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Eonological parameters of *Fetească regală* wines subjected to aging with oak chips of and barrel during three periods of time

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Abstract Oenological parameter of "Fetească regală" wines aged for 30, 60 and 90 days in oak barrel natur (untoast) and with oak chips with natur and medium toast, have been analysed. Statistical analysis showed difference among wines due to the ageing variants and time. Regarding alcoholic content, the parameter decreased with time for all ageing variants. Volatile acidity increased with time, due to the oxidation of ethanol. The total acidity, the non-reducing dry extracts and total dry extract slightly decreases in aged samples during time. Alcohol content and volatile acidity were enological parameters most influenced by the ageing time and toast factors. The other oenological parameters were singnificantly dependent on toast, but time and interaction between time and toast, had no influence. This changes are important to be known by the winemaker in order to produce high quality wines.

Key words

Fetească regală, oenological parameters, oak chips, barrel, toast, time, ageing

Nowadays, more than 140 *Vitis vinifera* and interspecific grape varieties are cultivated in Romania, but only about 40 wine grape varieties of these are found on surfaces larger than 100 ha (Antoce and Călugăru, 2017). "Fetească regală" is the most important and appreciated grape variety in Romania with a planted surface of 17.47 % from percentages calculated as reported to the total surface of main varieties (Antoce and Călugăru, 2017; Coldea et al., 2014). The variety is cultivated mainly in Romania (Transylvania and Moldavia) and also in Moldova, Slovakia, Hungary and Austria. The wines ranges between table wine and high-quality ones, are dry and fresh and have acidity and specific flavour. Aging wines in oak barrels is an ancient tradition aimed to improve wine quality and complexity and contribute to enhance the sensory characteristics and increased stability. Wine composition is modified due to the compunds extracted from woods (Dumitriu et al., 2017). The most important stage in oak barrel production is toasting, because it influence the chemical composition of oak wood. Qualitative and quantitative compounds extracted from oak barrels depends on several factors (botanical and geographical origin of the wood, toast level, contact time between wine and wood, the reuse of the oak barrels) (Dumitriu et al., 2017; Li et al., 2015). Oak barrel aging is expensive and requires long periods of times. In the last years, the use of small fragments for macerating or aging of toasted wood is common, as it coveys similar tastes and aromas to the wine as those obtained with oak barrel, but faster (the increased surface of the fragments accelerates the extraction of the compounds) and cheaper (Hernandez-Orte et al, 2014; Rubio-Bretón et al., 2018). On the market, oak fragment can be found in a variety of forms from shavings (cubes, beans, dominoes, pencils, blocks), oak powder and staves. Also, oak fragments may be found on market on different toast degree (del Alama Sanza M., 2006). Most studies in the literature address the effect of the interaction between wood type and toasts level on red wines (Fernandez de Simon et al., 2014, Dumitriu et al., 2017, Manolache et al., 2018). There are few research about white wines aged in wood barrels or with chips, most on Chardonnay wines (Guchu et al., 2006; Gutierrez Alfonso, 2002; Spillman et al., 2004; Herrero et al., 2016). Oenological composition helps the winemakers to classify the wine due to the differences among wines from the same wine growing region, the same year harvest, ripped at the same stage, with the same winemaking treatments, but aged in

barrels or with chips with different toast degree and during different periods of time (Dumitriu et al., 2017).

Until now, there is no research on "Fetească regală" aged wines in barrels or with oak chips. The aim of this study is to evaluate oenological parameters and statistical tools to differentiate "Fetească regală" wines aged for 30, 60 and 90 days in oak barrels natur (untoast) and with oak chips with natur and mediu toast degree.

Materials and Methods

In the present study a Romanian grape variety was used "Fetească regală". Generally, this indigenous grape variety is used to obtain dry white wines with a great aging potential (Bora et al., 2014; Moroșanu et al., 2018).

