

STUDY OF PHENOLIC COMPOUNDS IN RED WINES OBTAINED IN IAȘI VINEYARD BY DIFFERENT MACERATION-FERMENTATION METHODS

STUDIUL COMPUȘILOR FENOLICI DIN VINURILE ROȘII OBTINUTE ÎN PODGORIA IAȘI PRIN DIFERITE METODE DE MACERARE-FERMENTARE

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***Abstract.** The grape contains a high quantity of phenolic compounds responsible for color, which are transmitted into wines during the maceration-fermentation processes. The present paper's objectives are to monitor the extraction level, concentration evolution and analysis of these phenolic compounds in conjuncture with different maceration-fermentation procedures used. By approaching modern analytical methods, the correlation of studied phenolic compounds concentration variation with the maceration-fermentation technique will be facilitated. In order to underline this correlation, grapes from Fetească neagră and Merlot varieties were used, harvested at technological maturity from Iași-Copou vineyard; the grapes were processed into wine by using six maceration-fermentation techniques and the obtained products were physical and chemical analyzed for the different phenolic compounds responsible for color. The results reflect the extraction degrees for the analyzed compounds from the obtained wines.*

Key words: phenolic compounds, maceration, extraction, color

***Rezumat.** Strugurii roșii conțin o cantitate însemnată de compuși fenolici responsabili de culoare, transmiși în vinuri în timpul proceselor de macerare-fermentare. Prezenta lucrare are ca obiective urmărirea gradului de extracție, evoluția concentrației și analiza acestor compuși în conjunctura diferitelor procedee de macerare-fermentare folosite. Se urmărește, de asemenea, stabilirea unei corelații între variația concentrației compușilor fenolici studiați cu metoda de macerare-fermentare folosită. Pentru a evidenția această corelație, s-au folosit struguri aparținând soiurilor Fetească neagră și Merlot, recoltați la maturitate tehnologică din podgoria Iași-Copou; aceștia au fost vinificați prin șase modalități de macerare-fermentare, iar vinurile astfel obținute au fost analizate fizico-chimic din punct de vedere al concentrațiilor diferiților compuși fenolici responsabili de culoare. Rezultatele obținute reflectă gradele de extracție a compușilor analizați din vinurile realizate.*

Cuvinte cheie: compuși fenolici, macerație, extracție, culoare

INTRODUCTION

Red wines, as the name shows, differ from white ones, mainly because of their high content of phenolic compounds, out of which some are specific to them (anthocyanins). They are distinguishable by their aroma and taste which also are

different from those in the white wines. Compared to aromatic wines, whose odor and taste are defined by many terpenic compounds, the red ones have a varietal aroma that is given by different compounds (for example, in the case of Cabernet sauvignon, a great importance regarding aroma is represented by pyrazines)[2]. Regarding the above information, it is understood that, in red wine technology, the main objective is extraction from skins and diffusion in must of these constituents that imprint the wine with their specific characteristics. The extraction of colored compounds and aromas from grape skins, its main deposit, is done in the pre-fermentative and even in the fermentation stages, though maceration [1,5].

The present paper wants to underline the technological conditions which favor the phenolic compounds extraction from red grapes, in order to optimize the wine making methods and to obtain wines with superior chromatic characteristics.

MATERIAL AND METHOD

Research concerning the influence of different maceration-fermentation technological processes on the extraction degree for phenolic compounds from red grapes has been conducted in the Oenology Laboratory of the University of Agricultural Studies and Veterinary Medicine Iasi. Therefore, Fetească neagră and Merlot grape varieties have been harvested from Copou vineyard, which were processed by using six maceration fermentation techniques: classical maceration, ROTO-tanks maceration, thermo-maceration, micro-wave maceration, ultrasound maceration and cryo-maceration. The characteristics of each maceration-fermentation technique used are:

– Classical maceration: selected yeasts were added to the must, which was in contact with the skins for 3 days at 20 °C; when the alcoholic concentration reached 9%, the must was separated from the skins and the fermentation process continued until all sugars were depleted [2];

– ROTO-tank maceration: selected yeasts were added to approximate 40 L of marc, which was maintained in rotating tanks for three days, and the phases separation was conducted when the alcoholic separation reached 9%; the fermentation process ended in glass containers until all sugars were depleted [1,6].

– Microwave maceration: 5 kg marc underwent microwave irradiation for 10 minutes at 650 W power; the marc was then pressed; the fermentation process was conducted as stated above [4].

– Thermo-maceration: the marc was subjected to thermal treatment at 60-75 °C, for 30 minutes. A device for thermal treatment in must was used, with the following technical characteristics: tank capacity 20-40 kg, maximum temperature 80°C, maximum productivity 40 kg/hour, power 10 kW. The minimal threshold for marc thermal treatment is 50°C. After the thermal treatment, the fermentation process took place as in the above cases.

