is the following: y = 0.089060x+0.048200 (3). Where x = A769.9 nm and y is equivalent K⁺ content in g⁻L⁻¹, r = 0.9893, calibration range (0.5-8.0 g⁻L⁻¹) and air - acetylene flame composition was 2.0 g⁻L⁻¹. The equation of the calibration curve for Na⁺ is the following: y = 0.272080x+0.0076800 (4). Where x = A589.0 nm and y is equivalent Na⁺ content in g⁻L⁻¹, r = 0.9992, calibration range (0.25-1.0 g⁻L⁻¹) and air - acetylene flame composition was 1.8 L⁻min⁻¹.

Calcium is determined directly in diluted wine by atomic absorption spectrophotometry after the addition of an ionization suppression agent (LaCl₃*7H₂O, 50 g·L⁻¹) (OIV-MA-AS322-04A) (OIV 2012). The equation of the calibration curve for Ca²⁺ is the following: y = 0.032412x+0.010300 (5). Where x = A422.7 nm and y is equivalent Ca²⁺ content in mg·L⁻¹, r = 0.9954, calibration range (0.5-10.0 mg·L⁻¹) and air - acetylene flame composition was 2.0 L·min⁻¹.

Statistical analyses: Statistical analyses were performed using Statgraphics Centurion XVI® software, (StatPoint Technologies, Inc, U.S.A.). In this study, we applied a one-way ANOVA procedure and multifactor ANOVA that was designed to construct a statistical model describing the impact of two categorical factors Xj (different variants of prefermentative maceration and tartaric stabilization) on a dependent variable Y (CIE Lab parameters, total phenolic content and anthocyanin content of wines). The statistic displayed Lavene's test the null hypothesis that the standard deviations of every physico-chemical parameter within each of the 8 levels of maceration time are the same. We used as an alternative to the standard analysis of variance that compares level medians instead of means the Kruskal-Wallis Test. This test is much less sensitive to the presence of outliers than a standard oneway ANOVA and should be used whenever the assumption of normality within levels is not reasonable. Mood's Median Test is another method of determining whether or not the medians of all q materials are equal. It is less sensitive to outliers than the Kruskal-Wallace test, but is also less powerful when the data come from distributions such as the normal.

Results and Discussion

Statistical testing of physico-chemical parameters: The means and standard deviations of the physico-chemical parameters of 'Băbească neagră' wines obtained by the prefermentative maceration process with different exposure times and tartaric stabilization processes are presented in Tab. 1. Total acidity (TA) also showed similar mean values, especially for the variants that did not undergo the tartaric stabilization process. All other parameters showed average values with variability to a smaller or larger degree depending on the impact of different periods of maceration and the tartaric stabilization processes exerted on them. To determine any differences between the mean values of the studied parameters, we applied multiple range tests (oneway ANOVA) that compared the means using different multiple sample comparison procedures (Costello et al. 2012). For alcoholic strength, there was no statistically significant difference between any pair of means at the 95.0 % confidence level (superscript letters from Tab. 1).

Among the wines that had not undergone the tartaric stabilization process (TA), the total acidity parameter yielded 1 pair that had statistically significant differences at the 95.0 % confidence level. The wines that did undergo this treatment (TA-TSW) had 10 pairs with statistically significant differences at the 95.0 % confidence level (Tab. 1).

In the case of the pH parameter of wines that did not undergo a tartaric stabilization process, 19 pairs that had statistically significant differences at the 95.0 % confidence level were identified and the means formed 4 homogenous groups. The pH values of the wines that did undergo stabilization treatment (pH-TSW) had 24 pairs with statistically significant differences at the 95.0 % confidence level (Tab. 1). The conductivity parameter of wines that did not undergo a tartaric stabilization process (X) had 26 pairs with statistically significant differences at the 95.0 % confidence level. The wines that did undergo the stabilization treatment (X -TSW) had 27 pairs with statistically significant differences at the 95.0 % confidence level and formed 7 homogenous groups out of a total of 8, within which there were statistically significant differences (Tab. 1).

To describe the impact of a single categorical factor X (in our case prefermentative maceration time) on a dependent variable Y (physico-chemical parameters of 'Băbească neagră' wines), we used the one-way ANOVA procedure. Tests were run to determine whether there were significant differences between the means, variances, and/or medians of the physico-chemical parameters of 'Băbească neagră' wines at the different levels of prefermentative maceration time (Tab. 2).

We found a significant influence of the maceration time on the mean values of most parameters at the 95.0 % confidence level, except for alcoholic strength and total acidity, which had much a higher P-value than 0.05 (Tab. 2).

