EFFECT OF PREPARATIONS SUBSTITUTING THE USE OF BARRIQUE BARRELS ON PARAMETERS OF WHITE WINE

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This paper presents the results of a study on the application of toasted oak chippings (also called oak chips) on parameters of white wines (cv. 'Malverina') of different processing procedures. Experimental variants differed in the moment of chip application (i. e. either into must or wine) and in the degree of toasting (medium, high). The following parameters were determined in wine samples: total titratable acidity, contents of glucose and fructose, pH, and concentrations of tartaric, malic, acetic, and citric acid. Spectrometry (DPPH test) was used to determine the antioxidant activity of the wines. In variants with addition of chips into the must, the antioxidant activity was lower by 50 % than in the control samples. Variants with chips added into the wine showed an increase tendency in the antioxidant activity (on average also by 50 %). This experiment also involved a sensory analysis of the wines. The primary and secondary aromas of the control samples were more varietal and floral, whereas in variants with added chips spicy tones and vanillin aromas predominated.
Keywords: analytical parameters, antioxidant activity, 'Malverina', oak chippings, sensory analysis

Wirkung von Barrique-Ersatzpräparaten auf bestimmte Parameter von Weißwein. Diese Arbeit präsentiert Ergebnisse einer Studie über die Anwendung von getoasteten Eichenholz-Chips auf bestimmte Parameter von Weißweinen (cv. 'Malverina') verschiedener Verarbeitungsverfahren. Die Untersuchungsvarianten unterschieden sich hinsichtlich des Zeitpunktes des Chips-Einsatzes (d. h. entweder in Most oder Wein) und des Toasting-Grads (mittel, hoch) der Chips. Die folgenden Parameter wurden in Weinproben bestimmt: titrierbare Säure, Gehalte an Glucose und Fructose, pH-Wert und Konzentrationen von Weinsäure, Äpfelsäure, Essigsäure und Zitronensäure. Die antioxidative Aktivität der Weine wurde spektrometrisch (DPPH-Test) bestimmt. In Varianten mit Chips-Zusatz in den Most war die antioxidative Aktivität um 50 % niedriger als in den Kontrollproben. Varianten mit Chips-Zusatz in den Wein zeigten eine Zunahme in der antioxidativen Aktivität (im Durchschnitt auch um 50 %). Diese Untersuchung beinhaltete auch eine sensorische Analyse der Weine. Die Primär- und Sekundäraromen der Kontrollproben waren sortentypischer und blumiger, während in Varianten mit Chips-Einsatz würzige Töne und Vanillearomen vorherrschten.
Schlagwörter: analytische Parameter, antioxidative Aktivität, 'Malverina', Eichenholz-Chips, sensorische Analyse

At present, barrique wines are very trendy products offered by the wine market. The principle of their production is based on the fact that they are stored and aged in oak barrels that give them a typical (barrique) aroma. If the barrels are burnt ('toasted') inside, the aged wine acquires a specific taste and aroma (Twede, 2005). This aroma resembles the taste of vanilla, toast, coffee or coconuts; it can also be characterised as roasted or smoky (Sauvageot und Feuillat, 1999). Especially red wines of this character are looked for very frequently and customers rate them very positively. Nevertheless, barrique barrels may also show a positive effect on white wines. The application of this wine-making method facilitates the production of full-bodied wines with a vanilla and/or woody aroma (Ribéreau-Gayon et al., 2006; Perez-Magarino et al., 2011). However, toasted oak barrels are usually very expensive. Another disadvantage of these casks is their difficult sanitisation, the complicated control of the fermentation process and their relatively small volume. Due to these facts some preparations were developed that could 'replace' these barrels. These preparations may be added either to the must, the mash or to the produced wines. Toasted oak chippings (also called 'chips') are the most frequently-used substitutes, mainly because they are also accessible to small producers (i. e. the so-called 'hobby winemakers'). An addition of chips into wine also facilitates winemaking in stainless containers with a controlled fermentation process (Moreno-Arribas and Polo, 2009).The vintner is not limited by the container volume and, last but not least, it is also possible to eliminate problems associated with sanitation because these substitutional preparations can be used only once (Cano-Lopez et al., 2008). The aim of this study was to evaluate the effects of the aforementioned substitute preparations on parameters of white wine made from the 'Malverina' variety.