– Cryo-maceration: fresh grapes have been slowly frozen -30 °C, and then a fast destemming and crushing process took place; selected yeasts were added to the warmed must at 12°C. The fermentation process took place as in the above cases.

– Ultrasound maceration: represents the easiest way to destroy the cellular wall and obtain the extract. Ultrasound cavitation builds powerful forces which mechanically destroy the cellular wall and improve the transfer. As the compound that must be solved is surrounded by an insoluble structure, in order to extract it, the cellular wall needs to be destroyed. Therefore, the destemmed grapes were subjected to this treatment for 15

minutes. This process is not widely used in red grape processing technology but it is successful in obtaining aromatic white wines.

Many technological operations used were common to all variants: crushing and total destemming, SO₂ treatment of the marc, (doses of 0,05 g/L) in order to insure antioxidant and antiseptic protection, proteolytic enzymes addition in order to increase fluid extraction, adding the same yeast to the must, *Saccharomyces oviformis* (S.C.D.V.V. Iași collection) – characterized by a high alcoholigenous capacity, SO₂ resistance and non-foaming effect –, marc pressing using a low-capacity pneumatic press, alcoholic and malo-lactic fermentation (using endogenous lactic bacteria), oenological gelatin treatment (doses of 0,1 g/L), racking, filtration with a sterile filter and bottling [7].

The obtained wines were analysed: density, total acidity, volatile acidity, free and total SO₂ content, reducing sugar content, alcoholic concentration, and non-reducing dry extract. Also, the phenolic compounds were analysed: total anthocyanins content, total polyphenolic index, Folin-Ciocalteu index, color determination (CIELAB 76), anthocyanins' profile and phenolic acids content. An UV-VIS Analytik Jena Specord 200 spectrophotometer and a Hewlett-Packard HP-1100 HPLC with C18 column were used [8].

RESULTS AND DISCUSSIONS

The main compositional characteristics of the obtained wines (samples 1-12) are shown in table 1, while the analyses results regarding polyphenolic compounds are stated in tables 2 and 3.

Nine important anthocyanins were identified and quantified from the obtained wines (Fetească neagră and Merlot), by applying the six technological methods previously described: delphinidin (Dp-3-gl), cyanidin (Cy-3-gl), petunidin (Pt-3-gl), peonidin (Pn-3-gl) , malvidin (Mv-3-gl); peonidin acetilate (Pn-3-gl-ac), malvidin acetilate (Mv-3-gl-ac), peonidin cumaril (Pn- 3-gl-p-cum) and malvidin cumaril (Mv-3-gl-p-cum). From the registered data, we can conclude that the maceration-fermentation methods using ultrasounds and low temperature are not recommended for obtaining red wines with high phenolic compound concentration. By analyzing the other data – the Folin Ciocalteu index and chromatic parameters – the previous conclusion cannot be denied.

From all of the above, one can state that a high efficiency in extracting phenolic compounds responsible for the red color in wines can be obtained by using either classical maceration, either ROTO-tank maceration, but one must keep in mind that the marc should undergo a thermal treatment and a pectolytic enzyme addition.

Table 1

**Physical-chemical characteristics of wines obtained from Fetească neagră and Merlot grape varieties
Through different maceration-fermentation procedures**

Sample	Alcohol (%)	TA (g/L)	VA (g/L)	Free SO ₂ (mg/L)	Total SO ₂ (mg/L)	Sugars (g/L)	Density (g/L)	NRE (g/L)
FETEASCĂ NEAGRĂ								
Classical maceration	10,8	6,9	0,21	41	168	<4	0,9932	19,8
ROTO-tank maceration	11,2	7	0,18	15	53	<4	0,9953	26,3
Microwave maceration	11,2	6,6	0,2	10	71	<4	0,9922	18,3
Thermo-maceration	11,1	6,4	0,18	37	184	<4	0,9923	18,3
Cryo-maceration	11,7	4,5	0,17	25	80	<4	0,9898	13,7
Ultrasound maceration	11,3	7	0,21	29	111	<4	0,9930	20,9
MERLOT								
Classical maceration	11,6	6,34	0,46	16,45	70,77	<4	0,9930	24,2
ROTO-tank maceration	11,44	8,15	0,44	18,01	115,15	<4	0,9927	25,0
Microwave maceration	11,2	6,64	0,48	15,83	126,02	<4	0,9913	20,3
Thermo-maceration	10,94	7,55	0,44	17,07	92,81	<4	0,9925	22,9
Cryo-maceration	11,86	5,74	0,52	15,52	68,29	<4	0,9923	25,0
Ultrasound maceration	10,81	6,64	0,66	20,79	167,61	<4	0,9923	22,2

