STUDY ON THE INFLUENCE OF VARIOUS MACERATION-FERMENTATION TECHNIQUES ON THE COLOUR OF RED WINES FROM FETEASCĂ NEAGRĂ GRAPES

STUDIU PRIVIND INFLUENȚA DIFERITELOR TEHNICI DE MACERARE-FERMENTARE ASUPRA CULORII VINURILOR ROŞII OBȚINUTE DIN STRUGURI DE FETEASCĂ NEAGRĂ

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Abstract. *The typicity of wines colour is a difficult but important problem that can be solved by correct quantification of a large segment of aspects. The objective of the present study was to evaluate the colour of red wines produced from Fetească Neagră grape variety from 3 Romanian vineyards, through different maceration procedures (traditional, thermo, roto-tank and carbonic). The identification of anthocyanins in wines made from the Romanian traditional grape variety Fetească Neagră was carried out and their profile was determined using HPLC. The relationship of anthocyanin profile and specific characteristics with different maceration treatments was investigated. The results showed that the different maceration treatments exerted important differences on the content of anthocyanins and important variations in the colour of Fetească neagră wines. The different kinds of maceration and the location of vineyards influenced the percentage of each anthocyanin in the specific profile of Fetească Neagră wine.*
Key words: anthocyans, Fetească neagră, maceration, colour difference.

Rezumat. *Tipicitatea culorii vinurilor este o problemă dificilă, dar importantă, care poate fi rezolvată prin cuantificarea corectă a unui segment larg de aspecte. Obiectivul prezentului studiu a fost de a evalua culoarea vinurilor roşii obținute din soiul de struguri Fetească neagră recoltat din 3 podgorii din România și vinificat prin proceduri diferite de macerare (clasic, termomacerare, cisterne rotative și macerație carbonică). S-a avut în vedere identificarea cu ajutorul HPLC a antocianilor și a profilului acestora în vinurile obținute din soiuri de struguri tradițional românesc Fetească neagră. A fost investigată relația dintre profilul antocianilor şi caracteristicile specifice induse de diferitele tratamente de macerare folosite. Rezultatele au arătat că diferitele tratamente de macerare prezintă influențe semnificative asupra conținutului de antociani şi induc variații importante în culoarea vinurilor de Fetească neagră. Variantele de macerare studiate şi locația podgoriilor influențează procentul de participare al fiecărui antocian în profilul specific pentru vinul Fetească neagră.*
Cuvinte cheie: antociani, Fetească neagră, macerare, diferență de culoare

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INTRODUCTION

Anthocyans are pigments of red, blue and purple colours, mainly occurring in cellular vacuoles of grape skin (Odăgeriu et al, 2007). Anthocyans are important compounds for characterization of red grape varieties; they are chemical markers in distinguishing varietal red wines (Zamfir et al, 2008). It is known that the mutual relations of anthocyans (the anthocyans profile) belongs to the vine variety, even though their absolute content in ripe grapes varies a lot and depends on factors that concern the climatic factors, such as intensity of light and temperature (Odăgeriu et al, 2008). Although the wine anthocyans composition is firstly determined by the genetic factor of the grape sort, the vinification parameters also have an important impact. It was shown that the maceration parameters have a significant influence on extraction of anthocyans from grape skins (Zamfir et al, 2009). The conditions of maceration, fermentation and maturation of wine influence the anthocyans composition, which is very significant, because the total concentration and composition of anthocyans determine the colour of red wines (Cotea et al, 2007).

Feteasca neagra is a Romanian local red variety of *Vitis silvestris*, which acquires its superior quality in the Iasi, Dealu Bujorului and Panciu vineyards where wine with a protected geographic origin are produced. This grape variety is very important for production of high-quality red wines.

MATERIAL AND METHOD

The experiments were done during September 2011 – March 2012, at the Oenology Laboratory of the University of Agricultural Studies and Veterinary Medicine “Ion Ionescu de la Brad” Iasi.

Vinification was carried out on 1000 kg of the Fetească neagră grape variety by random sampling in the 3 vineyards. The grapes were harvested at the full maturity.

