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STUDIES ON THE ANALYTICAL CHARACTERISTICS OF WINES OBTAINED FROM VINE VARIETY WITH BIOLOGICAL RESISTANT

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Abstract

Obtained grapes from vine varieties with biological resistance (the varieties of interspecific vines): Seyval, Admira, Radames, Valérien, Brumăriu and Purpuriu were vinified by traditional methods, and the obtained wines were analysed in terms of composition and as aromatic profile, compared with a control wine, of table wine, provided from an vinifera vines. The chemical analysis of wines have referred to alcoholic strength, total acidity, volatile acidity, total dry extract, glycerol, phenolic compounds and anthocyanins for the Purpuriu variety etc. The aromatic profile of wines was determined by the gas chromatography method coupled with mass spectrometry.
The result of analysis showed that Admira, Seyval and Radames wines presents a high alcoholic strength (11.8 vol% alcohol, 12.1 vol% alcohol, respectively 11.0 vol% alcohol) as well as higher values of the total dry extract and glycerol, compared to the control variety.
Volatile acidity of wines was slightly elevated for most varieties with biological resistance, a higher value presenting at the Pupuriu wine. In the case of Purpuriu wine was analysed the content from the phenolic and anthocyanins compounds, their value being reduced.
In terms of the content in volatile compounds, the main analysis of compounds show a predominance of 3-methyl-1-butanol (Isoamyl alcohol), 2-methyl propanol, Isoamyl acetate, Ethyl propanoate, both in the case of varieties with biological resistance, even in the vinifera variety case. Isoamyl acetate showed high values in the case of varieties with biological resistance and very low values in the case of vinifera wine.
Overall, the wines from the two groups of grapes varieties showed different values of the main volatile compounds, standing out a clear difference between them.

Key words: *vine varieties with biological resistance, volatile compounds, gas chromatography/mass spectrometry methods.*

INTRODUCTION

The varieties with biological resistance are interspecific hybrids (American varieties x *vinifera* varieties) with a good and very good resistance to major diseases and pests of vine, including *phylloxera* (Grecu V., 2010). The limited number of treatments that they are required for the culture of these varieties of vines has made they to be called ecological varieties and the grape consumption, as the products resulting from their processing to show a increased interest for the consumer (Visan L. et al., 2007).
In terms of the vinification varieties with biological resistance, the current legislation in the field of the *Vine and of Wine* foresee their usage only as table wine, due to lower quality and a lower resistance in time, compared with the *vinifera* varieties.
However, some varieties with biological resistance for wine have the ability to accumulate a good concentration of sugars and respectively lead to the obtaining of wines with a high alcoholic strength, comparative with *vinifera* varieties.
Also, the composition parameters, such as the total content of dry extract, in glycerol etc., they can record higher values, wich can be compare with *vinifera* varieties.

MATERIALS AND METHODS

Grapes from the varieties with biological resistance, Admira, Seyval, Valerién, Radames, Brumăriu and Purpuriu and a variety of table *vinifera*, as a control variety, were vinified

