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INFLUENCE OF THE AGING OF APPLE WINES WITH OAK CHIPS ON THE QUALITY AND SENSORY PARAMETERS OF THE WINES

– Research paper –

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Abstract: This study investigated the effect of the oak chips added at different dosages and stages of apple wine production on its oenological, antioxidant and sensory properties. In the study, medium-toasted or heavily-toasted oak chips (at a concentration of 3 or 6 g/L) and ethanol extracts prepared from them (at a dose of 1 or 2 mL/100 mL) were used. Basic oenological parameters (OIV), total polyphenol content (Folin-Ciocalteu method), antioxidant activity (ABTS assay), color (CIELAB) and organoleptic assessment of the wine were evaluated. Although the use of chips had no significant effect on the ethanol content and wine extract, it caused a significant change in volatile acidity. Furthermore, the antioxidant activity of the wines correlated closely with the concentration of polyphenolic compounds and these parameters were significantly higher after contact with medium-toasted chips. The apple wines had high values of lightness and yellow color, and the addition of chips did not significantly affect the color parameters (CIELAB). In the sensory assessment, the control wine (without chips) received the lowest scores, while significantly higher scores were given to the wine with the addition of 6 g/L of heavily-toasted chips, which was characterized by intense smoky, toasty, woody and vanilla notes.

Key words: oak chips, maceration, aging (maturation), apple wine

INTRODUCTION

The domestic apple tree *(Malus domestica* Borkh.*)* is one of the most widely cultivated fruit species in the world. The chemical composition of the fruit is one of the factors affecting the quality of apple wines. The main chemical compounds that make up the aroma profile of apples are carboxylic esters, alcohols, terpenes, aldehydes, ketones, ethers and free acids, such as ethyl acetate (which gives a pleasant peachy aroma), ethyl butyrate (apple aroma) and methyl anthranilate (grape aroma), eugenol (clove aroma), geraniol (rose aroma), limonene (citrus), linalool (rosewood aroma) and myrcene (apricot, walnut and orange aroma) (Dixon and Hewett, 2000) (El Hadi et al., 2013).

Aroma is one of the most important qualitative characteristics of wine and, along with color and flavor, is the primary consumer perception. The use of wood in the fermentation and/or aging (maturation) process of wine is a common practice in most of the wine beverage producing regions of the world (Martinez-Gil et al., 2022). The aging process varies depending on the type of wine. For red wines, there are two stages of aging: oxidative, during contact with wood, and reductive, after the wine is bottled (Oberholster et al., 2015) (Carpena et al., 2020). White wines can also be aged in wood, but for many years it was thought that this process masked the fresh ingredients of the beverage and thus compromised its quality.

Today, the use of wood in the production and aging of white wines is becoming more common, especially for wines with more complex aromas (Carpena et al., 2020) (Ortega-Heras et al., 2007) (González-Centeno et al., 2020). White wines that were aged in barrels made of *Quercus alba* wood received higher average ratings than those that were aged in barrels made of other types of wood (Jordão and Cosme, 2022). During the wine aging, changes in the sensory characteristics occur and aromas associated with wood become noticeable, such as

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vanilla, coconut, almond and smoke (Dumitriu et al., 2019) (Dumitriu et al., 2020). Lu et al. (2024) showed that wines aged in oak barrels exhibited more toasty, leathery, and smoky notes, but the aromas of fruit, flowers, and caramel were less pronounced. Non-volatile and volatile compounds are distinguished among the substances extracted from the oak. The former includes mainly ellagitannins, which influence, among other things, the sensation of astringency and terpenoids, which are perceived as sweet and aromatic. Volatile compounds include, but are not limited to, lactones, furanones, volatile phenols, volatile aldehydes, esters, isoprenoids, pyranones, and acetic acid (Casass et al., 2021). The content of guaiacol, syringol, trans-isoeugenol, furfural alcohol, vanilla, cis-whisky lactone allowed the distinction between medium toasted and light toasted barrels (Lu et al. 2024).

Despite its many advantages, the aging of wine in barrels has several disadvantages. These vessels are expensive to produce, have a limited shelf life, and the probability of growth of microorganisms that cause wine spoilage increases with each batch. In addition, aging wine in barrels requires a long period of time, usually 6 to 18 months, during which the volume of the wine is constantly reduced as a result of evaporation through the pores, absorption by the wood and losses through the gaps between the staves (Carpena et al., 2020). The use of oak chips~~,~~ instead of barrels~~,~~ is one of the alternative methods of wine aging. This method typically involves the use of small pieces of wood or wood chips that are dipped into the wine to impart the characteristics of barrel aging to the wine (Rubio-Bretón et al., 2018). The use of oak chips is a less costly, time-saving and more sustainable method that still produces products of comparable quality to those obtained by aging in barrels, thanks to a better transfer and incorporation of volatiles with woody notes (Tao et al., 2014). Wines aged with American oak chips showed a greater intensity of coconut and vanilla notes and an increase in the degree of astringency and bitterness than wines aged in barrels (Jordão and Cosme, 2022). Chardonnay wine from Romania aged for a short time (3 months) in barrels and with oak chips showed a similar quality. However, it was significantly cheaper. In addition, in white wines, the use of oak chips helps to prevent the oxidation of volatile aromatic compounds that occurs during barrel aging and gives the wines a hint of oak without compromising their fresh and fruity characteristics (Stegarus et al., 2021).

The aim of the study was to evaluate the effect of the addition of oak chips at different stages of apple wine production on the oenological, antioxidant and sensory properties of the beverages. Oak chips with different degrees of toasting were added during fermentation or wine aging and the ethanol extracts of these oak chips were also used. The basic oenological parameters of the wines and their antioxidant properties and total polyphenol content were assessed. In addition, color analysis and sensory evaluation of wines aged with oak chips were performed.

MATERIALS AND METHODS

Material

The Elisa apple – the main research material – came from the pomological orchard of the University of Agriculture in Krakow (Poland).

Dry yeast Oenoferm Tipico F3 (Erbslöh Geisenheim GmbH, Germany), intended for white wines, was used for ethanol fermentation. Before inoculation, the yeast was rehydrated according to the protocol provided by the manufacturer.

Medium-toasted or heavily-toasted oak chips (provided by Malinowy Nos, Poland) or an ethanol extract prepared from them were used in the research. The ethanol extract was prepared by adding 5 g of appropriate chips to 50 mL of 70% ethyl alcohol and mixing (magnetic stirrer) the whole for 3 h at room temperature.

Diammonium salt of the 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic) acid (ABTS diammonium salt), (±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox), a phosphate buffer saline (PBS: 0.01 M phosphate buffer, 0.0027 M potassium chloride, 0.137 M sodium chloride; pH 7.4 at a temperature of 25°C) were purchased from SIGMA-Aldrich (Germany). The chemicals: sodium carbonate (Na_2CO_3), sodium pyrosulfite ($Na_2S_2O_5$), potassium persulfate ($K_2S_2O_8$) and ethanol (analytical grade) were purchased from POCh (Gliwice, Poland).

Must preparation

Washed and crushed fruits (laboratory crusher) were mixed with a pectinolytic preparation (Pectinex® YieldMASH, Novozymes) at a ratio of 80 μL per kg of pulp and left for 24 h at 20°C. The must was then pressed out of the fruit by a hydro-press (Zottel, Slovenia). The extract content in the must was adjusted to 20°Blg with sucrose and the acidity was adjusted to 4.5 g/L with acid. The must

prepared in this way was sulfurized at a dose of 50 mg SO_2/L.

Fermentation and wine aging

0.5 L of apple must was poured into each of 15 fermentation bottles made of green glass. At the same time, a fermentation balloon was prepared, in which 10 liters of must were placed. Before the start of the fermentation stage, medium- or heavily-toasted oak chips were added to the fermentation bottles at a concentration of 3 g/L or 6 g/L, while the must in the balloon was fermented without the addition of wood chips. Yeast was rehydrated according to the manufacturer's instructions and added to all samples at a dose of 0.3 g/L. Fermentation was carried out at 20°C (± 2°C) for 28 days.