MATERIAL AND METHODS

EXPERIMENTAL DESIGN

This experiment was performed with 'Malverina' grapes produced in the Mendeleum gene-pool vineyard in Lednice na Moravě (alt. 176 m a.s.l). The sugar content of grapes was 21 °NM (i. e. the Czech standardized saccharimeter; °KMW = 0,732 x °NM + 3,2). Harvested grapes were crushed and macerated with skins for a period of 18 hours; afterwards the mash was pressed and the must was left to settle for 24 hours. In order to start the fermentation, a special type of *Saccharomyces cerevisiae* yeast was used (SIHA-CRYAROME®; Eaton, Nettersheim, Germany). Subsequently the following experimental variants in 5l-demijohn bottles were established:

EXPERIMENT A – MUST

Variant I - Medium degree of toasting; Variant II - Heavy degree of toasting; Variant III - Control (i. e. without chips). All variants were established in three replications. Chips were added in doses of 10 g. The fermentation was finished after 19 days and the young wine was racked into 3l-demijohn bottles for sedimentation, racking and preparation of samples for tasting (i. e. for the sensory examination and evaluation of the wines). At this time the samples for the analytical investigation of the wines were also taken.

EXPERIMENT B – WINE

In this experiment, wine made within the framework of Variant III (Control) of Experiment A was used. A clarified wine sample was poured into 5l-demijohns and thereafter the oak chips were added. There were also three variants in this case, viz. Variant I - Medium toasting of chips; Variant II - Heavy toasting of chips; Variant III – Control, no chips added). These variants also were established in three replications and chip samples were also 10 g. Over the course of the following six months, individual samples for the spectrophotometric determination of the antioxidant activity were taken at the following time intervals: one week; one month; two months; four months, and six months. After the final sampling, the wine was bottled before being tasted.

CHARACTERISTICS OF CHIPS USED IN THE EXPERIMENT

Experimental oak chippings (Erbslöh, Geisenheim, Germany) were made of wood from English oak (*Quercus robur*) species imported from France. The diameter of these chippings was less than 2 mm. The oak wood

was originally used for making barrique barrels. Chips were of the following degrees of toasting: OakyVin FM (French oak, medium toasting) and OakyVin FH (French oak, heavy toasting)

ESTIMATION OF ANALYTICAL PARAMETERS

The aforementioned parameters were estimated using the ALPHA apparatus (Bruker, Karlsruhe, Germany). The ALPHA spectrometer is a compact FTIR analyzer based on the principle of ATR sampling. This method of sampling considerably simplifies the preparation of samples for analysis. This means that samples of clarified wine were analysed directly, (i. e. without any adjustments) while those of musts and fermenting wine were centrifuged at 13,400 rpm for a period of six minutes. Prior to the measuring of the first sample, the apparatus was thoroughly rinsed with distilled water and the background was measured using deionized water as a blank sample. For analyses, 1 ml of clear wine was sampled with a syringe; one half of which (0.5 ml) was used for the rinsing of the system while the remaining half was used in the subsequent three measurements. Depending on the method of calibration (musts/fermenting wine/fermented wine), the recorded data were automatically software-evaluated and tabulated.

DETERMINATION OF ANTIOXIDANT ACTIVITY (DPPH)

This procedure was performed according to the method proposed by Sochor et al. (2010) . A 150 µl volume of reagent (0.095 mM 2,2-diphenyl-1-picrylhydrazyl - DPPH·) was incubated with 15 µl of the sample. The absorbance was measured at 505 nm for a period of ten minutes.

SENSORY ANALYSIS

The sensory evaluation was performed by a group of five experts. The following parameters of individual wine samples were evaluated: fruitiness, degree of toasting, wine purity, harmony of smell, and woody tones in the taste. In variants with chips added into wine, the following parameters were evaluated in addition to those mentioned above: varietal character, freshness, tastes of vanilla, caramel, coffee, and hazelnut, spicy aroma and flower aroma (both in the smell and the taste). Samples were always presented in the following sequence: Variant III (Control), Variant I (Medium toasting) and Variant II (Heavy toasting). The evaluation was organised as a blind tasting.