After destemming and crushing three quarters of the total quantity- vineyard, pectolitic enzymes were added to better extract the colour. Maceration was performed in four ways: the classical maceration, roto-tanks maceration, thermo-maceration and carbonic maceration.

Treatment 1 (V1). Classical maceration: 50 kg of pomace was kept in static vessels, selected yeasts for fermentation were added, the cap was punched 3 times / day, for 3 days. After pressing, the wine was transferred into classic glass vessels, to complete its alcoholic and malolactic fermentation. The maceration process proceeded at a temperature of maximum 25 °C.

Treatment 2 (V2). Thermo-maceration: The pomace (50 kg) was divided in three. Two thirds of the obtained must was heated up to 80 °C and then transferred onto the pomace, bringing the temperature of the total mass up to 60 °C. When everything cooled, a 3 day maceration followed, with 3 times / day homogenization. After pressing, the wine was transferred into classic glass vessels, to complete its alcoholic and malo-lactic fermentation.

Treatment 3 (V3). Roto-tanks maceration: 50 kg of pomace was kept in roto-tanks, selected yeasts for fermentation were added, the content was homogenized 3 times/ day for 5 minutes, clockwise and anti-clock wise. The roto-tanks maceration lasted for 3 days. After pressing, the wine was transferred into classic glass vessels, to complete its alcoholic and malolactic fermentation. The maceration process proceeded at a temperature of maximum 29 °C.

Treatment 4 (V4). Carbonic maceration: The selected grapes (50 kg), the remaining fourth of the total quantity were transferred into a mini carbonic maceration tank. This had at the bottom a grill under which fermenting must of Fetească neagră (with the same physical-chemical characteristics as the one above) was kept. The grapes are positioned without being crushed. After 12 days, the obtained mass is destemmed and pressed; fermenting yeasts are added, the wine being transferred into classic glass vessels, to complete its alcoholic and malolactic fermentation

Chromatic parameters of the analysed wine samples were calculated according to CIE Lab 76 method, taking into consideration the registered absorption spectrum for each wine sample (Odăgeriu et al., 2007, 2008; Zamfir et al., 2008; Zamfir, 2009). A Specord S200 spectrometer and calculator were used. An automated registration and classification of absorption spectrums was copied in a file. To minimize analysis errors when determining absorbencies, specific vials were used, with an optical characteristic of 1.0 cm. The spectres were processed with a soft realised within the research group, for obtaining the chromatic parameters (L, a, b, C, H°), colour intensity (I) and hue (N).

The colour differences were also calculated with the ΔE 2000 formula, it was considered that, for values of ΔE smaller that the unity, the colours of two wines are seen as identical, or otherwise said, they cannot be sensorial differentiated.

Analysis of anthocyans was performed in a Hewlett-Packard HP-1100 high-performance liquid chromatography. The injected sample volume was 20 μL. Separation of anthocyans was carried out at the column C18 (250 mm × 4,6 mm, 5 μm particle size) at 25 °C. The chromatographic method conditions were as follows: mobile phase flow rate: 1,2 mL/min; DAD detection in the visible at 518 nm; mobile phase A: water:formic acid:acetonitrile 87:10:3; mobile phase B: water:formic acid:acetonitrile 40:10:60, with the elution program being a gradient starting from 6% to 60% to mobile phase B for 35 min. Anthocyans were identified in correlation to the retention time, elution sequence, and UV-VIS spectral properties.

For statistical evaluation of data obtained we applied Multifactor ANOVA statistical tests type. The Multifactor ANOVA procedure is designed to construct a statistical model describing the impact of two or more categorical factors Xj on a dependent variable Y. Tests are run to determine whether or not there are significant differences between the means of Y at the different levels of the factors and whether or not there are interactions between the factors. In addition, the data may be displayed graphically in various ways, including a multiple scatter plot, a means plot, and an interaction plot.

RESULTS AND DISCUSSIONS

Classification of the obtained wines according to their colour is similar to the order given by tracings of the absorption curves of the studied wines.