after a classic scheme under similar conditions. The wines obtained were analyzed in terms of physico-chemical: alcoholic strength (vol% alcohol), total acidity (g/L sulfuric acid), volatile acidity (g/L acetic acid), total dry extract (g/L) and glycerol (g/L). Based analyzes were performed by standard methods (Tardea C., 1980): ebulliometer method for alcoholic strength; titrimetric method for total acidity; distillation method *Saunier-Cazenave* for acidity volatile; Tabarié method for total dry extract and volumetric method for glycerol.
The polyphenolic composition of Purpuriu wine was judged by the content in polyphenols and anthocyanins. Analyzes have been carried out in the wine by UV-VIS spectrometry techniques (Giusti M, 2001). Total content of polyphenols have been determined by IPT technique (g/L gallic acid) (Ribereau-Gayon J, 1978). The anthocyanins were determined by the discoloration technique with SO_2 (Dallas C., 1994).
Specific Extraction of Volatile Compounds.
200 mL of wine, placed in a conical flask, were successively extracted (3 x 20 min) at 0°C with 3 x 25 mL of freshly distilled dichloromethane and then centrifuged for 15 min. The three organic extracts were pooled, dried with anhydrous sodium sulfate and concentrated to 5 mL in a Danish concentrator (45°C), then to 1 mL under a stream of nitrogen (Baek H. et al., 1997; Serot Th. Et al., 2001).
Quantitative analysis of volatile compounds identified in wines by GC/MS.
1μL from each extract was injected into an HP 5-MS capillary column with dimensions: 30 m x 0.25 mm x 0.25 mm (film thickness). Column temperature: 30° C for 10 min., followed by temperature gradient 10° min^{-1} up to 80° C, then gradient of 25° C/min. up to 250°C where stationed 10 minutes. Detector and injector temperatures are: 280° C and 250°C resp. Carrier gas is He, flow-0.5 mL min^{-1}. MSD conditions are: temperature 180°C ion source, ionization energy 70 eV, mass limit of 20-400 amu, electronic multiplier voltage 1700V, scan rate 1.60 s^{-1}. Injection mode: split, opening after 60 sec. and the split flow: 20 mL min^{-1}.
Quantitative determination and identification of volatile compounds based on the comparison of retention indices (RI), mass spectra and the estate of odors. Identification is based on the standard MS library Wiley (Serot Th., 2001; Visan L., 2007).

RESULTS AND DISCUSSIONS

The results show that wine obtained from the control *vinifera* variety is slightly superior towards the studied varietals, regarding the quality of wine, which was expected.
However, among the varieties studied there are valuable varieties, that we can recommend for the production of table wines.
Of these it distinguish especially the varieties Admira, Seyval and Radames, which present superior values of main quality parameters. At the sensory analysis them were positive appreciated.
The analyzed varieties belong to the same ecopedological areas and were subject to the same cultural techniques.
Therefore, the examined parameter values reflects their own genetic potential of the variety. The obtained wine from the Seyval variety presents a *alcoholic strength* of 12.1 vol%, the variety being already known that it has a good ability to accumulate sugars, so with a high alcoholic potential. Also with a higher alcoholic strength, is presented the Admira wine (11.8 vol%) and Radames (11.2 vol%). The Brumariu variety accrues sugars in lower quantity and produce wine with a lower alcoholic strength (9,8 vol%). Below the average varieties lies the Purpuriu variety with 9,4 vol% alcohol (Figure 1).
Total dry extract represent an important element in the analytical characterization of a wine. Its value at the Romanian wines vary in larger limits between 13 and 35 g/L, higher values it show at the wines from quality varieties. From this point of view, the analyzed wines were within the permissible limits, even with high values. The Seyval variety (20.1 g/L), Admira (19 g/L) and Radames (17.4 g/L) shows superior values of the extract, values comparative with the control variety, *vinifera* (Figure 1).
The glycerol enters, together with other constituents in composing of dry extract of the wine; due the sweet taste, his presence prints a softness to the wine, harmony, suppleness; occurs, also in preserving the flavors.

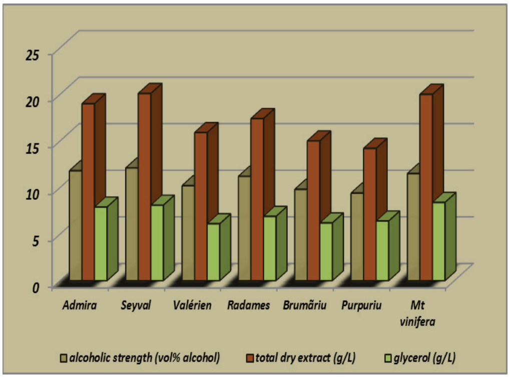

Figure 1. The Main Physico-Chemical Parameters Analyzed wines

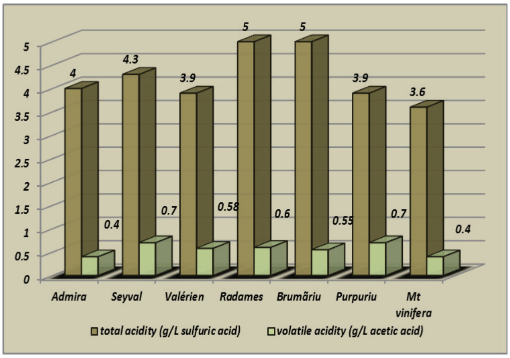

Figure 2. Total Acidity and Volatile Acidity of the Analyzed wines

The values content in glycerol are superior in the case of wines Seyval, Admira and Radames, representing approx. 1/11 from the alcoholic weight content of wines (Figure 1).

