AROMA PROFILE BASED ON ODOR ACTIVITY VALUES AND AROMATIC SERIES ANALYSIS IN RED WINES SUBJECTED TO DIFFERENT AGEING TYPES BY AMERICAN AND FRENCH OAK CHIPS

PROFILUL AROMATIC BAZAT PE VALORILE ACTIVITĂȚII ODORANTE ȘI PE SERIILOR AROMATICE ÎN VINURILE ROȘII SUPUSE DIFERITELOR TIPURI DE MATURARE CU CHIPS-URI DIN STEJAR AMERICAN ȘI FRANCEZ

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Abstract. Use of oak wood during the process of wine ageing is an ancient and common practice in most of the world's wine producing regions. In this study, the contribution of a chemical compound to the aroma of a wine was evaluated by determining the odour activity value (OAV). OAV, was calculated as the ratio between the concentration of an individual compound and the perception threshold reported in the literature. The analytical aroma profile was established by using the OAVs of each odorant compounds exhibiting similar odor descriptor grouped in an aroma series. Major aroma contributors in the Fetească neagră wine aged with American and French oak chips were the fruity, chemistry, fatty and floral series followed by the woody series. Red wines analyzed at 1.5 and 3 months present similar behaviour, however wines aged with 5 g/L of French oak chips distinguished from the rest when using PCA.

Rezumat. Utilizarea lemnului de stejar în procesul de maturare a vinului este o practică veche și comună în majoritatea regiunilor producătoare de vin din lume. Contribuția unui compus chimic la aroma unui vin a fost evaluată prin determinarea valorii activității odorante (VAO). Aceasta din urmă, VAO, a fost calculată prin raportul dintre concentrația unui compus individual și pragul de percepție raportat în literatura de specialitate. Profilul de aromă analitică a fost stabilit prin utilizarea VAO a fiecărui compus odorant care prezintă un descriptor olfactiv similar grupat într-o serie aromatică. Principalele arome care contribuie la vinul de Fetească neagră, maturat cu chips-uri din stejar american și francez, au fost seriile fructe, chimic, onctuos și floral, urmată de lemnos. Vinurile roșii analizate la 1,5 și 3 luni prezintă un comportament similar, dar vinurile maturate cu 5 g/L chips-uri din stejar francez se disting de restul cu ajutorul PCA.

Cuvinte cheie: serii aromatice, VAOs, chips-uri din stejar american și francez, vin roșu

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INTRODUCTION

Red wine produced from *Fetească neagră* (*V. vinifera*) is a popular alcoholic drink consumed in Romania known to have potential health benefits related to its phytochemical composition.

Ageing is a fundamental technological process that bring to a harmonious development of aromatic, gustatory and chromatic attributes of wine (García-Carpintero *et al.*, 2012), thus reflecting the global quality of wine. Therefore, during wine ageing, groups of subtle reaction occur, which tend to improve the taste and flavor of wine over time. The composition of wine is complex and changes continuously during ageing. Volatile compounds related to aroma have a significant impact on the quality of wine and, hence, for consumer acceptance. Various chemical classes of compounds are responsible for the aroma wines, such as, esters and terpenes are well-known to confer to floral or floral characters (Capone *et al.*, 2013), alcohols and aldehydes own green leafy aroma characters (Kaluaa and Boss, 2009); methoxypyrazines are strongly linked to green capsicum descriptors. Meanwhile, C13-norisoprenoids generally contribute too many flavours (Peinado *et al.*, 2004) in fruits and wines, such as berry, tobacco, honey, balsamic and violet aromas. Ageing can modify these compounds and give wines their distinct fragrances.

The use of oak pieces aims to accelerate the chemical reaction rates (esterification, polymerization, condensation and oxidation, spontaneous clarification), which take place within wine (Gómez Gallego *et al.*, 2015). Oak chips uses in ageing have been reported to improve the sensory attributes of young wine (Dumitriu *et al.*, 2016).