RESULTS AND DISCUSSIONS

EXPERIMENT A – MUSTS

Values of some basic analytical parameters are presented in Table 1. Contents of individual acids in the must before the separation of samples into individual variants is presented in the column with the heading 'must'. There are another three columns in this table, viz. Control (i. e. the variant without chips), Medium (i. e. the variant with the addition of medium toasted chips) and Heavy (i. e. the variant with the addition of heavily toasted chips).
The content of acetic acid after the end the fermentation of 'must variants' is also presented in Table 1. In spite the fact that the differences between mean values were not great, it was possible to see that in variants with the addition of medium toasted chips, there were marked differences in measured values after their addition into experimental musts. However, when using toasted oak chippings, acetic acid usually plays an important role. Ribéreau-Gayon et al. (2006) mentioned that toasting might result in the development of (above all) furan aldehydes, volatile phenols and fatty acids (especially acetic acid). The increasing intensity of toasting reduced their concentrations. The measured values, however, did not corroborate this statement. In 'must variants', the highest concentration of acetic acid was recorded in the control sample; in this case (i. e. in Variant III), no chips were added to the must and the average measured value was 0.45 g/l. The lowest concentration was recorded in Variant II (with medium toasting of chippings, viz. 0.37 g/l). In Variant I (with heavily toasted chippings) the average concentration of acetic acid was 0.43 g/l.
One of the negative aspects of the application of toasted wood was the fact that, due to the toasting, the content of acetic acid in wood had increased (Moreno-Arribas and Polo, 2009). After the toasting, the concentration of acetic acid was increased due to the thermodegradation of lignins to esters of acetic acid. This explains

the fact why the concentration of acetic acid was higher in Variant I (Heavy toasting) than in Variant II (Medium toasting).

Phenolic substances contained in wine show antioxidant properties and participate in the so-called antioxidant activity of wine. It is generally known that the concentration of total polyphenols is directly proportional to the antiradical activity (Sochor et al., 2014). In variants with a direct addition of chips to the must, the highest and the lowest antiradical activities were recorded in Variant III and Variant II (40.46 mg/l and 21.23 mg/l, respectively.). Values of antiradical activity are presented in Figure 1. In this context it is important to remember that the addition of chips is also associated with the supply of a certain quantity of oxygen; in must (and also in wine), this element shows an oxidative effect. Due to the effect of oxygen, a chemical reaction takes place in the course of which phenols are converted and alcohol is transformed to acetaldehyde (and vice versa). However, this method of oxidation is a slow process (Moreno-Arribas and Polo, 2009).

Table 1: Values of some selected analytical parameters

	Must		Control		Medium wine		Heavy wine	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Acetic acid (g/l)	x	x	0,4	0,04	0,4	0,05	0,4	0,05
Alcohol (%)	x	x	11,1	0,74	11,6	0,68	10,7	0,47
Citric acid (g/l)	0,1	0,01	0,1	0,11	0,0	0,00	0,0	0,00
Fructose (g/l)	108,0	0,85	9,5	0,65	6,0	6,89	4,2	0,45
Glucose (g/l)	x	x	0,7	0,38	3,9	6,64	1,2	0,43
Glycerol (g/l)	x	x	6,3	0,66	5,8	0,59	5,8	0,25
Lactic acid (g/l)	x	x	0,1	0,12	0,1	0,10	0,0	0,05
Malic acid (g/l)	2,4	0,03	2,3	0,23	2,8	0,22	2,6	0,23
pH	3,3	0,05	3,3	0,02	3,2	0,04	3,2	0,03
Saccharose (g/l)	x	x	0,3	0,26	0,3	0,56	0,2	0,34
Tartaric acid (g/l)	5,6	0,08	2,8	0,19	3,7	0,18	4,1	0,15
Total acids (g/l)	5,7	0,02	5,3	0,17	6,3	0,31	5,7	0,18
Sugar (g/l)	x	x	9,1	0,67	9,8	13,55	4,6	0,55

