A STUDY ON THE SULPHUR CONCENTRATION AND SENSORY PROPERTIES OF WINE IN BOTTLES WITH CORK AND GLASS STOPPERS

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In this study, properties of wine stored for twelve months in transparent bottles with two types of glass stoppers and with two types of cork stoppers were evaluated. Experiments were performed with samples of white, rosé and red wine. Bottles were stored in darkness at two different temperatures, 13 °C and 22 °C. During the experiment and also after its end, concentrations of free and total sulphur dioxide and sensory parameters of stored wine were monitored. As far as the residual concentration of sulphur dioxide (SO_2) was concerned, the best results in the variant with a new type of the glass stopper were recorded because its permeability for the sulphur dioxide was the lowest. Cork stoppers were more permeable for oxygen and for that reason they were suitable for red wine stored for longer periods. On the other hand, however, better results with the storage of white and rosé wine were obtained in bottles with glass stoppers because they protected stored wine against undesirable oxidation.
Keywords: glass stopper, cork stopper, sulphur dioxide, sensory analysis

Untersuchung der Schwefelkonzentration und der sensorischen Eigenschaften von Wein in Flaschen mit Naturkork und Glasverschluss. In dieser Untersuchung wurden die Eigenschaften von Wein, der zwölf Monate in transparenten Flaschen mit zwei verschiedenen Glasverschlüssen und mit zwei Typen von Naturkork gelagert wurde, ausgewertet. Die Experimente wurden mit Proben von Weiß-, Rosé- und Rotwein durchgeführt. Die Flaschen wurden im Dunkeln bei zwei verschiedenen Temperaturen (13 °C und 22 °C) gelagert. Während des Experiments und auch danach wurden sowohl die Konzentrationen von freiem und gesamtem Schwefeldioxid wie auch die sensorischen Parameter aufgezeichnet. Hinsichtlich der Restkonzentration von Schwefeldioxid (SO_2) wurden die besten Ergebnisse bei der Variante mit einem neuen Typ von Glasverschluss gefunden, da seine Durchlässigkeit für Schwefeldioxid die niedrigste überhaupt war. Naturkorken waren durchlässiger für Sauerstoff, und aus diesem Grund sind sie für länger gelagerte Rotweine geeignet. Andererseits wurden jedoch bei der Lagerung von Weiß- und Roséweinen in Flaschen mit Glasverschluss bessere Ergebnisse erzielt, weil diese den Wein gegen unerwünschte Oxidation schützen.
Schlagwörter: Glasverschluss, Naturkork, Schwefeldioxid, Sensorik

In general, bottle closures are one of the most important factors that influence the quality development of wine (Godden et al., 2005) and cork represents one of materials that are most frequently used for sealing wine containers (Giunchi et al., 2008). However, this type of closures has also certain disadvantages and, moreover, the world production of natural cork cannot keep pace with the increasing demand for this raw material. Oxidation and some other wine defects caused just by cork closures are the main reasons of the dissatisfaction of wine growers (He et al., 2013). For that reason it is necessary to pay attention to alternative materials that could satisfy the demand and, at the same time, replace cork closures in such a way that they would eliminate shortcomings of cork closures.

As far as bottle closures are concerned, the main requirement of winemakers is that they should not influence or change the quality of bottled wine (Chatonnet et al., 2010). However, results of some studies indicate that cork as a natural material communicates with wine, so that some specific substances are transferred into bottled wine (Callejon et al., 2007; Sefton and Simpson, 2005). This transfer may run not only in the direction from cork into wine but also vice versa (Capone et al., 1999). So, for example, it is possible that oxidation of phenols may take place in the course of storage of bottled wine. The variability of raw cork and its changing properties represent the main reasons why wine producers are looking for some new materials and for alternative solutions of this problem (Gibson et al., 1981). The main reasons of looking for new and/or improved stoppers are efforts to increase wine quality and to control it in a better way. Thanks to these efforts, problems associated with the preservation and improvement of wine quality, are nowadays in focus of our interests and new types of stoppers are evaluated more and more. So, for example, relatively cheap and practical, plastic and crown screw caps and closures (Fortes et al., 2004) are frequently used as well as different other alternatives to cork caps (Pereira, 2007).

Glass stoppers represent a real innovation in the field of wine making. Their major advantages involve sensory neutrality, inertness, impermeability, hardness, and attractiveness for consumers (Revi et al., 2014). In contradistinction to cork closures, glass stoppers prevent the onset of microoxidation and this may show a positive effect on some selected types of wine. This means that it still remains to be questionable if the microoxidation of wine is a desirable feature of produced wine or not and if it can be influenced by the type of bottle closure.

The aim of this study was to test advantages and disadvantages of cork and of a new type of glass stoppers and to evaluate effects of these closures on the quality development of bottled wine.

MATERIAL AND METHODS

CHEMICALS

0.02 mol/l iodine solution, 1 mol/l NaOH solution, 0.5 % starch solution, and 16 % H_2SO_4 solution were obtained from Lach-Ner (Neratovice, Czech Republic).