C. I. ZAMFIR *et al*.

Table 1

Classical enological parameters of 'Băbească neagră' wines obtained by prefermentative maceration with various exposure times

Parameter	BN-0 h	BN-3 ½ h	BN-7 h	BN-10 ½ h
Alcoholic strength (% vol. alc.)	$14.04 \pm 0.17^{\text{n.s.}}$	$14.03 \pm 0.19^{\text{n.s.}}$	$14.02 \pm 0.15^{n.s.}$	$14.03 \pm 0.17^{\text{n.s.}}$
Sugars $(g \cdot L^{-1})$	1.25 ± 0.01^{bcdefgh}	0.5 ± 0.01^{acdefgh}	0.97 ± 0.02^{abdefgh}	2.51 ± 0.02^{abcefgh}
$VA(g \cdot L^{-1} C_2 H_4 O_2)$	$0.46 \pm 0.01^{\text{cdefgh}}$	$0.48 \pm 0.02^{\text{cdefgh}}$	0.62 ± 0.01^{abdefgh}	0.56 ± 0.01^{abch}
TA $(g \cdot L^{-1} C_A^2 H_a^2 O_a^2)$	7.21 ± 0.13^{d}	$7.11 \pm 0.12^{n.s.}$	$7.17 \pm 0.17^{n.s.}$	6.91 ± 0.19^{a}
TA-TSW $(\vec{g} \cdot \vec{L}^{-1} \circ \vec{C}_A H_c O_c)$	$6.96 \pm 0.18^{\text{defgh}}$	$6.84\pm0.15^{\rm deg}$	6.79 ± 0.14^{de}	6.44 ± 0.17^{abc}
pH	3.13 ± 0.03^{bdefgh}	$3.19 \pm 0.02^{\text{acde}}$	3.15 ± 0.02^{cdefgh}	3.24 ± 0.03^{abcfg}
pH-TSW	3.08 ± 0.02^{bcdefgh}	3.13 ± 0.01^{acdefgh}	3.31 ± 0.03^{abdefgh}	3.47 ± 0.02^{abcefh}
χ (mS·cm ⁻¹)	3.83 ± 0.02^{bcdefgh}	4.35 ± 0.03^{acefgh}	4.57 ± 0.01^{abdefgh}	4.33 ± 0.02^{acefgh}
χ -TSW (mS·cm ⁻¹)	3.42 ± 0.03^{bcdefgh}	3.99 ± 0.01^{acdefgh}	4.29 ± 0.03^{abdefgh}	3.84 ± 0.02^{abcefh}
Parameter	BN-14 h	BN-17 ½ h	BN-21 h	BN-24 ½ h
Alcoholic strength (% vol. alc.)	$14.03 \pm 0.19^{\text{n.s.}}$	$14.02 \pm 0.18^{\text{n.s.}}$	$14.03 \pm 0.16^{n.s.}$	$14.02 \pm 0.12^{\text{n.s.}}$
Sugars $(g \cdot L^{-1})$	3.03 ± 0.03^{abcdfh}	3.74 ± 0.03^{abcdegh}	3.04 ± 0.02^{abcdfh}	3.64 ± 0.02^{abcdefg}
$VA(g \cdot L^{-1} C_2 H_4 O_2)$	0.55 ± 0.02^{abcfg}	0.58 ± 0.01^{abceh}	0.58 ± 0.02^{abceh}	0.53 ± 0.02^{abcdfg}
TA $(g \cdot L^{-1} C_A H_c O_c)$	$6.98 \pm 0.16^{\text{n.s.}}$	$7.05 \pm 0.17^{\text{n.s.}}$	$7.04 \pm 0.20^{n.s.}$	$7.14 \pm 0.15^{n.s.}$
TA-TSW $(g \cdot L^{-1} \circ H_{c}O_{c})$	6.50 ± 0.15^{abc}	6.58 ± 0.16^{a}	6.55 ± 0.13^{ab}	6.58 ± 0.19^{a}
pH	3.27 ± 0.01^{abcfgh}	$3.19\pm0.02^{\mathrm{acde}}$	3.20 ± 0.01^{acde}	$3.22\pm0.03^{\rm ace}$
pH-TSW	3.57 ± 0.02^{abcdg}	3.61 ± 0.03^{abcdgh}	3.50 ± 0.02^{abcef}	3.54 ± 0.04^{abcdf}
χ (mS·cm ⁻¹)	4.24 ± 0.02^{abcdfgh}	4.5 ± 0.03^{abcdeg}	4.42 ± 0.03^{abcdefh}	4.47 ± 0.02^{abcdeg}
χ -TSW (mS·cm ⁻¹)	3.69 ± 0.02^{abcdfgh}	3.77 ± 0.01^{abcdegh}	3.85 ± 0.01^{abcefh}	89.89 ± 0.03^{abcdefg}

VA: volatile acidity; TA: total acidity; χ : conductivity; TSW: tartaric stabilized wine. Data are means of triplicate determinations ± standard deviation over the three replications in one wine sample. Different superscript letters indicate statistically significant differences at the 95.0 % confidence level between each pair of means within each row. The method used to discriminate among the means is Fisher's least significant difference (LSD) procedure.