Samples in bottles were weighed during fermentation to determine fermentation kinetics. Weight was measured on average every 2 days until alcoholic fermentation was complete (defined as a difference in sample weight of less than 0.01 g). After fermentation, the wines fermented in the presence of wood chips were decanted and aged in glass bottles (at 5°C) for 3 months.

Half of the wine from the fermentation balloon was decanted from the sediment, poured into glass bottles (0.5 L each) and aged with the addition of medium- or heavily-toasted oak chips, in doses of 3 g/L or 6 g/L. The rest of the wines from the balloon were poured into glass bottles (0.5 L each), and ethanol extract of medium or heavily-toasted oak chips was added in the amount of 1 or 2 mL/100 mL of wine. All the wines were aged at 5°C for 3 months.

The control wine was apple must inoculated with yeast as described above and fermented and aged in the bottles under at the same conditions as other samples, but without wood chips addition.

All tests and analyses were performed in at least three physical replicates.

Determination of alcohol content, extract, total acidity and volatile acidity

Alcohol and extract concentration, total acidity, and volatile acidity were determined according to the OIV methodology (OIV, 2014). The wine samples were subjected to straight distillation. The oscillating densimeter (DMA 4500 M, Anton Paar, Austria) was used to determine the alcohol and extract content of the distillates and distillation residues, replenished to original volume. Total acidity was measured by potentiometric method (pH-meter, CP-505, Elmetron, Poland) and results were expressed in g malic acid/L. Volatile acidity was determined by the titration method (pH-meter, CP-505, Elmetron, Poland) after previous steam distillation, and expressed in g acetic acid/L.

Assessment of antioxidant activity

The antioxidant activity (AOX) was assessed by the ABTS method (as described by Tarko et al. 2009). ABTS radical was generated by chemical reaction between 7 mM aqueous solution of diammonium salt of 2,2'-azynobis (3-ethylbenzthiazoline-6-sulfonate) and 2.45 mM potassium persulfate solution ($K_2S_2O_8$). The solution was kept overnight in the dark at room temperature to complete the reaction and to stabilize the ABTS cation. During the analysis, the concentrated ABTS solution was diluted with phosphate buffer saline (PBS) at pH 7.4 to obtain a solution with an absorbance value of A = 0.70 ± 0.02 ($ABTS_{0.7}$), measured with a spectrophotometer (Beckman DU 650, USA) at a wavelength of 734 nm. 100 µL of the appropriate diluted samples were added to 1 mL of $ABTS_{0.7}$ and the absorbance was measured at 6 minutes after mixing. The antioxidant capacity of the samples was calculated using a standard curve generated with solutions of synthetic vitamin E (Trolox; 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid, Sigma) and expressed in mg of Trolox/100 mL.

Total polyphenol content (TPC)

The total polyphenol content (TPC) was determined by the Folin-Ciocalteu method (Tarko et al. 2009). 45 mL of redistilled water was added to 5 mL of diluted wines. Then, 0.25 mL of Folin-Ciocalteu reagent (water dissolved at 1:1 v/v, Sigma) and 0.5 mL of 7% Na_2CO_3 (POCh) were added. The samples were incubated in the dark for 30 minutes before measuring the absorbance with a spectrophotometer (Beckman DU 650, USA) at the 760 nm (against PBS as a blank). The results of the total polyphenols content were obtained on the basis of the calibration curve and were expressed as mg of (+)-catechin per 100 mL.

Designation wine colors L*a*b* (CIELAB)

Color was assessed by tristimulus colorimetry on the basis of transmittance spectra using the equations proposed by the International Commission of Claristics (CIE 1986). The wine was centrifuged (15 min, 5000 rpm, MPW 350R centrifuge) and the absorption spectrum was measured (380-770 nm, CM-5 spectrophotometer, Konica Minolta). The CIELAB parameters were calculated: *L** (perceptual lightness; in the range from 0 – black to 100 – white), and chromatic coordinates: *a** and *b**. The a* axis is relative to the green–red opponent colors, with negative values toward green (–100) and positive values toward red

(+100), while the b* axis represents the blue–yellow opponents, with negative numbers toward blue (–100) and positive toward yellow (+100). Based on these coordinates, other color parameters were determined: h_{ab} (hue) and C^*_{ab} (chroma, also called color saturation or intensity), which were calculated using the formulas (1) and (2) (CIE 1986):

$$h_{ab} = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (1)$$

$$C^*_{ab} = [(a^*)^2 + (b^*)^2]^{\frac{1}{2}} \quad (2)$$

in which: h_{ab} – hue; C^*_{ab} – chroma; a^*, b^* – color coordinates.

To distinguish the color more accurately, the color difference was also calculated using the formula (3) (Spagna et al., 1996):

$$\Delta E = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{\frac{1}{2}} \quad (3)$$

Organoleptic assessment of wine

The basic sensory evaluation of the wines was carried out by a panel of 15 sensory experts using a 5-point scale (1 = bad to 5 = very good) with weighting factors (indicated in brackets). The following attributes were evaluated: color (0.1), clarity (0.1), aroma (0.2) and taste (0.6). In addition, the perceptibility of aromas (fruity, herbal, bitter, toasty, woody, smoke, chocolate, seasoning, caramel, coconut, vanilla, foreign) and tastes (sweet, sour, bitter, astringent, fruity, herbal, toasty, woody, smoke, chocolate, seasoning, caramel, coconut, vanilla, foreign) were also assessed.

Statistical analysis

InStat v. 3.01 software (GraphPad Software Inc., San Diego, USA) was used for statistical analysis. One-way analysis of variance (ANOVA) with Tukey's post hoc test was performed. The Kolmogorov-Smirnov test was used to assess the normality of the distribution. Each experiment was performed in at least three replicates and results are presented as the arithmetic mean with standard deviation (± SD).

RESULTS

The presence of wood chips did not have a significant effect on the course of the alcoholic fermentation, and the different phases of the process were the same for the control sample and for the samples with the addition of various oak chips. The initial fermentation of the settings lasted three days, and then a turbulent fermentation took place (7 days), associated with intensive release of carbon dioxide and weight loss of the samples. Then began the silent fermentation phase, during which the yeast slowly consumed the unused sugars in the must, which lasted until the 28th day of the experiment. The total weight change for each of the variants was 44 g, which was about 4% of the initial weight of the setting.

Effect of oak chips addition on oenological and antioxidant properties of apple wines

One of the main end products of sugar metabolism by the yeast *Saccharomyces cerevisiae* is ethyl alcohol. The final ethanol content in wine depends, among other things, on the sugar concentration, the fermentation temperature and the yeast strain used. The content of ethyl alcohol in the control wine was 10.93% (v/v), and the wines fermented or aged with oak chips ranged from 10.81% (v/v) to 10.99% (v/v), and the differences were not statistically significant (Table 1). A statistically significant difference in the alcohol content was observed only in wines with the addition of alcoholic extracts from oak chips, in which the ethanol content was 3.4 –to 9.7% higher than in the control sample, depending on the dose of extract and the degree of chips toasting. However, the increased strength of the wines resulted from the physical addition of ethyl alcohol (with extracts). The content of total extract in all the wine samples ranged from 1.95 to 2.2 °Blg, but the differences, regardless of the addition of oak chips, were not statistically significant.

The must obtained from apples of the Elisa variety had a total acidity of 3.48 g/L, and after undergoing fermentation and aging, the control wine had a total acidity of 5.94 g of mallic acid per liter (Table 1). The addition of oak chips during the fermentation stage resulted in a significant decrease in total acidity, which ranged between 5.58 and 5.67 g/L (Table 1), depending on the dose and toasting degree of oak chips. It is important to note that the mere addition of chips to the must during fermentation caused a statistically significant reduction in acidity, regardless of their dose or the degree of their toasting.

A completely opposite effect was obtained when the wood chips or the extract were added at the stage of wine maturation. The greatest differences were observed when wines were aged with the addition of alcoholic extracts from oak chips; an increase of the total acidity up to 7.24 g/L was reported in wines with 2 mL of extract from heavily-toasted wood chips. However, it should be emphasized that the dose of the extract was not statistically significant in relation to this parameter (Table 1). On the other

hand, the degree of roasting exerted a significant impact; extracts from heavily-toasted chips resulted in a greater increase of total acidity than extracts from medium-toasted chips. When the wine aging was conducted in the presence of oak chips, only heavily-toasted chips contributed to the increase in total acidity. Thus, the degree of chips toasting was a parameter with the greatest impact on the total acidity of the wine, when added during the maturation stage.