Table 2

**Phenolic indexes, total anthocyanins quantities, visual characteristics
(L, a, b) –CIE Lab 76 method values**

No.	Sample	TPI	FCI	Total anthocyanins (mg/L)	Luminosity L	a r (+) - v (-)	b g (+) - a (-)	Chromaticity	Hue
FETEASCĂ NEAGRĂ									
1	Classical maceration	28,83	52	211,13	95,47	4,57	1,63	0,16	0,91
2	ROTO-tank maceration	48,91	48,72	238,12	89,37	12,29	2,21	0,36	0,77
3	Microwave maceration	29,7	23,67	355,82	94,94	5,65	1,26	0,17	0,83
4	Thermo-maceration	28,27	22,26	228,93	92,83	8,21	1,61	0,25	0,79
5	Cryo-maceration	9,09	6,79	33,92	98,43	0,49	1,22	0,06	1,51
6	Ultrasound maceration	23,29	39,43	112,58	97,18	2,12	1,85	0,11	1,17

Table 2 continuing

MERLOT									
7	Classical maceration	37,68	37,67	301,14	86,53	19,10	0,13	0,44	0,56
8	ROTO-tank maceration	39,75	39,11	301,87	86,52	17,93	1,11	0,45	0,63
9	Microwave maceration	16,85	11,04	166,18	94,95	6,81	0,99	0,17	0,70
10	Thermo-maceration	27,94	52,22	298,40	88,91	16,78	-0,05	0,36	0,52
11	Cryo-maceration	11,31	8,91	37,24	98,06	0,98	2,19	0,08	1,64
12	Ultrasound maceration	14,41	13,7	127,83	96,65	4,42	1,16	0,12	0,74

Table 3

Percentual values of the nine anthocyanins present in wines (% of total)

No.	Sample	Dp	Cy	Pt	Po	Mv	Po-a	Mv-a	Po-cm	Mv-cm	Σ Ant.ac +Ant.cm	Σ Ant.ac/ Σ Ant.cm	Mv/ Σ Mv -COOR	Σ Ant./ Σ Ant.- COOR
FETEASCĂ NEAGRĂ														
1	Classical mac.	10,45	1,63	11,64	11,72	56,65	0,53	0,61	1,97	4,81	7,93	0,17	10,45	11,62
2	ROTO-tank mac.	10,63	3,02	11,98	14,37	46,48	1,85	6,49	1,67	3,51	13,52	1,61	4,65	6,39
3	Microwave mac.	11,57	1,14	12,99	11,12	53,34	0,59	1,95	2,08	5,23	9,84	0,35	7,44	9,16
4	Thermo-mac.	10,05	1,62	13,74	10,62	55,55	0,87	2,11	1,52	3,92	8,42	0,55	9,21	10,87
5	Cryo-mac.	1,01	0,50	1,82	5,49	80,31	2,76	5,71	0,54	1,85	10,87	3,54	10,61	8,20
6	Ultrasound mac.	4,86	1,17	9,46	12,81	66,13	0,54	1,96	0,82	2,26	5,58	0,81	15,66	16,92
MERLOT														
7	Classical mac.	11,32	2,24	10,48	12,30	40,92	4,08	10,87	2,92	4,86	22,74	1,92	2,60	3,40
8	ROTO-tank mac.	9,71	2,89	10,20	13,39	41,55	4,56	11,40	2,34	3,96	22,26	2,54	2,71	3,49
9	Microwave mac.	7,40	0,64	10,45	6,33	63,53	1,59	4,56	1,19	4,31	11,65	1,12	7,17	7,58
10	Thermo-mac.	10,95	1,57	11,58	10,41	42,99	4,76	10,25	2,67	4,82	22,50	2,01	2,85	3,44
11	Cryo-mac.	0,74	0,22	2,43	7,54	83,27	0,82	2,72	0,55	1,71	5,80	1,57	18,81	16,25
12	Ultrasound mac.	8,13	1,30	9,77	11,30	58,77	2,33	5,55	0,65	2,20	10,73	2,76	7,58	8,32

CONCLUSIONS

The obtained values for determining the total polyphenolic index and the anthocyanins' concentration from all wine samples show that the highest degree of extraction was registered at ROTO-tanks maceration variant; high values were also registered at variants obtained with thermo-maceration. The wines obtained by cryo-maceration and ultrasound maceration had a low phenolic concentrations and low anthocyanins content (except malvidin-3-monoglucoside).

The obtained results by CIELAB 76 come to strengthen the previous mentioned issues: the wines obtained through thermal treatment have superior chromatic characteristics to those obtained through cryo-maceration or ultrasound maceration.

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