Table 2

Results of the statistical tests of the classical enological parameters of 'Băbească neagră' wines obtained by prefermentative maceration with various exposure times

Tartalaran	ANO	VA	Laven	e's test	Kruskal-V	Vallis test	Mood	's test
Tested parameters	F Ratio	P-value	Test stat	P-value	Test stat	P-value	Test stat	P-value
Alcoholic strength	0.01 n.s.	1.0000	0.08 n.s.	0.9989	0.62 ^{n.s.}	0.9988	1.24 ^{n.s.}	0.9898
Sugars	10567.05*	0.0000	0.51 ^{n.s.}	0.8154	22.43*	0.0021	24.00*	0.0011
VA	34.29*	0.0000	0.46 ^{n.s.}	0.8511	20.99*	0.0037	18.67*	0.0092
TA	1.14 ^{n.s.}	0.3886	0.11 ^{n.s.}	0.9964	6.91 ^{n.s.}	0.4384	2.67 ^{n.s.}	0.9140
TA-TSW	3.99*	0.0104	0.06 ^{n.s.}	0.9994	14.85*	0.0379	13.26 ^{n.s.}	0.0660
pН	12.11*	0.0000	0.54 ^{n.s.}	0.7894	19.29*	0.0073	13.33 ^{n.s.}	0.0643
pH-TSW	195.55*	0.0000	0.52 ^{n.s.}	0.8018	21.95*	0.0025	18.66*	0.0092
χ	289.94*	0.0000	0.36 ^{n.s.}	0.9103	22.27*	0.0022	24.00*	0.0011
χ-TSW	389.19*	0.0000	0.72 ^{n.s.}	0.6558	22.31*	0.0022	18.66*	0.0092

VA: volatile acidity; TA: total acidity; χ : conductivity; TSW: tartaric stabilized wine. The superscript symbol * indicates that these factors with *P*-value less than 0.05 have a statistically significant effect on the parameter at the 95.0 % confidence level. The superscript letters *n.s.* indicates that the factor does not have a statistically significant influence. All *F*-ratios are based on the residual mean square error.

The statistic displayed in Lavene's column test the null hypothesis that the standard deviations of every physicochemical parameter within each of the 8 levels of maceration time are the same. All of the tested parameters have a P-value higher than 0.05, which indicates that the null hypothesis must be accepted, and the standard deviations for every physico-chemical parameter within each of the 8 levels of maceration time are the same (Tab. 2).

The Kruskal-Wallis test tests the null hypothesis that the medians of every physico-chemical parameter within each of the 8 levels of maceration time are the same. The test shows that in the case of the alcohol strength and the total acidity parameters, we can accept the null hypothesis because these parameters have P-values greater than 0.05. Because all of the other test parameters have P-values less than 0.05, there is a statistically significant difference amongst the medians at the 95.0 % confidence level (Tab. 2). Mood's median test tests the hypothesis that the medians of all 8 samples are equal. In the case of four physico-chemical parameters (alcoholic strength, TA, TA-STW and pH), the hypothesis that the medians are equal was accepted by this test. All other parameters have P-values less than 0.05, indicating that the medians of the samples are significantly different at the 95.0 % confidence level (Tab. 2).

Statistical testing of phenolic compounds and chromatic parameters: The means and standard deviations of the total phenolic compounds and anthocyanins and the evolution of the 'Băbească neagră' wine chromatic parameters obtained by the prefermentative maceration process with various exposure times and tartaric stabilization processes are presented in Tab. 3.

The average total phenolic compound content (TPC) and anthocyanin content (ANTH) increased with the in-