The addition of oak chips or extracts from chips to the wines increased the volatile acidity by approx. 25% (from 0.08 g/L in control wine to 0.10 – 0.11 g/L in all types of wines). The low volatile acidity (about 0.1 g/L) of the obtained wines proves their good quality and excludes the occurrence of a defect, a vinegar spoilage, which is caused by the wine infection with acetic fermentation bacteria of the genus *Acetobacter (A. aceti, A. pasteurianus)* and is most often ~~is~~ manifested by an increase in ~~of~~ the described oenological parameter.

The addition of oak chips used in this study contributed to the increase in the antioxidant activity of the wines. A lower degree of toasting was more favorable for AOX (Table 1), and the only exception were the wines aged in the presence of heavily-toasted oak chips or their alcoholic extract, used at lower doses, they did not cause significant differences. Considering the addition of chips extract added at the stage of maturation, it can be seen that the wines with the addition of 2 mL of medium-toasted wood chip extract were characterized by the highest antioxidant potential (40.47 ± 0.44 mg Trolox/100 mL), but this value was not statistically different from a lower dose of the same extract. The use of chips instead of the extract resulted in an even greater increase in the antioxidant activity (up to ~56 mg/100 mL), with the obtained values depending more on the dose of chips and their degree of toasting than on the stage at which they were added.

Table 1. The effect of the addition of oak chips (medium- or heavily-toasted) or alcoholic extract of them on the oenological and antioxidant characteristics of apple wines

The degree of wood chips toasting	Dose of chips or extract	Ethanol content [%]	Extract content [°Blg]	Total acidity [g of mallic acid/L]	Volatile acidity [g of acetic acid/L]	Antioxidant activity [mg of Trolox /100 mL]	Total polyphenol content [mg of (+)-catechin / 100 mL]
Control	–	10.93±0.10 c	2.19±0.03 a	5.94±0.28 c	0.08± 0.00 a	32.76±1.90 f	8.01±0.43 f
wines with addition of chips during fermentation							
Medium	3 g /L	10.93±0.17 c	2.16± 0.08 a	5.58±0.17 d	0.11±0.02 b	46.09±2.83 d	11.89±0.41 d
	6 g /L	10.91±0.14 c	2.15± 0.06 a	5.61±0.14 d	0.11±0.01 b	56.00±2.21 e	16.82±0.62 e
Heavily	3 g /L	10.81±0.13 c	2.20± 0.02 a	5.63±0.13 d	0.11±0.01 b	38.21±1.23 g	8.30±0.25 b,f
	6 g /L	10.99±0.27 c	2.19± 0.02 a	5.67±0.27 d	0.11±0.01 b	41.12±1.02 b	10.04±0.45 c
wines with addition of chips during aging							
Medium	3 g /L	10.81±0.19 c	2.11± 0.05 a	5.87±0.10 c	0.11±0.01 b	44.26±0.45 d	11.28±0.56 d
	6 g /L	10.93±0.08 c	2.19±0.01 a	5.92±0.10 c	0.11±0.01 b	56.13±0.99 e	15.24±2.27 e
Heavily	3 g /L	10.87±0.04 c	2.14± 0.01 a	7.15±0.04 a	0.10±0.00 b	34.47±1.39 a,c,f	8.58±0.36 b,f
	6 g /L	10.87±0.02 c	2.13±0.01 a	7.17±0.00 a	0.10±0.01 b	36.52±0.17 c,g	9.90±0.54 c
wines with addition of alcoholic extract from wood chips during aging							
Medium	1 mL /100 mL	11.34±0.11 a	2.11±0.03 a	6.81±0.04 b	0.10±0.01 b	38.86±0.64 b,c,g	8.52±0.70 b,f
	2 mL /100 mL	11.99±0.02 b	2.11±0.03 a	6.95±0.10 b	0.10±0.10 b	40.47±0.44 b,g	8.90±0.63 b
Heavily	1 mL /100 mL	11.66± 0.30 a	1.95± 0.20 a	7.17±0.20 a	0.10±0.02 b	33.63±1.10 a,f	7.61±0.10 a,f
	2 mL /100 mL	11.87±0.16 b	2.06±0.07 a	7.24±0.07 a	0.10±0.00 b	35.53±1.31 a,c	8.10±0.32 a,b,f

The same letters within one column indicate no statistical significance, for $p < 0.05$.

The highest values of TPC reached 15.24 and 16.82 mg of catechin per 100 mL in wines with addition of medium-toasted oak chips at the stage of maturation and fermentation, respectively (Table 1). These values were almost twice as high as those obtained in the control wines.

Effect of oak chips addition on color parameters of apple wines

The color of wines is determined by properties such as hue, saturation (chroma) and lightness. These parameters depend mainly on the qualitative and quantitative concentration of anthocyanins. The influence of the addition of oak wood chips and their alcoholic extracts on the color parameters of wines are presented in Table 2.

The *L** value determines the lightness of the sample (pigment concentration). It can range from 0 (pure black) to 100 (pure white, the maximum lightness). In all the wines (except for the wine with 6 g/L of medium-toasted chips added at the fermentation stage) a very high lightness (*L** above 90 CIELAB units) was noted, caused by the low concentration of anthocyanins in the apples (Table 2). Wines with the addition of oak chips were darker (lower L*), but the reduction of the *L** value was slight and rather insignificant. The only significant changes in wine lightness compared to the control sample were reported for wine with medium-toasted oak chips added at the stage of maturation (however, the dose of chips at had no significant effect) and for wine with medium-toasted chips added at a dose of 6 g/L during the fermentation stage. The addition of medium-toasted oak chips in the three samples mentioned caused a reduction in the color lightness (L*), but could also be reflected in a lower clarity of such a wine.

Table 2. Effect of the addition of oak wood chips (medium- or heavily-toasted) or their alcoholic extracts on the color parameters of wines (CIELAB)

The degree of wood chips toasting	Dose of chips or extract	***L****	***a****	***b****	h_{ab}	C^*_{ab}	***ΔE***
Control	–	92.39 ± 0.11 a	-0.24 ± 0.01 a,c	6.56 ± 0.05 a	92.12 ± 0.04 a	6.56 ± 0.05 a	-
wines with addition of chips during fermentations							
Medium	3 g /L	91.64 ± 0.24 a,b	-0.21 ± 0.05 a,c	7.77 ± 0.26 f	91.55 ± 0.40 a,c	7.77 ± 0.26 f	1.42
	6 g /L	89.76 ± 1.40 d	-0.02 ± 0.16	9.63 ± 0.78 e	90.15 ± 0.94 d	9.63 ± 0.78 e	4.05
Heavily	3 g /L	91.89 ± 0.25 a,b	-0.13 ± 0.03 b,c,d,f	7.40 ± 0.14 b,f	91.01 ± 0.23 b,c,d	7.40 ± 0.14 b,f	0.98
	6 g /L	91.83 ± 0.03 a,b	-0.14 ± 0.05 b,c,d,e	7.89 ± 0.07 d,f	91.04 ± 0.34 b,c	7.89 ± 0.07 d,f	1.45
wines with addition of chips during aging							
Medium	3 g /L	91.26 ± 0.15 b,c	-0.24 ± 0.03 a,c	8.42 ± 0.28 d	91.61 ± 0.26 a,c	8.43 ± 0.28 d	2.18
	6 g /L	90.58 ± 0.27 c	-0.28 ± 0.05 a	9.90 ± 0.15 e	91.60 ± 0.28 a,c	9.90 ± 0.14 e	3.80
Heavily	3 g /L	91.94 ± 0.12 a,b	-0.12 ± 0.08 b,c,f	7.51 ± 0.07 b,f	90.94 ± 0.62 b,c,d	7.51 ± 0.07 b,f	1.06
	6 g /L	91.69 ± 0.23 a,b	-0.20 ± 0.08 a,c	8.35 ± 0.42 d	91.39 ± 0.48 a,b	8.36 ± 0.42 d	1.92
wines with addition of alcoholic extract from wood chips during aging							
Medium	1 mL /100 mL	92.36 ± 0.05 a	-0.25 ± 0.10 a,d	6.67 ± 0.19 a,c	92.16 ± 0.87 a	6.67 ± 0.19 a,c	0.11
	2 mL /100 mL	91.93 ± 0.10 a,b	-0.25 ± 0.06 a,e	7.20 ± 0.10 b,c	92.01 ± 0.49 a	7.20 ± 0.10 b,c	0.79
Heavily	1 mL /100 mL	91.88 ± 0.35 a,b	-0.08 ± 0.03 b,f	7.38 ± 0.26 b,f	90.60 ± 0.27 b,d	7.38 ± 0.26 b,f	0.98
	2 mL /100 mL	91.82 ± 0.36 a,b	-0.22 ± 0.02 a,c	7.63 ± 0.34 b,f	91.65 ± 0.17 a,c	7.64 ± 0.33 b,f	1.21

The same letters within one column indicate no statistical significance, for $p < 0.05$.