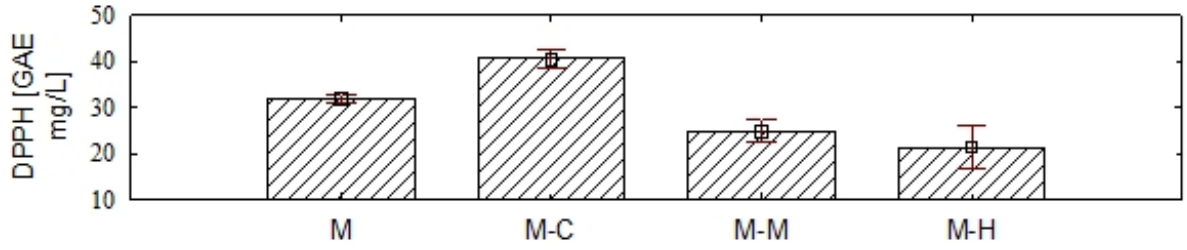

Fig. 1: Values of antioxidant activity of must variants
M – must before fermentation; M-C – control sample; M-M – medium toasted chips added to the must; M-H – heavily toasted chips added to the must

EXPERIMENT B – WINE

A survey of the development and changes in the antiradical activity recorded in individual wine samples by means of DPPH after the addition of chips to fermented wine is presented in Figure 2. Experimental wine samples were in contact with toasted chips for a period of six months. The highest antiradical activity was always observed in Variant II (Medium toasting) while the lowest one was observed in Variant III (control).

Measured values demonstrated that the antiradical activity showed an increasing tendency to depend of the period of time that the wine was in contact with oak chips. The highest values were recorded in Variant II; After the end of the maceration period, the average value of antiradical activity was 68.57 mg/l. In all experimental variants and, independent, of the moment of addition of chips, the most fundamental increase in the antiradical activity was recorded in the period before the last sampling, i. e. between the fourth and sixth month of maceration. A rapid increase in antiradical activity was also observed also in the initial stages of the experiment, i. e. between the 7th and the 30th day of maceration.

The character of measured values of antiradical activity can also be explained as follows: in the case of the addition of chips into the must, this activity was low because of the presence of oxygen that was supplied into the sample together with the chips. During the contact of chips with wine, the antiradical activity increased thanks to the leaching effect of alcohol that supported the extraction of phenolic substances (i. e. antioxidants) into wine.

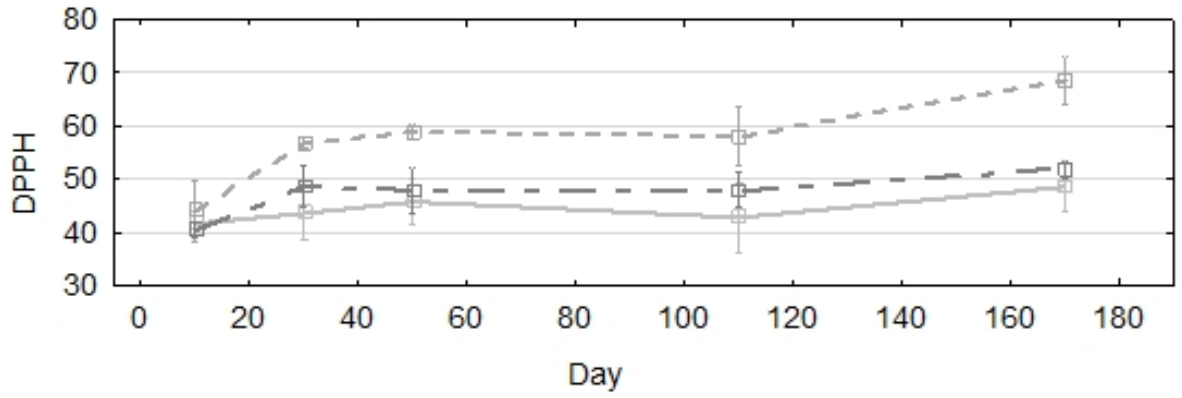

Fig. 2: Development of antiradical activity in individual wine samples
Variant II /dashed line/ – medium toas.; Variant I /double-dashed line/ – heavy toas.,
Variant III /solid line/ – control

Table 2: Mean scores of all samples calculated after the sensory evaluation of aroma