Total acidity has a great influence on the organoleptic characteristics of wines, concerning stability and maturation of wines.

It is noted from this point of view, that the total acidity of obtained wines from the biological resistance varieties is higher, compared with the *vinifera* variety, but without being affected the organoleptically balance of wine (Figure 2).

Volatile acidity is an extremely important component in the qualitative appreciation of wine. Volatile acids occur as by-products during alcoholic fermentation, as well as in other fermentations or processes, that occur during the wine evolution. It is known that the yeast influences the concentration in volatile acids from wine, which is why the wines were obtained by fermentation guided with yeasts selected from the same strain *Saccharomyces Cerevisiae.*

Thus, it appears that the most wines showed a high value of volatile acidity, except the Admira wine (0,40 g/L) with the same value of volatile acidity with the control wine (Figure 2).

In the case of Purpuriu wine, the value content in polyphenols and anthocyanin content (mg/L) have recorded low values (Figure 3, a, b).

Content analysis in wine flavors

Organic esters, originate from combining the organic acids with alcohols; many of esters have pleasant smell, some with a floral smell, fruity etc. In the analyzed wines, in higher concentration were identified many esters: *Isoamyl acetate*, pleasant smell, floral; Isoamyl

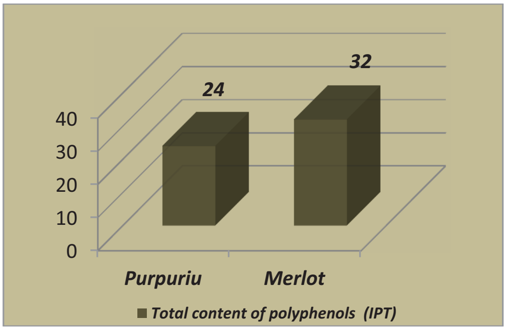

Figure 3 a. Polyphenol content in Purpuriu wine (IPT) compared to the control wine

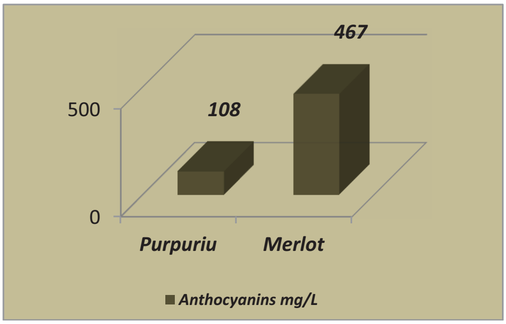

Figure 3 b. Anthocyanin content in Purpuriu wine (mg/L) compared to the control wine

acetate, the responsible ester for the bouquet the young wines, obtained from certain varieties; it is found in higher concentration in the studied wines from the varieties with biological resistance and in lower concentration in the *vinifera* wines (Table 1).

Table 1. Identified volatile compounds in the wines analyzed (µg/L)