Legend: L* – sample lightness from 0 to 100; a* - describes the color from green (-100) to red (+100); b* - describes the color from blue (-100) to yellow (+100); h_{ab} – hue; C^*_{ab} – chroma.

When concerning a* and b* values, the changes were more profound. As expected for white wines, the a* values were close to zero (although all had negative values) reflecting the almost complete absence of red color and a very slight tendency towards green color, especially in ~~for~~ the wines aged in contact with heavily-toasted chips or extracts added at the maturation stage. For the b* values, the addition of wood chips, both at fermentation and maturation stage (regardless of their degree of toasting), induced a statistically significant increase in yellow color. The hue angle (h_{ab}) can range from 0° to 90° (red to yellow), 90° to 180° (yellow to green), 180° to 270° (green to blue), and 270° to 0° (blue to red). The wines obtained had a hue angle between 90.15° and 92.16°, which corresponds to yellow colors with a slight tendency towards green. It's interesting that the addition of medium-toasted oak chips or their extract caused a reduction of the hue angle (enhanced the yellowish colors). As expected, the addition of oak chips significantly influenced the color saturation (C^*_{ab}). When oak chips were added, an increase in the degree of chip toasting resulted in a decrease in the color saturation, while in the case of the addition of chip~~s~~ extracts, the highest effect was observed in the case of extracts prepared from heavily-toasted chips.

Despite the differences described above, the color difference (ΔE) between the control wine (without chips) and the wines prepared in contact with oak wood chips or chip extracts showed that only a few wines showed values higher than two CIELAB units, indicating that the color difference could be detected by the human eye~~s~~ (Spagna et al., 1996). The highest ΔE values (4.05 and 3.80 CIELAB units) were reported for wines with oak medium-toasted chips added at a dose of 6 g/L at the fermentation and maturation stage, respectively. There were wines for which the color was also the most saturated (C^*_{ab} = 9.63 and 9.90, respectively).

Organoleptic assessment of wine

Organoleptic evaluation is one of the stages that allows to check the acceptability acceptance of the newly produced product by potential customers. It provides feedback on which variant of the product is most popular and has the highest sales potential.

Table 3 presents the results of the organoleptic evaluation of apple wines prepared with or without the addition of oak chips.

The highest score (4.3 points) was given to wine aged with 6 g/L of heavily-toasted oak chips. Lower scores were given to wines aged with heavily-toasted oak chip extract (3.7 to 3.9 points), wines aged with heavily-toasted oak chips at 3 g/L (3.8 points), and wines fermented with heavily-toasted oak chips at 6 g/L (3.7 points). However, in all these cases, the results were not statistically significant with the highest-rated wine (Table 3). The other wines with the addition of wood chips and the control wine were rated in the range of 3.4 to 3.6 points, and the differences between these samples were not statistically significant.

Table 3. The influence of the addition of oak chips (medium- or heavily-toasted) or their alcoholic extract on the basic organoleptic evaluation of wines

The degree of wood chips toasting	Dose of chips or extract	Organoleptic evaluation [points]
Control	–	3.4 ± 0.4 [a]
wines with addition of chips during fermentation		
Medium	3 g /L	3.6 ± 0.6 [a]
	6 g /L	3.5 ± 0.4 [a]
Heavily	3 g /L	3.5 ± 0.6 [a]
	6 g /L	3.7 ± 0.7 [a,b]
wines with addition of chips during aging		
Medium	3 g /L	3.4 ± 0.6 [a]
	6 g /L	3.6 ± 0.6 [a]
Heavily	3 g /L	3.8 ± 0.5 [a,b]
	6 g /L	4.3 ± 0.5 [b]
wines with addition of alcoholic extract from wood chips during aging		
Medium	1 mL /100 mL	3.5 ± 0.5 [a]
	2 mL /100 mL	3.6 ± 0.5 [a]
Heavily	1 mL /100 mL	3.9 ± 0.6 [a,b]
	2 mL /100 mL	3.7 ± 0.5 [a,b]

The same letters within column indicate no statistical significance, for $p < 0.05$.

Additionally, the sensory profiles for aroma and taste parameters of the wines obtained were made (Figures 1 to 6). Coconut and foreign flavors were not detected in any of the wines analyzed. The apple wines tested were characterized by intensely perceptible fruity, astringent and sour flavors (Figures 1 – 3). To a lesser extent, the panelists noted bitter and sweet taste.

The use of oak chips significantly altered the perception of some apple wine flavors. Toasty, woody, smokey, chocolate, caramel, and vanilla flavors were not evident in the control samples, but these flavors were identified in wines with the addition of wood chips. Sweet flavor was more noticeable in wines fermented and aged in the presence of wood chips, but the type and dose of chips had no statistically significant effect. Only in the case of the addition of the extract, the use of heavily-toasted wood chips increased the perception of sweet taste compared to controls and samples with the extract from medium-toasted wood chips. The sour taste sensation was intensified after the addition of wood chip extract. In most cases, significant changes in the perception of selected flavors were observed during the maturation of wines with wood chips, especially herbal, chocolate, woody and astringent flavors. Fermentation and the addition of extracts were less important. Bitterness was significantly more pronounced in wines aged with a higher dose of medium-toasted wood chips and extract from these chips (Figures 1 – 3). Typical wood aromas (woody, smoky, seasoning, caramel, vanilla) were detected mainly in wines aged with medium-toasted wood chips. Fermentation with these chips (Figure 1) and the addition of extract (Figure 3) slightly increased the described aromas of the wines.

The wines were also evaluated for the detectability of selected aromas (Figures 4 – 6). As in the case of flavors, most of the aroma analyzed were not detected in the control sample. The exceptions were fruity and bitter aromas, which were more pronounced in the control sample. The addition of oak chips during fermentation and maturation reduced the perception of fruity and bitter aromas. They were probably dominated by aromas typical of oak - woody, smoky and toasty. The aroma of spices did not change significantly compared to the control, during fermentation and maturation of the wines in the presence of wood chips, but the addition of extracts reduced the perception of this aroma (Figure 6). Most of the aromas detected were appropriate to the dosage of wood chips - larger amounts resulted in better perceptibility of wood-specific aromas. The degree of burning of the wood chips had no significant effect on the perceptibility of the aromas analyzed, except for the smoky aroma. In this case, the addition of heavily-toasted wood chips significantly increased the perception of smoky aroma in the samples fermented and aged in their presence, compared to the control and the addition of medium-toasted wood chips (Figures 4 and 5).