		Must			Wine	
		Control	Medium	Heavy	Medium	Heavy
Smell	Fruitiness	5.4	4.6	4.6	4.8	5.4
	Degree of toasting	1.2	4.4	3.8	12.8	10
Taste	Fruitiness	6.6	4.8	4.8	2.6	5.8
	Degree of toasting	1.2	5.6	10.8	6.6	5.6
	Woody tones	1.2	7.2	7.2	6.4	5.2

SENSORY ANALYSIS

As far as the smell was concerned, the following parameters were evaluated within the framework of sensory analysis: fruitiness and degree of toasting in the smell of wine and fruitiness, and the degree of toasting and woody tones in its taste. Points awarded for all parameters were summarized to calculate the average scores of individual samples. The results are presented in Table 2.
Variants with chippings added to the wine were further evaluated using a special table. Sommeliers evaluated individual types of those aromas that were typical for wines. The results are presented in the form of radial charts in Figure 3.

Pérez-Coello et al. (2000) stated that benzene compounds represent a group of substances determining a typical varietal character of the wine aroma. They involve aromatic alcohols, aldehydes and volatile phenols. When these substances are released from the chippings, their concentration in the wine increases. Increased concentrations of 4-ethylguaiacol and 4-vinylguaiacol can be determined just in wine samples that have been in contact with chippings. This means that the application of chippings supports the manifestation of a certain varietal character of the wine. In our experiments, however, the most pronounced varietal character was detected in the sample that was not treated with chippings (Variant III). As far as its smell and taste were concerned, the average results of this sample evaluation were always 7.6 points. Nevertheless, the varietal character could also occur in samples treated with chippings. In this case, however, their typical aroma was dominating, and for that reason the varietal character was not overly pronounced. The aroma of the sample with medium toasted chippings (Variant I) was mostly evaluated as that of vanilla (5.2 points), hazelnut (2.6 points) and/or spicy (5.6 points). As far as the taste was concerned, caramel was dominant with 4.4 points followed by coffee (2.6 points). A hazelnut taste was also very pronounced (5 points). In the variant with heavily toasted chippings (Variant II), the vanilla aroma was again dominant (5.2 points); and this was followed by coffee aroma with 3 points. As far as the taste was concerned, freshness, vanilla and spicy character were evaluated as the most pronounced (all three received 6 points) and that of caramel was evaluated with 4.4 points. The degradation of lignin resulted in the occurrence of a new series of volatile phenols that were extracted from wood. Vanillin was again the most frequent aromatic substance. This compound occurs in many types of wood and it also plays the most important role. It was also discovered that the degree of toasting did not influence the concentration of vanillin in wine significantly (Herjavec et al., 2007). In our opinion, it is possible to accept this theory, especially when regarding the fact that the evaluation of the vanilla aroma was either very similar or the same in both variants with the application of chippings, because vanilla dominated not only in smell, but also in taste. Vanillin concentrations in wine ranged from 0.3, to 0.8 mg/l. As usual, the perception threshold of this compound is 0.5 mg/l, but in synergy with other aromatic substances its aroma is often much more intensive than it would correspond to its concentration in wine (Steidl and Leindl, 2003). According to experts, the flower aroma (both in smell and taste) was most pronounced in the control sample (5.6 points). The aroma of the sample with medium-toasted chippings was the least pronounced and was evaluated with only 2 points.
Flak et al. (2013) studied effects of various types of oak wood on the quality of red wine. Concurrently, they compared the effects of revitalised barrique barrels, new barrique barrels and application of chips into already used barrique barrels. The woody taste was detected more or less in all variants under study. The best results of sensory analysis, however, were obtained in the variant with revitalised barrique barrels.
Flesch et al. (1998) studied the effect of wine on endotelium-dependent vasorelaxation (i. e. its effect on the myocardial infarction) and found out that this was a specific property of barrique red wines, obviously due to their high content of phenolic substances.
Herjavec et al. (2007) evaluated the quality of white wine fermented in barrels made of Croatian oak wood and found out that compounds present in this wood not only changed the sensory properties of the wine, but

Fig. 3: Aromatic profiles of wine: A – smell (control sample); B – taste (control sample); C – smell (medium-toasted chippings); D – taste (medium-toasted chippings); E – smell (heavily-toasted chippings); F – taste (heavily-toasted chippings)

also the concentration of phenolic acids.