"Fetească regală" white grapes (*V. vinifera*) from Teaca (Lechinta wine growing region - center of Romania), were harvest at their optimal ripening stage on 2017 harvest. Stalked and crushed bunches were subjected to fermentation process at 12-14°C for 18 days. At the end of the alcoholic fermentation, the wine was transferred to the barrels and to damijones. The barrels with medium toasting degree were supplied by Transilvania Bois (Maramures, Romania). The barrels were made from *Querqus robur* grown in Arad county forests (East of Romania). The oak chips (*Querqus robur*) with two different toast level (natur and medium toast) were supplied by Sodinal (Romania). Two wood barrel of medium toast (225 l) and two damijones (10 l) for each oak chips type (4 g/l) were used.

The wines were aged during 30, 60 and 90 days. At the end of each period, wine sample from each variant were collect from oenological analysis.

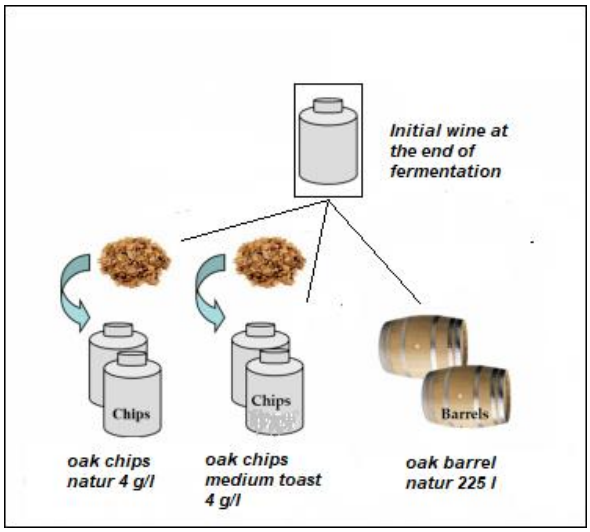

Fig. 1. Experimental design

The oenological analysis of wine were performed in the Winemaking Laboratory of the Faculty of Food Science and Technology Cluj Napoca and were applied in accordance to the methods of analysis described in the Compendium of International Methods of Analysis of wines and musts (O.I.V., 2016). During this analysis parameters were determined: alcohol (% vol.). was determined using the ebulliometric method. The volatile acidity (g/l acetic acid), the total acidity (g/l tartric acid), the total dry extract and non-reducing dry extract (g/l), the free and total SO_2 (mg/l) were determined according to Mureșan, 2008; Bora et al., 2015. The pH level was determined using WTW inoLab pH 7110.

Statistical analysis

In order to determinate the factors with a statistically significant effect on the oenological parameters and to evaluate the significance of interactions between factors, was made a multifactorial variance analysis using as factors the toasting degree and the aging time. The statistical processing of the results was primarily made to calculate the following statistical parameters: arithmetic average and standard deviation and then the statistical interpretation of the results were performed using the DUNCAN test, using the SPSS, version 24 (SPPS Inc. Chicago, IL, USA).

Results and Discussions

One of the important factors determining the quality of the wine is the physico-chemical composition of the grapes, which in turn is influenced by a number of agrobiological and climatic factors. For the production of white wines, the harvesting conditions and the degree of maturation of the grapes are of particular importance (Furtună, 2015).

The basic physic-chemical parameters of the analysed grapes are presented in Table 1 and it may be noticed that the raw material was harvested at technological maturity.

Table 1

Basic physico-chemical parameters of the 'Fetească regală' grapes and must

Grape variety	Must sugar content (g/l)	Must acidity (g/l sulphuric acid)	Gluco-acidic index	Potential alcoholic strength (%vol. alc.)
Fetească regală	210	5.85	20.41	12

The alcoholic strength has varied from a maximum of 12.62 % in the control sample and a minimum of 12.49 % for the sample aged for 90 days in oak barrel. The alcohol content (ethanol) decreases with the maturation period, with losses of about 0.3-0.5%. This was the result of evaporation loss of ethanol and its diffusion through barrel staves, which is higher than those of water during storage (Manolache et al., 2018; Dimitriu et al., 2017). In the case of dami-jones, part of the alcohol is converted into higher alcohols or other forms of compounds, or due the oxidation of alcohol and its conversion into aldehydes (Rubio-Bretón et al., 2018).