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Concentrations of the total phenolic compounds and anthocyanins and the evolution of the 'Băbească neagră' wine chromatic parameters obtained by prefermentative maceration with different

exposure times

Parameter	BN-0 h	BN-3 ½ h	BN-7 h	BN-10 ½ h	BN-14 h	BN-17 ½ h	BN-21 h	BN-24 ½ h
TPC (mg·L ⁻¹)	$122.59 \pm 2.24^{bcdefgh}$	143.05 ± 0.75 acdefgh	158.28 ± 0.57 abdefgh	$211.04 \pm 1.18^{abcefgh}$	224.55 ± 1.01 abcdfgh	$236.04 \pm 1.84^{abcdegh}$	$247.55 \pm 0.42^{\text{abcdefh}}$	258.73 ± 0.43 abcdefg
$TPC - TSW (mg \cdot L^{-1})$	$112.66 \pm 1.34^{\text{bcdefgh}}$	127.33 ± 0.83 acdefgh	$136.56 \pm 0.56^{abdefgh}$	$180.26 \pm 1.24^{abcefgh}$	$183.10 \pm 1.23^{abcdfgh}$	$186.12 \pm 1.46^{abcdegh}$	$190.47 \pm 0.62^{abcdefh}$	$193.80 \pm 0.32^{\mathrm{abcdefg}}$
ANTH (mg·L ⁻¹)	8.66 ± 0.20^{bodefgh}	34.33 ± 0.56^{acdefgh}	55.35 ± 1.14^{abdefgh}	66.84 ± 3.63^{abcefgh}	79.64 ± 3.24^{abcdfgh}	$100.95 \pm 5.16^{abcdegh}$	$117.65 \pm 4.91^{\text{abcdefh}}$	127.57 ± 1.62 abcdefg
ANTH $-TSW (mg \cdot L^{-1})$	$3.81 \pm 0.10^{\text{bodefgh}}$	21.39 ± 0.23 acdefgh	34.31 ± 1.04^{abdefgh}	49.09 ± 2.12^{abcefgh}	54.46 ± 1.13^{abcdfgh}	$60.73 \pm 2.16^{\text{abcdegh}}$	$77.50 \pm 1.19^{\text{abcdefh}}$	$88.82 \pm 1.02^{\text{abcdefg}}$
L	90.71 ± 0.12^{bcdefgh}	84.81 ± 0.19 acdefgh	81.49 ± 0.15^{abdefgh}	79.39 ± 0.18^{abcefgh}	$74.98 \pm 0.21^{\text{abcdfgh}}$	73.11 ± 0.24^{abcdegh}	$67.03 \pm 0.20^{\text{abcdefh}}$	66.65 ± 0.19^{abcdefg}
L - TSW	98.70 ± 0.15^{bcdefgh}	95.28 ± 0.17 acdefgh	94.45 ± 0.14^{abdefgh}	92.94 ± 0.12^{abcefgh}	91.95 ± 0.19^{abcdfgh}	90.60 ± 0.17^{abcdegh}	$89.15 \pm 0.15^{\text{abcdef}}$	88.98 ± 0.21 abcdef
а	$8.41 \pm 0.14^{\text{bodefgh}}$	18.78 ± 0.21 acdefgh	23.58 ± 0.18^{abdefgh}	19.24 ± 0.22^{abcefgh}	25.38 ± 0.17^{abcdfgh}	$27.09 \pm 0.15^{\text{abcdegh}}$	$37.13 \pm 0.17^{\text{abcdefh}}$	$35.29 \pm 0.20^{\mathrm{abcdefg}}$
a - TSW	$1.18 \pm 0.15^{\text{bodefgh}}$	4.19 ± 0.19 acdefgh	5.18 ± 0.18^{abdefgh}	6.44 ± 0.13^{abcefgh}	$7.20 \pm 0.18^{ m abcdfgh}$	8.17 ± 0.18^{abcdegh}	9.61 ± 0.16^{abcdefh}	$10.45 \pm 0.19^{\mathrm{abcdefg}}$