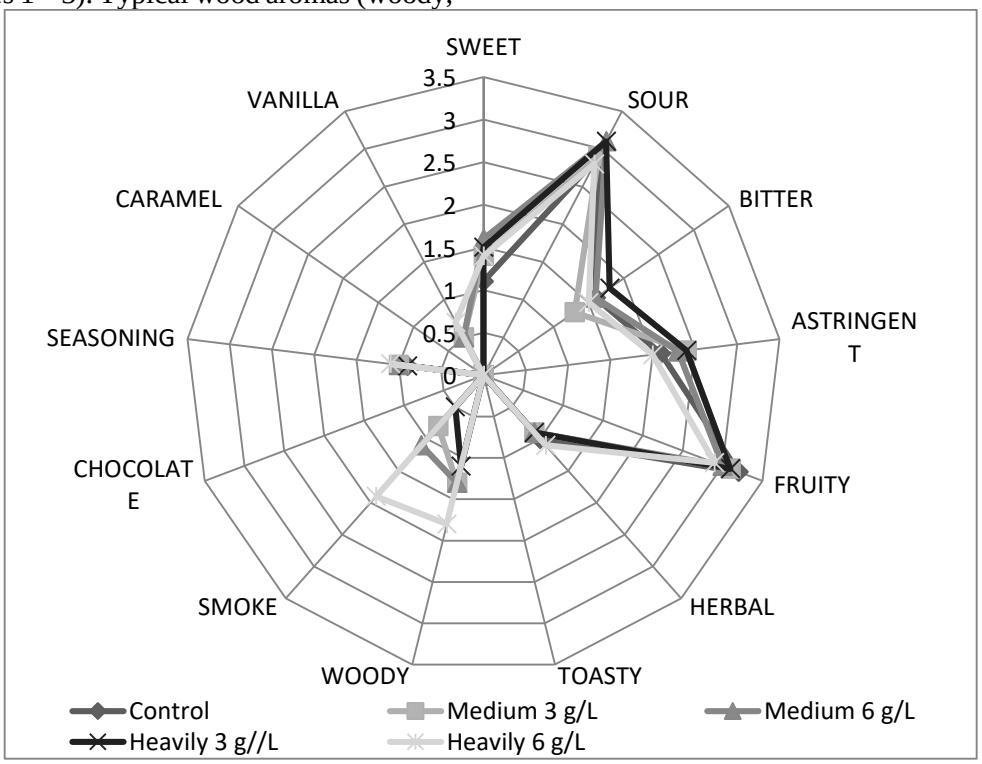


Figure 1. Effect of the addition of oak chips (medium- or heavily-toasted) during fermentation on the perceptibility of selected taste sensations in wines.

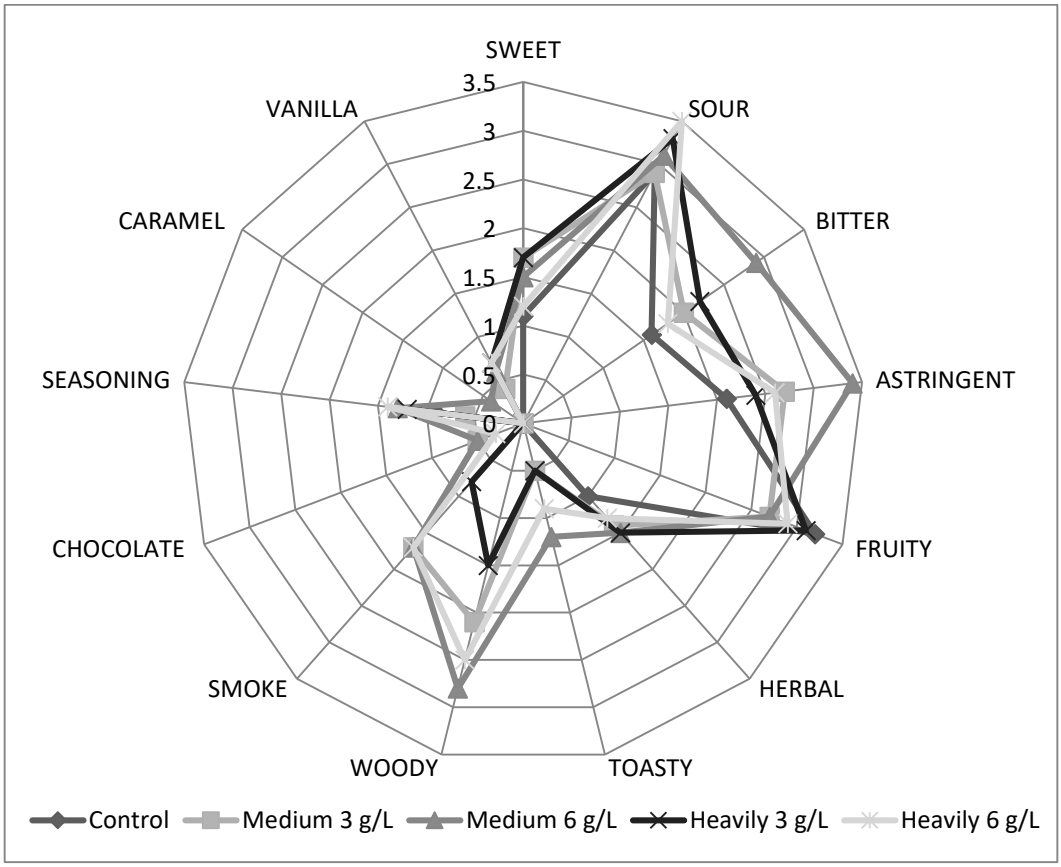


Figure 2. Effect of the addition of oak chips (medium- or heavily-toasted) during aging on the perceptibility of selected taste sensations in wines.

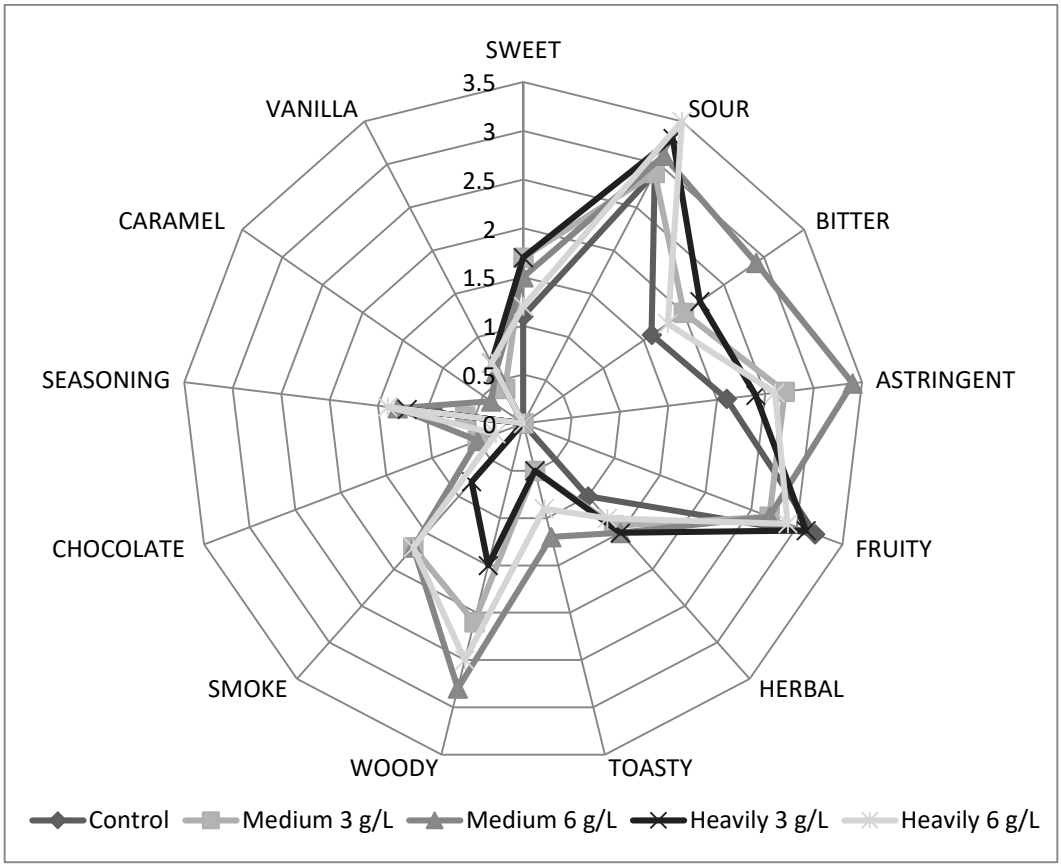


Figure 3. Effect of the addition of alcoholic extracts made from wood chips (medium- or heavily-toasted) during aging on the perceptibility of selected taste sensations in wines.