Volatile compounds	Admira	Radames	Seyval	Valérien	Purpuriu	Mt *vinifera*
2-Methylpropan-1-ol	658	432	1587	6198	2500	78.2
3-Methyl-1-butanol (Isoamyl alcohol)	1020	9306	11245	13274	10121	4864
2-Methyl-1-butanol	842.9	789.1	823.4	989.5	1011.0	3854
1-Hexanol	43.9	28.7	17.9	121.8	40.1	18
2-Ethylhexanol	0.00	1.1	0.4	2.3	0.4	5.8
2-Phenylethanol	1.2	0.5	0.5	0.6	0.4	1.5
3-Methylbutanal	38.0	4.2	10.2	6.3	39.25	11.2
2-Methylbutanal	21.08	24.54	13.9	6.8	10.2	35.14
Heptanaldehyde	0.00	11.3	62.4	0.9	0.8	0.5
2-Ethylhexanal	0.00	0.2	4.1	0.00	1.9	2.8
Benzaldehyde	1.8	2.7	2.1	5.3	9.2	17.2
Methyl ethanoate	0.00	4.8	5.9	10.2	2.9	0
Ethyl propanoate	282.8	270.9	319.8	539.6	582.1	3775.2
Propyl acetate	0.00	0.00	0.00	133.1	124.5	87.9
(3-Methylthi opropylisothiocyanate	0.00	0.00	4.2	1.5	245	0
Methyl propanoate	984	541.2	632.5	501.2	412.0	42
2-Methylpropyl ethanoate	323.2	238.48	41.2	214.9	239.7	87.8
3-Methylbut-1-yl ethanoate	41	0.4	0.3	1.12	1.0	0
Ethyl butanoate	249.3	2.8	264.2	519.03	189.5	1915
(E)-2-Butenoic acid ethyl ester	0.00	4.9	5.2	1.4	1.0	0
2-Methylbutanoic acid, methyl ester	164.1	15.7	34.1	25.3	14.0	265
Isoamyl acetate	467.4	1152.4	730	1258.9	695.6	58.2
Ethyl pentanoate	0.00	0. 7	3.9	12.5	32.1	0
Methyl hexanoate	0.7	3	2.5	8.2	2.4	4
Ethyl octanoate	79.13	135.2	59.6	296.5	42.7	10.4
Ethyl hexanoate	512.3	640.1	390.1	998.9	378.4	15.3
Ethyl acetate	2.5	1.9	2.1	13.4	2.5	1.3
Methyl octanoate	0.7	1.2	0.9	4.3	1.1	6.4
2-Octanone	0.3	0.5	0.3	0.00	0.7	0
Linalol	0.2	4.5	0	2.1	0	15
Limonene	54.12	198.7	145.1	482	325.1	2.4
β-pinene	2.5	6.9	11.1	21.4	5.1	0.9

Other esters identified in higher concentration in the studied wines: *Ethyl propanoate* (pine-apple-like odor), with high concentration in *vinifera* wine; *Ethyl butanoate* (it has a fruity odor, similar to pineapple) with high concentration in *vinifera* wine;

Methyl propanoate (is a volatile ester with a sweet, fruity, rum-like odor); in the *vinifera* wine is found in low concentration; *2-Methylpropyl ethanoate* (like many esters it has a fruity or floral smell at low concentrations and occurs naturally in raspberries, pears and other plants.

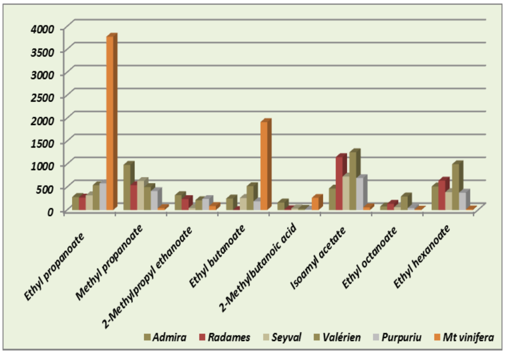

Figure 4. The main esters of a Analyzed wines (µg/L)

At higher concentrations the odor can be unpleasant and may cause symptoms of central nervous system depression such as nausea, dizziness and headache); *Ethyl hexanoate* ; is an apple-flavoured ester; in the *vinifera* wine is found in low concentration (Figure 4).

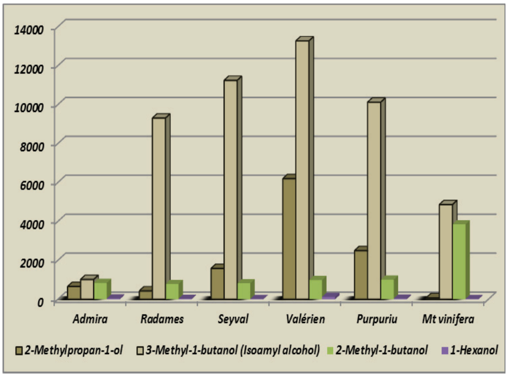

Figure 5. Concentration of higher alcohols

In terms of *higher alcohols*, were identified: *2-methylpropan-1-ol*, in high concentrations in wines of hybrids and in much lower concentration in *vinifera* wine. Isobutanol (2-methylpropan-1-ol) is produced naturally during the fermentation of carbohydrates and may also be a byproduct of the decay process of organic matter; 3-*Methyl-1-Butanol (Isoamyl alcohol);* it is one of the components of the aroma of *Tuber melanosporum*; *2-Methyl-1-butanol* (it is also one of the components of the aroma of *Tuber melanosporum*, the black truffle).