þ	$5.41 \pm 0.10^{\text{bodefgh}}$	7.45 ± 0.11 acdefgh	$7.20 \pm 0.12^{\text{abdefgh}}$	9.98 ± 0.11 abceh	$10.7 \pm 0.12^{\mathrm{abcdg}}$	$10.74 \pm 0.13^{\mathrm{abcdg}}$	$10.01 \pm 0.09^{\text{abcefh}}$	$10.65 \pm 0.15^{\mathrm{abcdg}}$
b - TSW	2.40 ± 0.12^{bodefgh}	$6.02 \pm 0.14^{\rm acdefgh}$	7.11 ± 0.11 abdefgh	$9.71 \pm 0.10^{\rm abcefgh}$	11.04 ± 0.14^{abcdfgh}	12.49 ± 0.09^{abcdegh}	13.26 ± 0.12^{abcdefh}	13.94 ± 0.13 abcdefg
C	$10.00 \pm 0.10^{\text{bodefgh}}$	20.20 ± 0.15 acdefgh	24.66 ± 0.17^{abdefgh}	21.67 ± 0.18^{abcefgh}	$27.55 \pm 0.17^{\text{abcdfgh}}$	29.15 ± 0.15^{abcdegh}	$38.46 \pm 0.19^{\text{abcdefh}}$	$36.87 \pm 0.17^{\mathrm{abcdefg}}$
C - TSW	2.74 ± 0.12^{bodefgh}	7.33 ± 0.12 acdefgh	8.80 ± 0.13^{abdefgh}	$11.65 \pm 0.14^{\rm abcefgh}$	13.18 ± 0.11^{abcdfgh}	14.92 ± 0.17^{abcdegh}	$16.37 \pm 0.14^{\text{abcdefh}}$	$17.43 \pm 0.16^{\text{abcdefg}}$
H°	32.72 ± 0.09^{bodefgh}	21.65 ± 0.12^{acdegh}	16.99 ± 0.13^{abdefg}	27.42 ± 0.12^{abcefgh}	$22.85 \pm 0.11^{\text{abcdfgh}}$	$21.63 \pm 0.16^{\mathrm{acdegh}}$	$15.09 \pm 0.12^{\text{abcdefh}}$	$16.79 \pm 0.15^{\mathrm{abdefg}}$
H° - TSW	$64.59 \pm 0.11^{\text{bodefgh}}$	55.16 ± 0.11 acdefgh	53.89 ± 0.18^{abdefh}	56.46 ± 0.17^{abcefgh}	56.87 ± 0.20^{abcdgh}	56.80 ± 0.18^{abcdgh}	54.08 ± 0.16^{abdefh}	53.14 ± 0.19^{abcdefg}
WCI	0.36 ± 0.02^{bodefgh}	0.59 ± 0.02^{acdefgh}	$0.72 \pm 0.03^{\text{abdefgh}}$	0.84 ± 0.01^{abcefgh}	$1.03 \pm 0.02^{\text{abcdfgh}}$	$1.11 \pm 0.04^{\text{abcdegh}}$	1.40 ± 0.05^{abcdef}	$1.41 \pm 0.04^{\mathrm{abcdef}}$
WCI - TSW	$0.07 \pm 0.01^{ m bodefgh}$	0.2 ± 0.01 acdefgh	0.26 ± 0.02^{abdefgh}	$0.34 \pm 0.04^{ m abefgh}$	$0.38 \pm 0.03^{\mathrm{abcfgh}}$	0.45 ± 0.02^{abcdegh}	$0.51 \pm 0.01^{\mathrm{abcdef}}$	$0.52 \pm 0.03^{\mathrm{abcdef}}$
WCH	0.99 ± 0.02^{bodefgh}	$0.85 \pm 0.02^{\mathrm{acdefg}}$	$0.79 \pm 0.01^{\text{abdefh}}$	$1.02 \pm 0.01^{\rm abcefgh}$	$0.94 \pm 0.02^{ m abcdgh}$	$0.93 \pm 0.01^{ m abcdgh}$	0.79 ± 0.02^{abdefh}	$0.83 \pm 0.01^{\mathrm{acdefg}}$
WCH - TSW	$1.7 \pm 0.03^{ m bcdefgh}$	1.47 ± 0.03^{ade}	$1.47 \pm 0.02^{\text{ade}}$	$1.53 \pm 0.02^{\rm abcgh}$	$1.52 \pm 0.02^{\rm abcgh}$	$1.51\pm0.02^{ m agh}$	$1.44 \pm 0.03^{\text{adef}}$	$1.45 \pm 0.02^{ m adef}$
CIEDE 2000	9.65	14.12	16.48	13.88	17.59	18.16	22.89	22.14
CWCS								
CWCS - TSW								
			-			-		