Regarding the volatile acidity the limits of variability were rather small between aged samples, with a minimum of 0.22 g/l acetic acid for the initial wine sample (Table 2). Volatile acidity increased with time, due to the oxidation of ethanol (conversion of the alcohol into aldehydes and then, under the influence of acetic bacteria, in acetic acid) (Moreno and Peinado, 2012). The increase in volatile acidity also indicates the extraction of phenolic and volatile carboxylic acids from wood (Canas, 2017). In addition, in barrel aged wines a high volatile acidity was detected compared with chips aged wines in dami-jones, may be due to microbiological factors (level of presence of bacteria).

Total acidity (titratable acidity) is defined as the total substances with acid reaction present in wine, which can be titrated with an alkaline solution in the presence of an indicator. The total acidity for initial wine sample (7.11 g/l tartric acid) decreases slightly as the maturation period increases, a part of the compounds extracted from the oak wood (barrel staves or chips) may combine with organic acids and become volatile compounds, thus influencing the total acidity. Total acidity values obtained are specific vine variety investigated, and also the values of aged samples are in the normal rage.

The total extract represents all non-volatile matter which in certain physical conditions do not volatilize. From the chemical point of view, the total extract is represented by: fixed organic acids (tartaric, malic, succinic acid, lactic acid), glycerol, 2,3-butylene glycol, sugars, tannins and dyes, nitrogen, pectin, gums, etc. (Bora et al., 2015). Non-reducing dry extract is the difference between the total extract and the total sugars. Total dry extract and non-reducing dry extract in initial wine sample was 22 g/l, and 20.26 g/l, respectively. As expected, the values of non-reducing dry extracts and total dry extract slightly decreases in aged samples during time.

Comparing the results of free SO_2 content with the legislation, it can be seen that all produced wines have a much lower content than the one required by law. The highest amount of total SO_2 was registered in the initial wine sample (31.50 mg/l). Free SO_2 decreases due to evaporation and conversion to bonded sulfur. SO_2 losses during the maturation process can also be attributed to measurable oxygen permeability through oak barrels.

Table 2

Enological parameters of the overall sample of wines aged for 30, 60 and 90 days with oak chips with two toast degree and aged in oak barrels with light toast degree

Parameter	Initial wine	Oak chips natur			Oak chips medium toast			Oak barrels natur		
		30 days	60 days	90 days	30 days	60 days	90 days	30 days	60 days	90 days
Alcohol (% vol.)	12.62 ±0.03 ^{a}	12.59± 0.03 ^{b}	12.56± 0.04 ^{c}	12.54± 0.03^{cd}	12.58± 0.03 ^{b}	12.55± 0.02 ^{c}	12.53± 0.04 ^{d}	12.55± 0.03 ^{c}	12.53± 0.02^{d}	12.49± 0.03^{e}
Volatile acidity (g/l acetic acid)	0.22± 0.01^{e}	0.31± 0.01^{d}	0.32± 0.01^{c}	0.32± 0.02^{c}	0.31± 0.01^{d}	0.32± 0.10 ^{c}	0.33± 0.02^{c}	0.31± 0.01^{d}	0.36± 0.01^{b}	0.40± 0.01^{a}
Total acidity (g/l tartric acid)	7.11± 0.10 ^{a}	6.91± 0.09^{b}	6.82± 0.07^{c}	6.71± 0.03^{d}	6.98± 0.12^{b}	6.86± 0.10^{c}	6.74± 0.05^{d}	6.97± 0.12^{b}	6.86± 0.10^{c}	6.74± 0.05^{d}
Total dry extract (g/l)	22.00± 0.20^{a}	21.89± 0.35^{ab}	21.82± 0.45^{ab}	21.67± 0.57^{c}	21.94± 0.37^{a}	22.0±0. 60^{a}	21.78± 0.67^{b}	21.94± 0.37 ^{a}	22.00± 0.60^{a}	21.78± 0.67^{b}
Non-reducing dry extract (g/l)	20.26± 0.05^{a}	20.23± 0.05^{b}	20.20± 0.04^{cd}	20.18± 0.02^{d}	20.21± 0.04^{c}	20.19± 0.03 ^{cd}	20.17± 0.03^{d}	20.21± 0.04^{c}	20.19± 0.03 ^{cd}	20.17± 0.03^{d}
Free SO_2 (mg/l)	31.50± 0.50^{a}	30.33± 0.57^{b}	28.67± 0.57 ^{b}	27.00± 1.00^{c}	29.33± 1.52 ^{b}	27.67± 1.52 ^{c}	26.00± 2.00^{d}	29.33± 0.57 ^{b}	27.00± 1.00 ^{c}	24.67± 0.57^{e}
Total SO_2 (mg/l)	95.67± 0.57^{c}	97.00± 1.00 ^{b}	97.00± 1.00 ^{b}	97.00± 2.00^{b}	98.67± 1.52^{a}	97.67± 0.57 ^{b}	97.33± 1.25 ^{b}	97.00± 1.00 ^{b}	97.00± 1.00 ^{b}	97.00± 2.00 ^{b}
pH	3.22± 0.010^{c}	3.23± 0.005 ^{b}	3.24± 0.005^{a}	3.24± 0.005^{a}	3.22± 0.010 ^{c}	3.23± 0.005^{b}	3.24± 0.010 ^{a}	3.22± 0.010 ^{c}	3.23± 0.005^{b}	3.24± 0.010 ^{a}