The order established by the values of total phenolic compounds of studied wines is the same with the hierarchy established based on anthocyans' content, the order established by absorption spectrums of each wine and the one drawn by digital simulation of wines' colour (tab. 1).

Table 1

Values of chromatic parameters of wines from Fetească neagră

No.	Grape variety	Colour computerised simulation	L	a	b	C	H
1	F.N.-V2-D. Bujorului		23.29	56.04	37.06	67.18	33.48
2	F.N.-V2-Adamachi		24.16	56.57	36.12	67.12	32.56
3	F.N.-V1-D. Bujorului		24.25	57.19	39.18	69.32	34.41
4	F.N.-V3-D. Bujorului		24.39	56.69	38.85	68.72	34.42
5	F.N.-V1-Adamachi		25.51	57.60	39.16	69.65	34.21
6	F.N.-V2-Panciu		27.47	59.69	40.59	72.18	34.22
7	F.N.-V3-Adamachi		28.63	59.05	36.35	69.34	31.62
8	F.N.-V3-Panciu		31.23	60.16	33.46	68.84	29.08
9	F.N.-V1-Panciu		33.45	62.50	37.93	73.11	31.25
10	F.N.-V2-Uricani		37.05	61.47	31.09	68.89	26.83
11	F.N.-V1-Uricani		41.55	61.99	29.71	68.74	25.60
12	F.N.-V3-Uricani		42.85	64.31	29.13	69.80	22.87
13	F.N.-V4-D. Bujorului		45.68	62.69	27.49	68.45	23.68
14	F.N.-V4-Adamachi		56.55	47.91	22.43	52.90	25.09
15	F.N.-V4-Panciu		58.59	45.89	18.28	49.03	21.24
16	F.N.-V4-Uricani		62.26	44.06	13.78	46.16	17.36

Analysing the colour differences obtained with the ΔE 2000 formula, the most rigorous one, it can be observed that the majority of wines can be sensorial differenced with some exceptions: variant F.N.-V1-Dealu Bujorului and variant F.N.-V3-Dealu Bujorului and F.N.-V1-Adamachi and variant F.N.-V2-Dealu Bujorului with variant F.N.-V2-Adamachi (fig. 1).

Nr. crt.	SOIUL	Nr crt	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	F.N.-V1-Dealu Bujorului	1	0,00	0,93	7,39	15,01	1,07	1,35	2,47	11,16	0,17	3,75	6,34	16,41	18,95	29,44	32,16	36,91
2	F.N.-V1-Adamachi	2	0,93	0,00	6,48	14,13	1,79	1,60	1,57	10,29	0,86	2,91	5,52	15,53	18,07	28,60	31,33	36,11
3	F.N.-V1-Panciu	3	7,39	6,48	0,00	7,89	7,98	7,28	5,08	4,29	7,32	3,88	2,50	9,27	11,83	22,81	25,61	30,38
4	F.N.-V1-R2-Uricani	4	15,01	14,13	7,89	0,00	15,42	14,64	12,88	4,00	14,92	11,33	8,93	1,49	4,02	15,54	17,98	21,90
5	F.N.-V2-Dealu Bujorului	5	1,07	1,79	7,98	15,42	0,00	0,87	3,33	11,52	1,08	4,17	6,63	16,80	19,35	29,89	32,56	37,27
6	F.N.-V2-Adamachi	6	1,35	1,60	7,28	14,64	0,87	0,00	2,95	10,72	1,31	3,42	5,81	16,02	18,58	29,23	31,89	36,60
7	F.N.-V2-Panciu	7	2,47	1,57	5,08	12,88	3,33	2,95	0,00	9,10	2,42	2,08	4,57	14,27	16,81	27,38	30,17	34,98
8	F.N.-V2-R2-Uricani	8	11,16	10,29	4,29	4,00	11,52	10,72	9,10	0,00	11,07	7,43	4,95	5,35	7,99	19,47	22,09	26,50
9	F.N.-V3-Dealu Bujorului	9	0,17	0,86	7,32	14,92	1,08	1,31	2,42	11,07	0,00	3,68	6,26	16,32	18,86	29,33	32,05	36,80
10	F.N.-V3-Adamachi	10	3,75	2,91	3,88	11,33	4,17	3,42	2,08	7,43	3,68	0,00	2,61	12,72	15,30	26,16	28,88	33,66
11	F.N.-V3-Panciu	11	6,34	5,52	2,50	8,93	6,63	5,81	4,57	4,95	6,26	2,61	0,00	10,28	12,91	24,07	26,75	31,49
12	F.N.-V3-R2-Uricani	12	16,41	15,53	9,27	1,49	16,80	16,02	14,27	5,35	16,32	12,72	10,28	0,00	2,74	14,48	16,79	20,57
13	F.N.-V4-Dealu Bujorului	13	18,95	18,07	11,83	4,02	19,35	18,58	16,81	7,99	18,86	15,30	12,91	2,74	0,00	11,63	13,81	17,46
14	F.N.-V4-Adamachi	14	29,44	28,60	22,81	15,54	29,89	29,23	27,38	19,47	29,33	26,16	24,07	14,48	11,63	0,00	2,78	6,74
15	F.N.-V4-Panciu	15	32,16	31,33	25,61	17,98	32,56	31,89	30,17	22,09	32,05	28,88	26,75	16,79	13,81	2,78	0,00	4,00
16	F.N.-V4-R2-Uricani	16	36,91	36,11	30,38	21,90	37,27	36,60	34,98	26,50	36,80	33,66	31,49	20,57	17,46	6,74	4,00	0,00
Nr. crt.	SOIUL	Nr crt	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