Kozlovic et al. (2010) studied, effects of Croatian and French acacia barrique barrels on the quality of Istrian wine. The results indicated that there were marked differences between the acacia and oak barrels, especially regarding concentrations of individual volatile phenols and oak lactones. In the course of the barrel aging of the wine, above all an increase in compounds such as furfural, 5-methylfurfural, guaiacol, eugenol and trans-eugenol was recorded.

Laszlavik et al. (1995) aged red wine in Hungarian oak barrels for a period of six months. Within this time interval, the phenolic composition of wine and the levels of gallic, protocatechuic, vanillic, syringic, caffeic, coumaric, and ellagic acids, as well as those of vanillin, 5-hydroxymethyl-2-furaldehyde, and 5-methyl-2-furaldehyde were changing. Ellagic and gallic acids were the compounds that played the most important role within the process of wine aging and their concentrations increased in dependending on the type of wine and wood.

Liberatore et al. (2010) monitored changes in the quality of white wine ('Chardonnay') fermented in barrique barrels and aged on the lees. They tested altogether three variants, viz. fermentation and aging in a stainless tank, in a barrique barrel with yeast (i. e. using the sur -lie-method) and in a barrique barrel after the removal of a part of lees after the end of the fermentation.

Matejicek et al. (2005) studied changes in the contents of phenolic compounds present in red wine aged in barrique barrels; particular attention was paid to concentrations of derivatives of benzoic and cinnamic acids and of furaldehyde. Together with p-hydroxybenzaldehyde, vanillin, 2-furaldehyde, 5-methoxy-2-furaldehyde and 5-methyl-2-furaldehyde, the following compounds were detected in extracts from natural wood, toasted wood and wine: gallic, protocatechuic, p-hydroxybenzoic, vanillic, caffeic, syringic, p-coumaric, ferulic, benzoic and ellagic acids. Syringaldehyde was only detected in the toasted wood. The presence of ellagic acid could be a good indicator of the maturity of wine aged in barrique barrels. The presence of furaldehydes can also be onsidered as a typical parameter of barrique wine because no traces of this compound were detected in samples of natural (i. e. non-barrique) wine.

Rapp et al. (1996) mentioned that volatile phenolic compounds (e. g. vanillin, eugenol, and syringaldehyde) occur in wine due to the reaction of alcohol with lignin that takes place during wine storage in barrique barrels.

CONCLUSIONS

In samples with the addition of chippings into must the following parameters were analyzed: titratable acids, glucose, fructose, pH, tartaric acid, malic acid, acetic acid and citric acid. In this case, the difference between the control and wine with the addition of chippings was not too big. This means that – in general – toasted oak chippings did not markedly influence the aforementioned basic parameters of analyzed samples.

When estimating the antioxidant activity it was demonstrated that the addition of chippings into the must was associated also with the supply of a certain amount of oxygen and that this resulted in the occurrence of oxidative tones, both in the must and the wine. Because of this, the highest antioxidant activity was found in the control sample, whereas the lowest one was found in Variant II with the heavily toasted chippings (the difference was approximately 50 %).

The moment when the chippings were applied plays an important role in the process of antioxidant activity. If chippings were applied to the must, the resulting antioxidant activity was markedly lower than in the cases when chippings were added to the wine for a period of six months. Recorded values showed that the antiradical activity increased depending on the duration of the period of wine contact with the chippings. The highest antiradical activity was recorded in samples containing medium-toasted chippings; in this variant, the antioxidant activity had nearly doubled. The lowest values of antioxidant activity were found in Variant III (i. e. without chippings). If chippings were added to wine samples, the antiradical activity increased due to the presence of alcohol because it supported the extraction of phenolic substances (i. e. antioxidants) from oak wood.

Within the framework of these experiments, the Variant III (control) showed the best varietal character as well as the most pronounced flower aroma (both in smell and taste), while in variants with chippings a spicy character and vanilla tones were dominant (caramel and coffee tones were not too distinctive).

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