intensity; WCH: wine color hue; CIEDE 2000: color difference between non stabilized wine and stabilized wine; CCS: computerized color simulation). Data are means of triplicate determinations ± TPC: total phenolic compounds; ANTH: anthocyanins; L: clarity; a: red-green color component; b: yellow-blue color component; C: chroma; H^{*}: tone; TSW: tartaric stabilized wine; WCI: wine color standard deviation over the three replications in one wine sample. Different superscript letters indicates statistically significant differences at the 95.0 % confidence level between each pair of means within each row. The method used to discriminate among the means is Fisher's least significant difference (LSD) procedure. creasing maceration time (Tab. 3). A reduction in the total phenolic compound (TPC) and anthocyanin content was found when comparing the values of these parameters from tartaric stabilized wines (TSW) and nonstabilized wines. To determine whether there were any significant differences between the mean values of the phenolic compounds, we applied multiple range tests to compare the means using different multiple sample comparison procedures. All wines, both the tartaric stabilized and non-stabilized, had 28 pairs that had statistically significant differences in the phenolic compound content at the 95.0 % confidence level and formed 8 homogenous groups from 8, within which there were statistically significant differences (see Tab. 3).

The chromatic characteristics of a wine are defined by the colorimetric coordinates: clarity (L), red-green color component (a), blue-yellow color component (b), and the derived magnitudes of chroma (C) and tone (H°).

The colorimetric coordinate of the chromatic characteristic L shows a decreasing trend with increasing maceration exposure time (Tab. 3). The values of this parameter in tartaric stabilized wines (L-TSW) were greater than in the non-stabilized wines. In the non-stabilized wines, had 28 pairs that had statistically significant differences at the 95.0 % confidence level and formed 8 homogenous groups from 8, within which there were statistically significant differences. In the tartaric stabilized wines, there were 27 pairs with statistically significant differences at the 95.0 % confidence level and formed 7 homogenous groups with statistically significant differences (see Tab. 3).

The red-green color component (a) of the chromatic characteristics of the studied wines showed a relative upward trend as the maceration time decreased, but the values of a-TSW parameter of the wines that were subjected of tartaric stabilization process were less than the values recorded for the UN-TSW wines (Tab. 3). For this parameter, all wines, both stabilized and non-stabilized, had 28 pairs that were identified with statistically significant differences at the 95.0 % confidence level and formed 8 homogenous groups out of a total of 8, within which there were statistically significant differences (see Tab. 3).

The blue-yellow color component (b) of the chromatic characteristics of the studied wines showed variations from BN 0 h to BN 24 $\frac{1}{2}$ h, but there was no obvious increase or decrease in this parameter after tartaric stabilization. For tartaric non-stabilized wines (UN-TSW), 24 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 5 homogenous groups out of a total of 8, within which there were statistically significant differences. In the tartaric stabilized wines (b-TSW), 28 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 8 homogenous groups out of a total of 8, within which there were statistically significant differences (Tab. 3). The chroma (C vs. C-TSW) parameter of the wines showed minor variations in the two categories of wines studied. For all of the wines, 28 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 8 homogenous groups out of a total of 8, within which there were statistically significant differences (Tab. 3).

The last parameter of the chromatic characteristics is tone (H°), which showed higher values in the stabilized wines compared to the non-stabilized wines. In the nonstabilized wines, 26 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 6 homogenous groups out of a total of 8, within which there were statistically significant differences. In the stabilized wines, there were 26 pairs identified that had statistically significant differences at the 95.0 % confidence level and formed 6 homogenous groups with statistically significant differences (Tab. 3).

Wine color intensity (WCI) values for both the stabilized and non-stabilized wines generally increased from variant BN-0 h to Bn-24 ½ h. In the non-stabilized wines, 27 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 7 homogenous groups out of a total of 8 within which there were statistically significant differences. In the stabilized wines, 26 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 6 homogenous groups with statistically significant differences (Tab. 3).

For the wine color hue (WCH) parameter in the nonstabilized wines, 25 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 5 homogenous groups with statistically significant differences. WCH parameter means at the 95.0 % confidence level. For the tartaric stabilized wines (WCH-TSW), 17 pairs were identified that had statistically significant differences at the 95.0 % confidence level and formed 4 homogenous groups with statistically significant differences. There was an overall increase in the parameter values following the tartaric stabilization process.

The color difference (ΔE) calculated between the L, a, and b chromatic parameters of the studied wines indicated that the wines can be sensorially differentiated based on these parameters; the results of color difference were greater than unity. This can be observed in the computer wine color simulations (CWCS, CWCS-TSW) shown in Tab. 3.

In the one-way ANOVA, we see a significant influence of the maceration time on the mean values of all parameters at the 95.0 % confidence level (Tab. 4). For the Lavene's test all of the parameters have P-values higher than 0.05, which means that the null hypothesis is accepted and that the standard deviations for every phenolic compound or chromatic characteristic parameter within each of the 8 levels of maceration time are the same (Tab. 4). The Kruskal-Wallis test shows *P*-values less than 0.05 for all test parameters, which means there is a statistically significant difference amongst the medians at the 95.0 % confidence level (Tab. 4).

Mood's Median Test shows P-values for the chi-square test less than 0.05, the medians of the samples are signifi-

Table 4

Results of the statistical tests to the total phenolic compounds, anthocyanins and chromatic parameters of 'Băbească neagră' wines obtained by prefermentative maceration with different exposure times