Fig. 1 - Nomogram of colour difference calculation between studied wines by ΔE 2000.

Area percentages values of each main anthocyan are the only ones based on which the studied wines can be differenced, with some exceptions like F.N.-V1-Dealu Bujorului, F.N.-V1-Adamachi and F.N.-V3-Dealu Bujorului. These presented the same value (0,45 ± standard deviation for each wine sample) when the ratio between the sum of participation percentage of acetylated anthocyans and the sum of participation percentage of coumarate anthocyans was calculated. The allure of the three chromatograms is very similar, situation explained by the use of the same grape variety, respectively Fetească neagră. These wines are the ones that were mentioned above as having no difference from a sensorial point of view, differences calculated with ΔE 2000 formula.

All the other variants were identified as different from a sensorial point of view, they have different values of the ratios between the sum of participation percentage of acylated anthocyans and the sum of participation percentage of coumarate anthocyans, as well as different values of the ratios of the area percentages' sum of monoglicosidic anthocyans and area percentages' sums of acylated and coumarate anthocyans. Even though the chromatograms had similar allures, they could be differentiated as shoen above.

After applying statistical tests ANOVA to the different types of wine made from Fetească neagră variety, harvested in different vineyards and who have applied several maceration-fermentation technology to show any influence on the characteristics that give them colour, it showed that the vineyard of origin of these grapes exert a distinct significant influence only on the proportions of participation of two anthocyanins, delphinidin-3-monoglicozide respectively petunidin-3-monoglicozide, while the other anthocyanins not show any effect on proportions calculated.

Table 2

Results of the ANOVA tests on main anthocyans from wines obtained from Fetească neagră grape sort