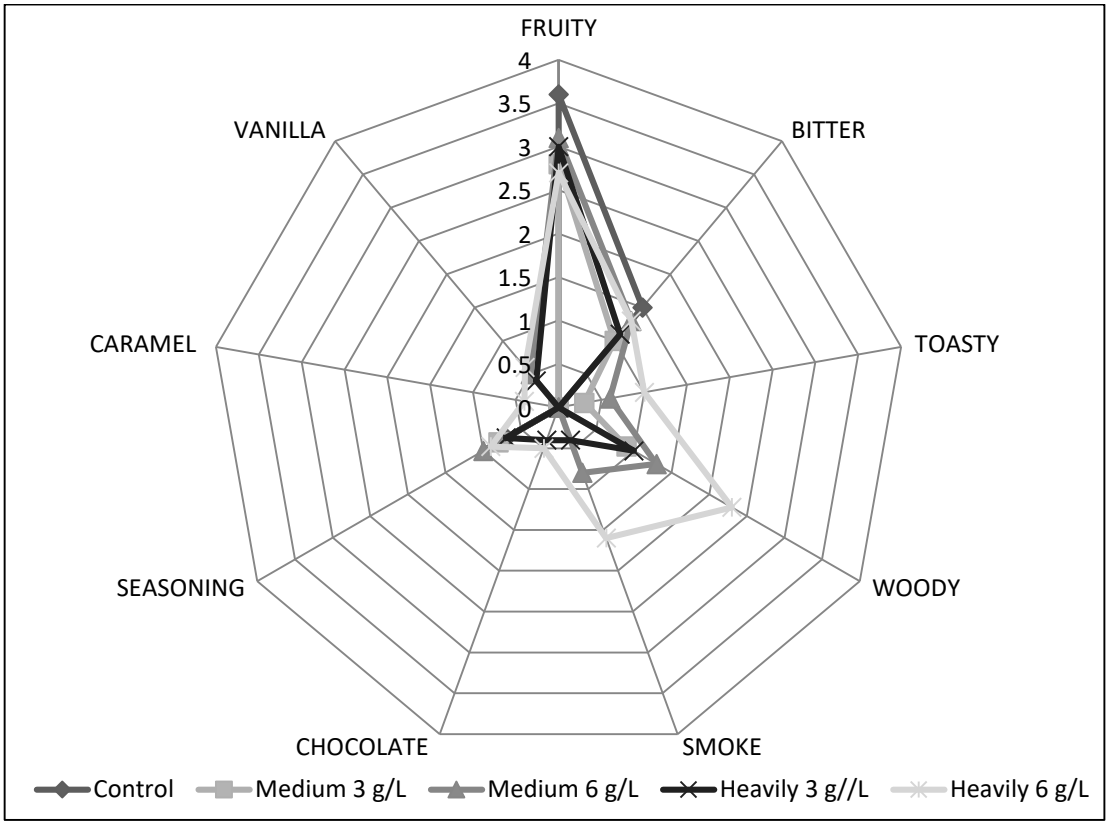


Figure 4. Effect of the addition of oak chips (medium- or heavily-toasted) during fermentation on the perceptibility of selected aromas of wines.

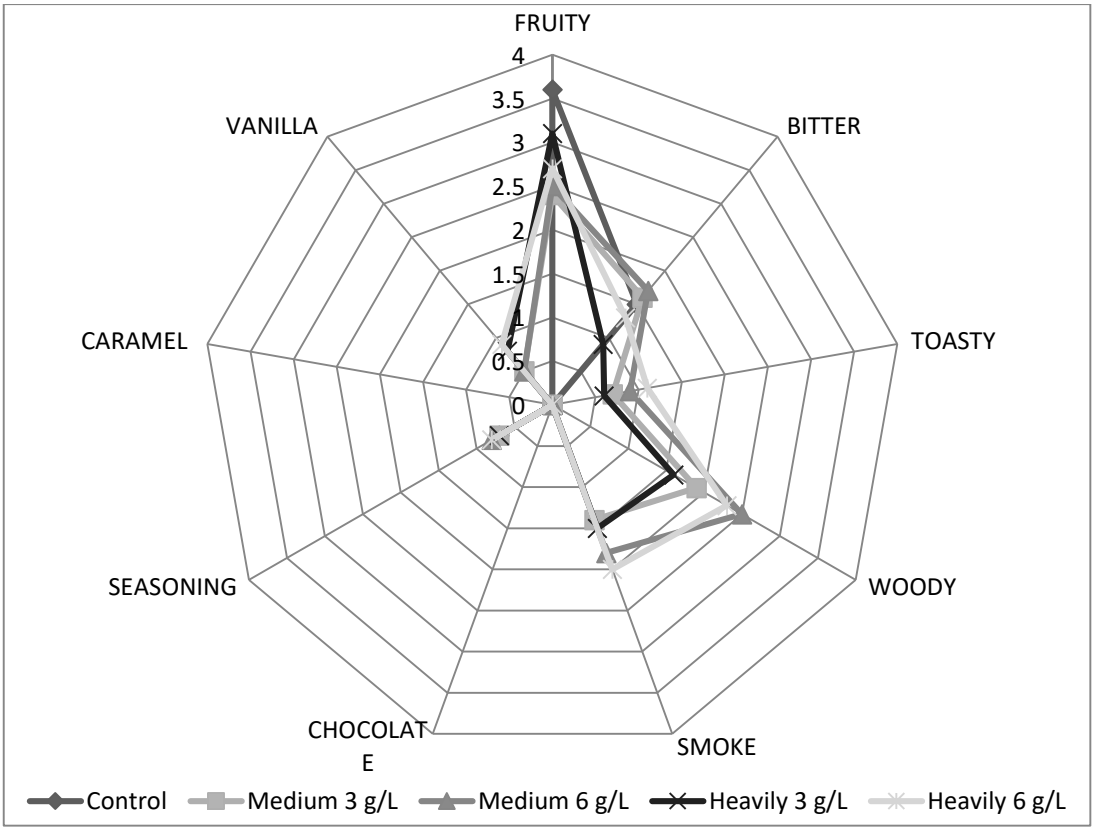


Figure 5. Effect of the addition of oak chips (medium- or heavily-toasted) during aging on the perceptibility of selected aromas of wines.