Test Deremeter	ANO	VA	Lavene	e's Test	Kruskal-V	Vallis Test	Mood	's Test
Test-Parameter	F-ratio	P-value	Test stat	P-value	Test stat	P-value	Test stat	P-value
TPC	5317.02*	0.0000	1.19 ^{n.s.}	0.3585	22.68*	0.0019	24.00*	0.0011
TPC - TSW	3012.81*	0.0000	0.67 ^{n.s.}	0.6921	22.68*	0.0019	24.00*	0.0011
ANTH	513.08*	0.0000	1.53 ^{n.s.}	0.2264	22.68*	0.0019	24.00*	0.0011
ANTH - TSW	1364.09*	0.0000	1.28 ^{n.s.}	0.3197	22.68*	0.0019	24.00*	0.0011
L	6053.02*	0.0000	0.15 ^{n.s.}	0.9913	22.57*	0.0020	24.00*	0.0011
L - TSW	1219.19*	0.0000	0.12 ^{n.s.}	0.9956	22.49*	0.0020	24.00*	0.0011
а	7812.59*	0.0000	0.09 ^{n.s.}	0.9978	22.68*	0.0019	24.00*	0.0011
a - TSW	934.90*	0.0000	0.06 ^{n.s.}	0.9994	22.68*	0.0019	24.00*	0.0011
b	904.42*	0.0000	0.09 ^{n.s.}	0.9976	21.48*	0.0031	18.66*	0.0092
b - TSW	3379.11*	0.0000	0.09 ^{n.s.}	0.9982	22.68*	0.0019	24.00*	0.0011
С	9719.38*	0.0000	0.11 ^{n.s.}	0.9960	22.68*	0.0019	24.00*	0.0011
C - TSW	3956.27*	0.0000	0.09 ^{n.s.}	0.9982	22.68*	0.0019	24.00*	0.0011
H°	6546.77*	0.0000	0.12 ^{n.s.}	0.9956	22.30*	0.0022	18.66*	0.0092
H° - TSW	1425.49*	0.0000	0.17 ^{n.s.}	0.9871	22.18*	0.0023	24.00*	0.0011
WCI	428.07*	0.0000	0.74 ^{n.s.}	0.6387	22.43*	0.0021	24.00*	0.0011
WCI - TSW	132.49*	0.0000	0.90 ^{n.s.}	0.5287	22.29*	0.0022	18.66*	0.0092
WCH	96.26*	0.0000	0.45 ^{n.s.}	0.8511	22.01*	0.0025	24.00*	0.0011
WCH-TSW	35.22*	0.0000	0.18 ^{n.s.}	0.9852	19.34*	0.0071	18.66*	0.0092

TPC: total phenolic compounds; ANTH: anthocyanins; L: clarity; a: red-green color component; b: yellowblue color component; C: chroma; H^{\circ}: tone; TSW: tartaric stabilized wine; WCI: wine color intensity; WCH: wine color hue. The superscript symbol * indicates that the factors with a *P*-value less than 0.05 have a statistically significant effect on the parameter at the 95.0 % confidence level. The superscript letters n.s. indicates that the factor does not have a statistically significant influence. All F-ratios are based on the residual mean square error. cantly different at the 95.0 % confidence level (Tab. 4). The multifactor ANOVA procedure is designed to construct a statistical model describing the impact of two or more categorical factors X_j (maceration-time factor and tartaric stabilization factor) on a dependent variable Y (physico-chemical, phenolic compounds and chromatic characteristics parameters). The first component attributable to the main effect of the maceration time factor measures the variability amongst the mean responses at each level of the factor. The second component attributable to the main effect of the tartaric stabilization factor measures the variability amongst the mean responses at each level of the factor.

Because the *P*-values are less than 0.05, the maceration time factor and the tartaric stabilization factor have a statistically significant effect on all the studied parameters at the 95.0 % confidence level (Tab. 5). The third component attributable to the interaction between the different factors occurs if the effect of a factor depends on the level of the other factor. For the TA parameter, the main effects of the maceration time and tartaric stabilization factors are not statistically significant. For all other parameters, the main effects are statistically significant at the 95.0 % confidence level (Tab. 5).

The values of K^+ , Na^+ and Ca^{2+} cations content before and after the tartaric stabilization, as determined by AAS, are shown in Tab. 6. The content of K^+ increases progressively with the increase of prefermentative maceration time, while Na^+ and Ca^{2+} cations concentration values present small differences from one sample to another. The studied wine samples present a decrease in the concentration of these cations, which demonstrates that the tartaric stabilization process occurred, and some of these cations formed potassium sodium tartrate and calcium tartrate.