ANTHOCYANS	**F - the influence of vineyard**	***F-crit***
Delfinidin-3-monoglicozide	6,300**	5,412
Cyanidin-3-monoglicozide	0,907 ns	3,259
Petunidin-3-monoglicozide	6,229**	5,412
Peonidin-3-monoglicozide	1,310 ns	3,259
Malvidin-3-monoglicozide	1,980 ns	3,259
Peonidin-3-monoglicozide acylated	3,085 ns	3,259
Malvidin-3-monoglicozide acylated	2,491 ns	3,259
Peonidin-3-monoglicozide coumarate	0,917 ns	3,259
Malvidină-3-monoglicozide coumarate	1,178 ns	3,259
Σgl/(Σacil+Σcum)	0,942 ns	3,259
Σacil/Σcum	0,922 ns	3,259
	F - the influence of maceration-fermentation technology	***F-crit***
Delfinidin-3-monoglicozide	18,095*	10,804
Cyanidin-3-monoglicozide	3,498***	3,490
Petunidin-3-monoglicozide	17,443*	10,804
Peonidin-3-monoglicozide	9,216**	5,953
Malvidin-3-monoglicozide	6,881**	5,953
Peonidin-3-monoglicozide acylated	8,909**	5,953
Malvidin-3-monoglicozide acylated	0,162 ns	3,490
Peonidin-3-monoglicozide coumarate	6,428**	5,953
Malvidină-3-monoglicozide coumarate	4,626***	3,490
Σgl/(Σacil+Σcum)	0,800 ns	3,490
Σacil/Σcum	1,073 ns	3,490

ns - no statistically significant influence where P-value □ p = 0.05;
* - Very significant statistical influence where P-value □ p = 0.001;
** - Distinct significant statistical influence where P-value □ p = 0.01;
*** - Significant statistical influence where P-value □ p = 0.05.

When evaluating the influence of maceration-fermentation technology on participation percentage values of anthocyanins, it is noted that the calculated proportions reports on anthocyanins malvidin-3-acylated monoglicozide not exercise any significant influence, the rest on other anthocyanins were showed statistical significant influence (cyanidin-3-monoglicozide and malvidin-3-monoglicozide coumarate), distinct significant influence (peonidin-3-monoglicozide, malvidin-3-monoglicozide, peonidin-3-acylated and peonidin-3-coumarate), and very significant influence (delphinidin-3-monoglicozid and petunidin-3-monoglicozid) (tab. 2).

CONCLUSIONS

Considering the use of maceration fermentation techniques adequate to composition characteristics and to sanitary parameters of the crop, wines that "simulate" very well the colour prints of wines obtained from other vineyards or other maceration-fermentation technologies could be obtained.

A clear influence of the origin of the harvest is registered on the percentage values of the anthocyans in Fetească neagră wines. These observations certify the fact that the compared wines are different from a maceration-fermentation technological point of view and also from the origin area of the grapes (viticultural centers or different vineyards).

As a general conclusion, testing the percentage value of petunidin-3-monoglicoside is the main factor that can differentiate wines according to origin vineyard or viticulture centre.

Of course, there are also other instrumental determinations, that, together with sensorial evaluations are useful and can generate high precision results, but they cannot be used by themselves. Wine's colour appreciation is strongly connected to all factors that can influence the physical-chemical and sensorial state of the studied wines.

REFERENCES

1. **Cotea V.V., Liliana Rotaru, Nechita B., Mustea M., Zamfir C.I., 2007** - *A model of traceability system in the viticultural sector of Romania for improving food safety.* XXXth OIV World Congress, Budapest, 10–16 June, Section 1.3.: Viticulture.
2. **Odăgeriu G., Niculaua M., Cotea V.V., Zamfir C.I., Nechita B., 2008** - *The variation of anthocyans profile specific to some red wines.* XXXIth OIV World Congress, Verona, 15–20 June, Section P II.C 100: Oenology - Methods of analysis.
4. **Zamfir C.I., Cotea V.V., Colibaba Cintia, Niculaua M., Chiriță Otilia, 2008** - *Study of anthocyans of Fetească neagră wines obtained by different maceration treatments.* XXXIth OIV World Congress, Verona, 15–20 June, Section P II.A 031: Oenology - Oenological techniques for the reinforcement of territorial and wine variety identity.
5. **Zamfir C.I., Odăgeriu G., Cotea V.V., Niculaua M., Colibaba Cintia, Georgescu O., 2009** - *Aspects Concerning the use of Chromatic Characteristics in the Differentiation and Classification of Red Wines from Local Grape Varieties.* Bulletin USAMV, nr. 66 (1-2)/2009, Print ISSN 1843-5254; Electronic ISSN 1843-5394.