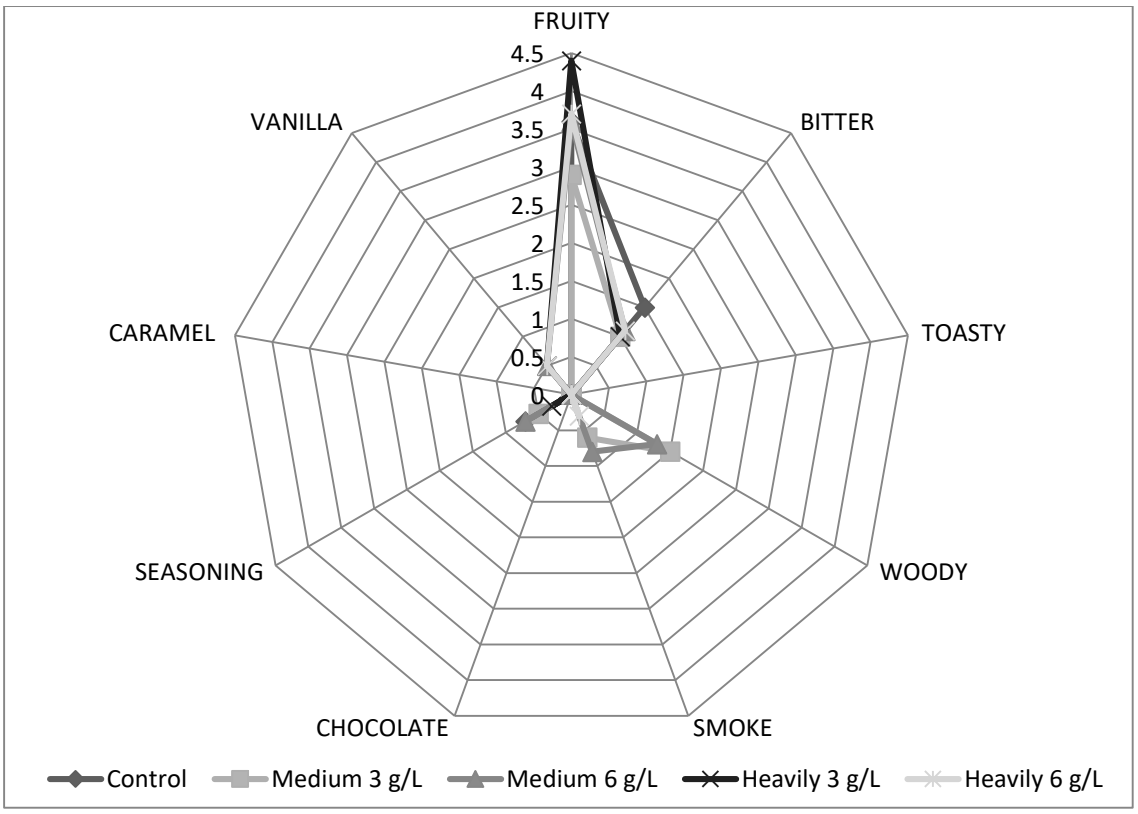


Figure 6. Effect of the addition of alcoholic extracts made from wood chips (medium- or heavily-toasted) during aging on the perceptibility of selected aromas in wines.

DISCUSSION

Our results showed that the addition of wood chips did not affect the course of the fermentation process and the kinetics of alcoholic fermentation of wines were similar to those observed in studies conducted by other researchers (Kemsawasd et al., 2015). The differences obtained between the ethyl alcohol and total extract content of the control wine (without any additives) and the wines fermented or aged with oak chips were statistically insignificant. The only exception in our research was the wine with the addition of alcoholic extracts from oak chips, in which the ethanol content was statistically higher than in the control sample and depended on the dose of extract and the degree of burning of the chips, but the increase in strength in the case of these wines resulted from the physical addition of ethyl alcohol along with the alcoholic extracts. These results are consistent with those conducted by others scientists. Dumitriu et al. (2020), Martinez-Gil et al. (2018), Alencar et al. (2019, and Nunes et al. (2020) also showed no significant effect of the addition of oak chips on the strength of the wine, regardless of the type of chips and their dose. In turn, Nunes et al. (2020) and Casassa et al. (2021) reported that neither the addition of oak chips to wines nor wine maturation in oak barrels significantly affected the content of total extract, confirming the results obtained in this study.

In our study, the must was obtained from Elisa apples. The total acidity of the control wine increased from 3.48 g/L (in the must before fermentation) to 5.94 g/L (at the end of the experiment). The addition of oak chips resulted in a significant reduction in total acidity, but only when added during the fermentation phase. In contrary, wines aged with oak chips presence, particularly those with the addition of alcoholic extracts, showed a significant increase in total acidity. The degree of burning of the chips, rather than the extract dose, was found to be the determining factor for the increase in total acidity. In studies conducted with musts from other apple varieties, such as: Idared, Shampion and Golden Delicious, the values obtained for the total acidity were similar to those obtained in the present study, and they were: 3.73 g/L; 3.39 g/L and 3.28 g/L, respectively (Januszek et al., 2020). Dumitriu et al. (2019) found a decrease in total acidity of red wines aged with oak chips; however, the type and amount of oak chips used, as well as the duration of the aging process, can influence this result. The decrease in acidity may be due to a reduction in tartaric acid content, likely caused by the precipitation of tartaric salts, and the

duration of the wine aging process has the greatest impact on this parameter (Dumitriu et al., 2020) (Casass et al., 2021) (Călugăr et al., 2020). Other studies (Nunes et al., 2020) (Baiano et al., 2016) have found no impact of the dose and type of oak chips added at the maturation stage on the total acidity of wines. This is consistent with the data obtained in this study for wines matured in the presence of medium-toasted oak chips. Any observed differences may be due to varying degrees of chips toasting, defined by a particular manufacturer as medium or heavy.

The increase in volatile acidity observed in the present study may be due to the hydrolysis of hemicellulose acetyl groups, as well as the oxidation of some wine phenols. In addition, the extraction of phenols and volatile carboxylic acids from wood may increase the volatile acid content of the final beverage (Canas, 2017). The results of other studies also showed a significant impact of the addition of oak chips during the maturation stage on wine quality. The increase in the volatile acidity of wines reported in this study was greater than that obtained in our study and amounted to about 0.2 – 0.3 g/L (Dumitriu et al., 2016).

The polyphenolic compounds of young wines are mainly phenolic acids, flavonols, flavanols, tannins and anthocyanins. Their profile and concentration depend, among other, on the fruit variety and ripeness, soil and climatic conditions, as well as the fermentation process and aging methods (Dumitriu et al., 2016) (Morel-Salmi et al., 2006) (Viviani et al., 2007) (Ivanova et al., 2012). During wine aging, the quantitative and qualitative composition is modified as a result of the copigmentation, condensation and polymerization processes. For wines, which are aged with oak chips, the degradation of lignin from oak wood is also important (Canas et al., 2019) (Gomez Garcia-Carpintero et al., 2012). In addition, the phenolic composition of the liquor is affected by the species of wood, the degree of toasting, the alcohol content in the wine, and the aging time, as well as the surface to volume ratio (Koussissi et al., 2009) (Ortega et al., 2010). The experiments carried out showed that the addition of oak chips at the fermentation stage significantly increased the antioxidant activity of the wines obtained, with medium-toasted chips having a stronger effect. On the other hand, wines aged with the addition of heavily-toasted oak chips or their extract did not differ significantly from the control. However, when medium degree of chips toasting was used, both the chips themselves and their extract added at the aging stage significantly increased the antioxidant potential. The antioxidant activity was closely correlated with the content of polyphenols (TPC) in the analyzed wines (R^2 = 0.93). It is known, that the antioxidant potential depends not only on the concentration of polyphenolic compounds, but also on their chemical structure (Dumitriu et al., 2016) (Satue-Gracia et al., 1997) (Dumitriu et al., 2015). As in the case of the antioxidant activity, the moment of addition of oak chips to the wine (fermentation or maturation) was not proved to have a significant impact on the content of polyphenols, whereas, both the dose of oak chips and the degree of roasting significantly influenced the TPC level. The polyphenols content increased proportionally to the dose, but inversely proportional to the degree of toasting. Similar results were received by Dumitriu et al. (2016), the authors revealed that the total antioxidant capacity of wines aged with oak chips was significantly higher in control wines (without oak chips) and depended on their degree of toasting and dose, with wines aged with medium toasting being characterized by the highest antioxidant capacity. Li et al. (2020) showed that Longyan white wines aged with the use of 6 g/L of oak chips had the highest total antioxidant capacity (47.85 U/ml), which was three times higher than that of the control samples. It is believed that high molecular weight polyphenolic compounds present in wine, such as ellagitannins extracted from wood, are responsible for the antioxidant activity. Eight ellagitannins have been identified in traditional oak species: castalagin, vescalagin, grandinin, and roburin (A, B, C, D, and E), with the two most abundant compounds being vescalagin and castalagin stereoisomers (Puech et al., 1996) (Vivas and Glories, 1996) (Martínez-Gil et al., 2020). The reason why wines with the addition of heavily-toasted oak chips showed lower antioxidant potential than wines with medium-toasted oak chips may be that the release of ellagitannins from the chips decreases with enhancing the toasting degree (Casass et al., 2021) (Navarro et al., 2016). Dumitriu et al. (2020) also proved that the content of phenolic compounds in wine and the antioxidant activity depend on the degree of wood toasting. Intensive wood toasting reduces the synthesis of phenols and caused the formation of volatile phenols with a smoky and spicy character.

The addition of oak wood chips and their alcoholic extracts influenced some of the color parameters of wines, mainly the values of b* and C^*_{ab}. All the wines had a very high lightness (L^* above 90 CIELAB units), which is due to the low concentration of anthocyanins in the apples. Wines with the addition of oak chips were slightly darker, but the reduction of the L^* value was rather

insignificant. The a* values were close to zero (which was expected for white wines made from apples), while for the b* values, the addition of wood chips both at fermentation and maturation stage induced a statistically significant increase in the yellow color, regardless of the degree of chip toasting. Therefore, the color intensity C^*_{ab} was highly positively correlated with the b* value (correlation coefficient $R^2 = 0.9999$).