Table 5

Results of the multifactor ANOVA statistical tests to the studied parameters of the 'Băbească neagră' wines obtained by prefermentative maceration with different exposure times and a tartaric stabilization process

		Main	effects		Intera	ctions
Test-Parameter	Mac. time	e factor	Tart. stab	. factor	1 -	- 2
	F-ratio	P-value	F-ratio	P-value	F-ratio	P-value
TA	4.36*	0.0017	81.55*	0.0000	0.71 ^{n.s.}	0.6666
pН	147.35*	0.0000	855.78*	0.0000	80.25*	0.0000
χ	612.16*	0.0000	5766.18*	0.0000	59.70*	0.0000
TPC	8261.76*	0.0000	12418.5*	0.0000	471.80*	0.0000
ANTH	1236.88*	0.0000	1305.58*	0.0000	46.96*	0.0000
L	6619.58*	0.0000	92040.71*	0.0000	1292.38*	0.0000
а	7155.15*	0.0000	121998.5*	0.0000	2011.17*	0.0000
b	3818.53*	0.0000	195.07*	0.0000	515.46*	0.0000
С	13102.88*	0.0000	111875.8*	0.0000	1508.06*	0.0000
H°	5962.87*	0.0000	655976.0*	0.0000	667.27*	0.0000
WCI	546.63*	0.0000	5412.80*	0.0000	94.97*	0.0000
WCH	92.62*	0.0000	10971.27*	0.0000	14.27*	0.0000

TA: total acidity; χ : conductivity; TPC: total phenolic compounds; ANTH: anthocyanins; L:clarity; a: red-green color component; b: yellow-blue color component; C: chroma; H°: tone; WCI: wine color intensity; WCH: wine color hue. The superscript symbol * indicates that the factors with a *P*-value less than 0.05 have a statistically significant effect on the parameter at the 95.0 % confidence level. The superscript letters n.s. indicates that the factor does not have a statistically significant influence. All F-ratios are based on the residual mean square error.

Table 6

The K⁺, Na⁺ and Ca²⁺ cations concentration in mg·L⁻¹ of 'Bǎbeascǎ neagrǎ' wines obtained by prefermentative maceration with various exposure times and a tartaric stabilization process

Parameter	BN-0 h	BN-3 ½ h	BN-7 h	BN-10 ½ h
K ⁺	561.64 ± 0.0020	821.02 ± 0.0018	974.18 ± 0.0007	1044.24 ± 0.0009
K^+ -TSW	513.55 ± 0.0036	633.15 ± 0.0090	663.10 ± 0.0043	626.35 ± 0.0079
Na ⁺	37.98 ± 0.0003	39.39 ± 0.0014	42.43 ± 0.0003	50.68 ± 0.0030
Na ⁺ -TSW	12.98 ± 0.0002	12.65 ± 0.0004	17.01 ± 0.0011	19.23 ± 0.0003
Ca ²⁺	89.00 ± 0.0003	109.33 ± 0.0007	162.83 ± 0.0012	188.09 ± 0.0004
Ca2+ -TSW	68.96 ± 0.0002	85.34 ± 0.0008	94.69 ± 0.0062	86.55 ± 0.0007
Parameter	BN-14 h	BN-17 ½ h	BN-21 h	BN-24 ½ h
K ⁺	1009.43 ± 0.0006	1056.37 ± 0.0002	1066.25 ± 0.0012	1101.51 ± 0.0002
K^+ -TSW	574.16 ± 0.0051	615.69 ± 0.0067	615.79 ± 0.0052	609.09 ± 0.0048
Na ⁺	45.88 ± 0.0001	40.84 ± 0.0002	42.42 ± 0.0001	50.42 ± 0.0001
Na ⁺ -TSW	14.76 ± 0.0011	12.89 ± 0.0006	13.45 ± 0.0049	14.34 ± 0.0005
Ca^{2+}	174.31 ± 0.0017	164.90 ± 0.0005	174.66 ± 0.0014	177.46 ± 0.0016
Ca2+ -TSW	59.52 ± 0.0006	51.97 ± 0.0043	50.25 ± 0.0002	52.03 ± 0.0017

TSW: tartaric stabilized wine.

Conclusions

The study showed that different prefermentative maceration process times applied to the 'Băbească neagră' grape variety (Vitis vinifera L.) exert a significant influence on the content of volatile acids, pH and conductivity. It was found that the prefermentative maceration process does not exert a significant influence on the alcoholic content or the total acidity in wines that have not been tartaric stabilized. Tartaric stabilization exerted a significant influence on the content of the following parameters in the studied wines: total acidity, pH and conductivity. The prefermentative maceration and tartaric stabilization processes significantly influenced the content of phenolic compounds and the color parameters. The total phenolic compound content, anthocyanin content and wine color intensity increased with increasing maceration times. The only parameter that showed no synergistic influence by the two factors (maceration time and tartaric stabilization) was the total acidity; all other studied parameters showed a clear interaction between these two factors.

It was found that the prefermentative maceration processes change only the content of K+, meaning that its concentration in the studied wines has increased proportionally with the time of prefermentative maceration.

Regarding the tartaric stabilization, the influence of this process on all the studied cations was revealed, in terms of the decreasing concentration of all wines tartaric stabilized.

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