Part of the aroma in wines, is acquired from the wood during the ageing process. The role of oak wood is fundamental since it releases important compounds that have great influence on the final wine characteristics, reducing the astringency, and improving important flavor characteristics, taste, color, phenolics and aroma (Rodríguez and Gómez-Plaza, 2011). The number and quantity of components released by the wood during the ageing process will depend on the species of wood, and the individual oak chips (including seasoning, manufacture, toasting, dosage and contact time), very important factors that will be affect the quality of red wines. Volatile compounds from oak wood require a powerful separation technique for their determination, being gas chromatography, preferably coupled to mass spectrometry (GC–MS) the main technique used to obtain chemical information (Lubes and Goodarzi, 2017). The extraction step previous to the chromatographic analysis is the most difficult task of the analytical methodology.

Wine aroma is the result of the volatile compounds that constitute it. Not every volatile contribute with the same intensity to aroma. The concentrationodour threshold ratio, well-known as the "odour activity value" (OAV), must be considered as the only norm to estimate the contribution of each compound to aroma, although interactions (antagonistic and additive effects) among different

aroma components occur in the matrix (Genovese *et al.*, 2013). Because an individual compound generally has several flavours, it is difficult to establish or evaluate global aroma profiles only using the odour activity values (OAVs) of volatiles. The organoleptic profiles of red wines are comprised by arranging the OAVs of the aroma compounds with identical descriptors into aromatic series. This activity reports quantitative information obtained by chemical analysis to sensory perceptions. Thus, simply and effectively examines and compares the aroma characters.

Nonetheless, there is lack of information about the effect of oak chips during ageing on the aromatic profile of wine. Hence, further in-depth researches have to be conducted to understand the mechanisms that impact on the organoleptic features of the aged wine. Therefore, the aim of this study was to assess the impact of oak chips as accelerating ageing techniques on the aromatic profile of a red wine.

MATERIAL AND METHOD

Fetească neagră variety grapes (*V. vinifera*) were grown in North-East Romania winemaking region and harvested in 2013. The maceration–fermentation process was made at 10-12 °C for 7 days. Afterwards, the grape skins were pressed to extract the remaining juice. The press wine was blended with the free run wine and the mixture was pumped off into stainless steel tanks to complete alcoholic and malolactic fermentations. The wine obtained was divided in 8 batches with 5 L each one and placed in independent glass vessels. The batches were aged with different types of oak wood chips (American and French) and different dosage (3 and 5 g/L) for 1.5 months and 3 months. The dimensions chips in centimetres was $0.5 \times 1.5 \times 0.2$ (width x length x thickness).

The contribution of a chemical compound to the aroma of a wine was evaluated by determining the odour activity value (OAV). OAV is a measure of the importance of a specific compound to the odour of a sample. The odour activity value (OAV) was calculated as the ratio between the concentration of an individual compound and the perception threshold reported in the literature (Francis & Newton, 2005). Furthermore, the analytical aroma profile was established by using the OAVs of each odorant compounds exhibiting similar odour descriptor grouped in an aroma series.

Statistical data analyses were performed using Statgraphics Centurion XVI of StatPoint Technologies Inc. (Warrenton, Virginia). Principal component analysis (PCA) was performed using R package "ggbiplot".

RESULTS AND DISCUSSIONS

Volatile compounds are essential for wine high quality, determining their aroma and varietal properties. Aroma compounds are present in wines, and flavour intensity depends on both concentration and threshold (Wu *et al.*, 2016). Anyway, just a restricted number of volatiles can be found at concentrations high enough to be perceived (OAV \geq 1) and considered as flavour contributors as well as active odorants (Genovese *et al.*, 2013).

In order to evaluate the overall aroma of the wine, the aroma compounds were grouped into different aromatic series according to their odour descriptor and each compound was assigned to one or more aromatic series based on their similar odour character. The series used in this work place contain compounds in groups with similar odour descriptors and these represent the main constituents for the aroma of *Fetească neagră* wines, namely chemistry, fruity, fatty, buttery, floral, green, citric fruit, toasty, spice and woody odours. The total intensities for each aromatic series were calculated as the sum of the OAVs for each of the compounds assigned to a given series. The results are represented in Fig. 1. One of the most evident differences between 1.5 and 3 months were the increase in all series, especially in the fruity and chemistry series, due to the high quantity of esters formed in the alcoholic fermentation. Other differences were that in general French oak chips present the highest series in comparison with series of American oak chips.