Average values, ± standard deviation (n=3).

*The difference between two values in the same row, followed by a common letter is insignificant (Duncan test p<0.5)

The total SO_2 represents the assemble of organic forms resulted by combining SO_2 with aldehydes, ketones, acids, sugars, uronic acids, oxidation products of sugars, phenolic compounds and other substances (Bora et al., 2015). In initial wine sample, the content of total SO_2 was 95.67 mg/l.

Table 3

Multiple analysis of variance (MANOVA) taking as factor aging time and toast level and interaction between the factors aging time and toast level

Parameter	**MANOVA**		
	Time	**Toast**	**Time X Toast**
Alcohol (% vol.)	*	***	ns
Volatile acidity (g/l acetic acid)	***	***	***
Total acidity (g/l tartric acid)	ns	***	ns
Total dry extract (g/l)	ns	ns	ns
Non-reducing dry extract (g/l)	ns	**	ns
Free SO_2 (mg/l)	*	***	ns
Total SO_2 (mg/l)	ns	*	ns
pH	ns	***	ns
*: p < 0.05; **: p < 0.01; ***: p < 0.001; ns: not significant			

In all variants, total SO_2 increases and has small changes with the aging time due to combining to different compounds released by barrel or chips. The pH increases very easily with the increase of the maturation period, it is directly proportional due to the slight decrease in total acidity. pH, ethanol and

titratable acidity have a direct influence on the ethanolization of wood components extracts (Ortega-Heras et al., 2010).

In order to determine whether major wine quality parameters has been influenced by the ageing time and toast degree, multiple analysis of variance was performed to determine which of the analysed factors had greater influence.

Alcohol content and volatile acidity were enological parameters most influenced by the ageing time and toast factors. Although, the interaction between time and ageing had insignificant influence on alcohol. Volatile acidity was significantly dependent on time, ageing and interaction between the two factros. Total acidity, non reducing dry extract, free and total SO_2 and pH were singnificantly dependent on toast, but the factors time and interaction between time and toast, had no influence. The dry extract was the only parameter which was not influence by any treatment.

Conclusions

In regard to the overall results, it can be concluded that oak chips and barrel treatment is an alternative technique for white wines to enhance the oenological parameters. The alcohol content (ethanol) decreases with the maturation period, for all ageing variants. Volatile acidity increased with time, in addition, in barrel aged wines a high volatile acidity was detected compared with chips aged wines in dami-johnes, may be due to microbiological factors. The total acidity, the values of non-reducing dry extracts and total dry extract slightly decreases in aged samples during time. Alcohol content and volatile acidity were enological parameters most influenced by the ageing time and toast factors. The other oenological parameters were singnificantly dependent on toast, but time and interaction between time and toast, had no influence. The use of chips results in a 'quickness' of ageing and involves much lower costs compared to barrel ageing, as well as the exclusion of costly works such as cleaning/washing of barrels that require labor.

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