2-Methyl-1-butanol was found in higher concentration in all wines, but especially in the *vinifera* wine (Chisholm M. et al., 1994); *1-Hexanol* was found in higher concentrations in the hybrids wines and lower in the *vinifera* wine (Figure 5).

The aldehydes are very important compounds in wine bouquet formation (Figure 6); in the wines analyzed were identified: *3-Methylbutanal (isovaleraldehyde);2-Methylbutanal (Butyraldehyde);heptanaldehyde; 2-Ethylhexanal (2-Ethylhexaldehyde); Benzaldehyde etc.*

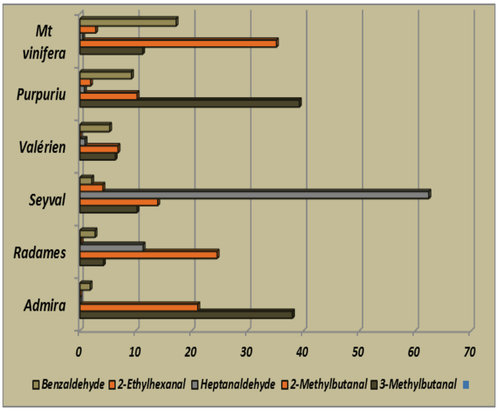

Figure 6. Aldehyde content of the Analyzed wines

Of the terpenes, were identified: *Linalool*, in low concentrations in the hybrid wines and in higher concentrations at the *vinifera* wine; *limonene*, characteristic to all the hybrid wines analyzed (Figure 7).

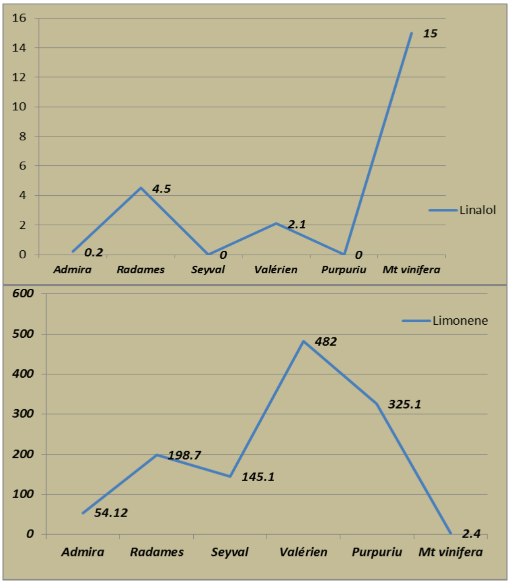

Figure 7. Concentration of terpenes

CONCLUSIONS

Of the analyzed wines, obtained from inter-specific hybrids (wines with biological resistance) showed superior values of main quality parameters (alcoholic strength, extract, glycerol, total acidity and volatile acidity) the Admira, Seyval and Radames wines; the values of these parameters are comparable to the *vinifera* table wines.

At the GC/MS analysis, regarding the volatile compounds and the concentration in flavours of the wines were identified:

- *Ethyl butanoate, Ethyl propanoate*, with a floral smell or fruity, both in the hybrid and *vinifera* wines;
- *Isoamyl acetate*, the responsible ester for the bouquet the young wines; it is found in higher concentration in the studied wines from the varieties with biological resistance and in lower concentration in the *vinifera* wine;
- *Methyl propanoate, 2-Methylpropyl ethanoate*; *Ethyl hexanoate:* in higher concentration in the studied wines from the varieties with biological resistance and in lower concentration in the *vinifera* wine;
- Of the higher alcohols, *2-Methylpropan-1-ol*, a was identified in higher concentrations in the wines of hybrids and in lower concentration in *vinifera* wine;
- Of terpenes, *linalool* is characteristic to the *vinifera* wine and *limonene* is characteristic to all wines of hybrids analyzed.

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