Interesting results were obtained for the color difference (ΔE). According to the Spagna et al. (1996), the color difference between two samples can be detected by the human eye only when the ΔE values are higher than two CIELAB units. However, according to the newest investigations of Martinez et al. (2001), a value around 3.0 CIELAB units should be considered as a preliminary estimate of the acceptable tolerance of by the human eye for red wines poured in standard wine sampler. In our study, only for a few wines were the color differences from the control wine (ΔE) large enough to be noticeable to the human eye. The highest ΔE values were reported for wines with the most saturated color, i.e. wines with oak medium-toasted chips added at a dose of 6 g/L at the fermentation and maturation stage. Delia et al. (2017) had tested chips prepared from various wood species (cherry, acacia, American oak and French oak) and demonstrated, that only white wines aged in the presence of acacia wood chips showed a ΔE value higher than 2 units. In another study, twenty-one different compositions of oak chips (French, American, and Chinese oak and their mixtures, various toasting levels) were added at a dose of 4 g/L to one-year-old wine made from *Vitis amurensis* and left for further for aging (6 months, 15°C) (Yu et al., 2022). The results showed that the wines darkened (lower L*) after aging, which would be due to their higher phenolic content, and oak chips increased the red hue of the wine. Although the a* value and h* angle value had significant differences, the b* and ΔE did not differ significantly before and after aging the addition of any of the oak chips tested. According to Ju et al. (2021), the color parameters of red wines are significantly correlated with anthocyanins profile and concentration. In the mentioned study, the L* values were negatively correlated with all tested anthocyanins, while a* and C^*_{ab} values were significantly positively correlated with cyanidin, cyanidin-3 glucoside, malvidin, malvidin-3-galactoside and malvidin-1,3-diglucoside, pelargonidin-3,5-diglucoside and delphinidin-3,5-diglucoside. The b* values were positively correlated with delphinidin and peonidin glycosides. Alencar et al. (2019), in their work, found that the color of wines was significantly affected not only by the fact of Syrah wine maturation in the presence of oak chips, but also by the type of oak chips used. Opposite results were obtained by Recamales et al. (2006) and Carvalho et al. (2015), which show the absence of significant differences in color parameters for wines made from white grapes. This was probably due to the different characteristics of red and white wines. Wines from red grape are characterized by different color parameters than white wines, they show a very low L* value, mainly due to the maceration of the grape skins during the production process, which results in a higher concentration of anthocyanins (Dumitriu et al., 2016) (Carvalho et al., 2015).

The results of the organoleptic evaluation of apple wines prepared with or without the addition of oak chips showed that the use of oak chips can be an interesting alternative for wine aging in barrels. Although there was no statistical significance in the scores given by the panel to the wines analyzed, with one exception, none of the wines evaluated scored lower than the control. The highest score (4.3 points) was given to the wine aged with 6 g/L heavily-toasted oak chips, and the result was statistically higher than the score of the control wine (3.4 points). Slightly lower scores (between 3.9 and 3.7) were given to wines aged with heavily-toasted oak chip extract, wines aged with 3 g/L heavily-toasted oak chips, and wines fermented with 6 g/L heavily-toasted oak chips. It means that heavily-toasted chips had a positive impact on the final score of the wine sensory evaluation.

In the wines evaluated, coconut and foreign flavors were absent, while intense fruity, astringent and sour flavors were perceived. As these were dry wines, this is in line with expectations. Apple wines are characterized as sour, fruity (Ruppert et al., 2021), and the perception of astringency depends primarily on the amount of polyphenols in the fruit (Sommer et al., 2022).

The use of oak chips significantly altered the perception of some apple wine flavors, giving them flavors that were not perceived in the control samples, such as toasty, woody, smoky, chocolate, caramel, and vanilla. In wines fermented and aged in the presence of wood chips, the sweet flavor was more noticeable, regardless of the type and dose of chips. However, when wood chip extract was added, the use of heavily-toasted chips increased the perception of sweetness compared to controls and samples with medium-toasted chips extract.

The sour taste sensation was intensified after the addition of wood chip extract, which may be have been due to the slightly higher strength of the wines (Joshi and Sandhu, 2000) and samples aged with heavily-toasted wood chips. In most cases,

significant changes in the perception of selected flavors were observed during the maturation of wines with wood chips, especially herbal, chocolate, woody and astringent flavors.

Wines aged with a higher dose of medium-toasted wood chips and extract from these chips showed significantly more pronounced bitterness. Typical woody aromas (woody, smoky, spicy, caramel, vanilla) were detected mainly in wines aged with medium-roasted wood chips, while fermentation alone with these chips and the addition of extract only slightly increased the described aromas of the wines. It has been shown (Kyraleou et al., 2015) (Gambuti et al., 2010), that the aging of wines in the presence of oak chips contributes to an increase in the detectability of vanilla flavor and sweetness, which the authors explain by the extraction of compounds from the wood, including vanillin. The astringency of wines aged with wood chips increased with the duration of the experiment, but after three months, the astringency was less noticeable (Kyraleou et al., 2015). According to Baiano et al. (2016), the increased perception of astringency in wines aged in the presence of oak chips may be due to increased extraction of tannins from the wood. The authors also showed that the perception of spicy, woody, and vanilla flavors increased after aging wines with oak chips.

A similar situation occurred with regard to aromas. Wines prepared with oak chips gained new aromatic notes not perceptible in the control wines. The exceptions were fruity and bitter aromas, which were more pronounced and intensely perceptible in the control sample and weaker in the wines with chips. Probably these aromas were dominated by stronger aromas typical of oak - woody, smoky and toasty.

In most of the samples, the perceptibility of the detected wood-specific aromas was proportional to the dose of wood chips, while the degree of wood chip toasting had no significant effect (except for the smoky aroma).

Interestingly, the spice aroma underwent significant changes (weaker perceptibility) only in wines made with the addition of extracts at the maturation stage. A study by Kyraleou et al. (2015) showed that spicy, woody, and toasty aromas increased when wines were aged in the presence of wood chips. Vanilla aroma did not appear until at least 3 months of aging. Other researchers (Baiano et al., 2016) reported that when wines were aged in the presence of oak chips, spicy and vanilla aromas increased, but the perception of fruity notes, which were prominent in control wines (without wood contact) throughout the experiment, decreased. Botha et al. (2020) reported that oak-derived trans-2-nonenal, trans-2-octanal and 1-decanal were responsible for the wood (planky) aroma, and the toasty and roasted coffee aroma was due to furfurylthiol and furan aldehydes formed during toasting.

CONCLUSIONS

In conclusion, it has been demonstrated that fermentation and maturation of apple wines with oak chips and alcoholic extract from oak chips does not significantly affect the ethyl alcohol content and total extract. However, the addition of oak chips and alcoholic extract from the chips significantly increases the volatile acidity of apple wines, and the higher the degree of oak chip toasting, the higher the volatile acidity of the wines. The antioxidant activity of apple wines is closely correlated with the concentration of polyphenolic compounds, with the highest values of both parameters observed in wines fermented and matured with the addition of 6 g/L of medium-toasted oak chips. The addition of oak wood chips and their alcoholic extracts significantly influenced two color parameters of the wines, i.e. the values of b* and C^*_{ab}. Wines with the addition of oak chips were slightly darker, but the reduction of the *L** value was rather insignificant; only wines with the addition of medium-toasted oak chips at a dose of 6 g/L during fermentation and maturation had color differences from the control wine (ΔE) large enough to be noticeable to the human eye. None of the wines analyzed were rated lower by the sensory panel than the control wine, and some of them even received higher scores. The best sensory evaluation was given to the wine with the addition of 6 g/L of medium-toasted oak chips at the maturation stage, which was distinguished by intense toasty, smoky, woody and vanilla notes. This means that the use of oak chips at the aging stage can be an alternative to obtain sensory attractive apple wines with the mouthfeel of oak barrel aging.

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