The red wines aged with American oak chips of Am3-3M (> 400) and French oak chips of Fr5-3M (> 650) showed relatively high levels of aromatic series, which suggested that these lastly oak types provided more powerful aroma than the other type. Meanwhile, the American oak chips of Am3-1.5M (< 180) and the French oak chip of Fr3-1.5M (< 210) displayed weak aroma.

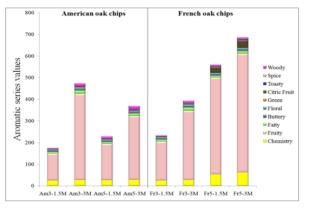


Fig. 1. Aromatic series values for American and French oak chips of aged wines. These results are shown as the mean values.

The chemistry series showed a higher intensity in wines aged with American oak chips (> 27) from Am5-3M and French oak chips (> 60) from Fr5-3M; the fruity series showed a higher level in American oak chips (> 350) from Am3-3M and French oak chips (> 500) from Fr5-3M; fatty aroma was rich (> 15) in American oak chips from Am3-3M and (> 14) in French oak chips from Fr3-3M.

Regarding the buttery series, the maximum value was found in Am3-3M (5.44) and Fr5-3M (5.08). In wines aged with American oak chips (Am5-3M) showed the highest floral flavour (7.33), followed by French oak chips (Fr5-3M) (7.54). The wines aged with French oak chips presented the maximum values for citric fruit series

in Fr5-3M (> 30) and American oak chips showed values much lower (< 0.7), which suggested that these types of flavour cannot be perceived by humans. The other series such as green, toasty and spice presented slight flavour (< 4.4). In addition, the woody series presented a higher intensity in wines aged with American oak chips (> 14) from Am5-3M and French oak chips (> 9) from Fr3-3M.

Principal component analysis (PCA) was used to evaluate the profiles of aromatic series were useful to discriminate and group the different types of wine selected in this research (Fig. 2). The first principal component (PC1) accounted for 53.8% of the total variation, while PC2 explained a further 29.4%. Plots of the PCA revealed that the 10 aromatic series were scattered in quadrants I and IV, showing their positive correlations with PC1.

The wines aged with American oak chips (Am3-3M and Am5-3M) and French oak chips (Fr3-3M) for 3 months were located on the positive side of PC1 and the negative side of PC2. Then, the wines aged with American oak chips (Am3-1.5M and Am5-1.5M) and French oak chips (Fr3-1.5M) for 1.5 months were located on the negative region of PC1 and PC2 (fig.2). The other wines aged with French oak chips (Fr5-1.5M) for 1.5 months were positioned on the positive side of PC2 and negative side of PC1 and the same type of wine aged for 3 months were located on the positive side of PC1 and PC2.

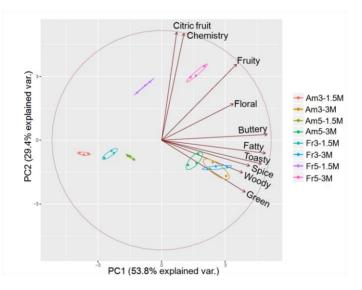


Fig. 2 Principal components analysis using as classifying variables the compounds analyzed in *Fetească neagră* wines

Wines produced with Am3-3M, Am5-3M and Fr3-3M were markedly different from the Fr5-3M samples and were characterized by concentrations of woody, spice, toasty aromatic series. In contrast, a wine aged with Fr5-3M was described with fruity, chemistry and citric fruit aromatic series.

CONCLUSIONS

1. These results confirm the importance of geographical origin of oak wood in the aromatic series of wines during ageing. Each wood transmits some aromatic components to wine in different quantities according to its characteristics and that provokes differences in OAVs and aromatic series of aged wines. Therefore, wines with different characteristics were obtained from the same wine, after 1.5 and 3 months of ageing with oak chips. The application of alternative techniques resulted in an increase in odour activity values at 3 months compared to the 1.5 months of ageing.

2. PC1 differentiated wines aged 1.5 months by other aged 3 months with both type of oak chips. PC2 separated French oak chips with 5g/L by all other sample. Also, French oak chips with 3g/L have a similar profile with American oak chips and cannot be differentiated.

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