CHARACTERISTICS OF BIOLOGICAL SAMPLES

In this experiment, wine samples of the varieties 'Pálava' (white) and 'Blaufränkisch' (rosé and red), vintage 2012, from the Mikulov wine sub-region (Czech Republic) were tested. Bottles containing white and rosé wine were closed with two different types of glass stoppers, i.e. with new and old types of Vino-Lok glass stoppers (Preciosa, Jablonec nad Nisou, Czech Republic), natural cork stoppers, and technical cork stoppers with natural cork disks attached on their two sides (Proneco, Brezova, Czech Republic). Bottles containing red wine were also closed with the new type of glass stoppers, natural cork stoppers, and technical cork stoppers.

CHARACTERISTICS OF WINE STOPPERS

Technical cork stoppers (made of cork granulate, 38 x 24 mm), natural cork stoppers (38 x 24 mm) and glass stoppers Vino-Lok were used. In order to seal the glass stopper in the bottle neck, a sealing ring made of a DuPont copolymer resin Elvax 550A + 2 % Elvax CE 9619-1 (DuPont, Wilmington, Delaware, USA) was used.

In this experiment, two types of Vino-Lok glass stoppers were used; High Top 18.2 (VL-O) and its new version High Top 18.5 (VL-N) (Fig. 1).

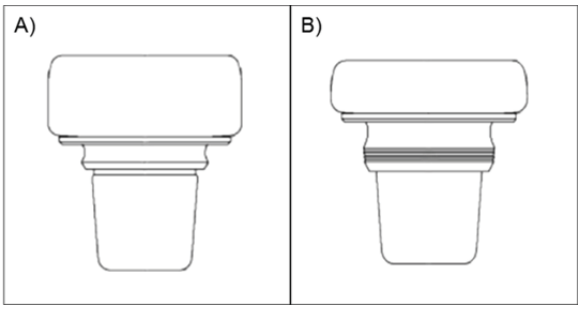

Fig. 1: Types of glass stoppers. A) Vino-Lok High Top 18.2 (VL-O = old type), B) Vino-Lok High Top 18.5 (VL-N = new type)

ESTIMATION OF SULPHUR DIOXIDE

The concentrations of free and total sulphur dioxide were estimated. For the estimation the method of iodometric titration was used (Ribéreau-Gayon et al., 1982). It is the Method OIV-MA-AS323-04B Type IV (Resolution Oeno 377/2009) (OIV, 2009). Correction was used to determine reductones using acetaldehyde. Estimations were performed in intervals of 1, 3, 6, 9 and 12 months of storage.

SENSORY EVALUATION

Selection of nine specialized expert assessors for sensory analysis of wines according to ISO 8586-2 (the second level test) from the Department of Viticulture and Oenology (Faculty of Horticulture, Mendel University in Brno, Lednice, Czech Republic) participated in a blind sensory evaluation of stored wine samples. In order to assure the objectivity of results, individual wine samples were presented in a random sequence. Each variant of wine, stopper and temperature was presented in three replications. The evaluation of wine was performed using the official OIV 100-points scale.

STATISTICAL METHODS

In order to verify the statistically significant differences between the stoppers, analysis of variance was used. This was done with the statistical software Statistica 10 (© StatSoft). This software was also used for the creation of graphs together with Excel software (Microsoft Office).

RESULTS AND DISCUSSION

This study deals with the quality changes and development of wine stored at different temperatures in bottles with cork and glass stoppers. The behaviour and concentrations of sulphur dioxide in bottles with different closures were monitored and the sensory analysis (tasting) of wine was performed after the end of a storage period of 12 months.

ESTIMATION OF SULPHUR DIOXIDE

Sulphur dioxide is an important antioxidant that protects stored wine against oxidation processes. Due to its antioxidative and antibacterial properties, SO_2 is the agent that is most frequently used for the protection of wine (Guaita et al., 2013). In wine, it occurs in its free and bound forms (Santos et al., 2013). Sulphur dioxide can be used as an indicator of the oxidative status of wine exposed to effects of oxygen (Godden et al., 2001). Volatile sulphur compounds play an important role also in the smell (odour) of wine because their even very low concentrations may become the cause that the wine has "gone off the taste" (Swiegers et al., 2005).

In the course of wine storage, concentrations and forms of sulphur dioxide change and it can be partly lost. There may be different causes for these losses, e.g. due to its oxidation in bottles with high concentrations of oxygen and/or its oxidation caused by already oxidised phenols; this usually results in production of sulphates and causes losses of total SO_2. Losses of SO_2 caused by its leakage through the cork are also important (Saidane et al., 2013). The loss of SO_2 is often correlated with the over-oxidation (i.e. browning tendency) of stored wine (He et al., 2013). Just because of these losses it is necessary to monitor concentration of SO_2 in wine and to check it also in the final product.

In this study, concentrations of sulphur dioxide were monitored during the whole storage period (i.e. within

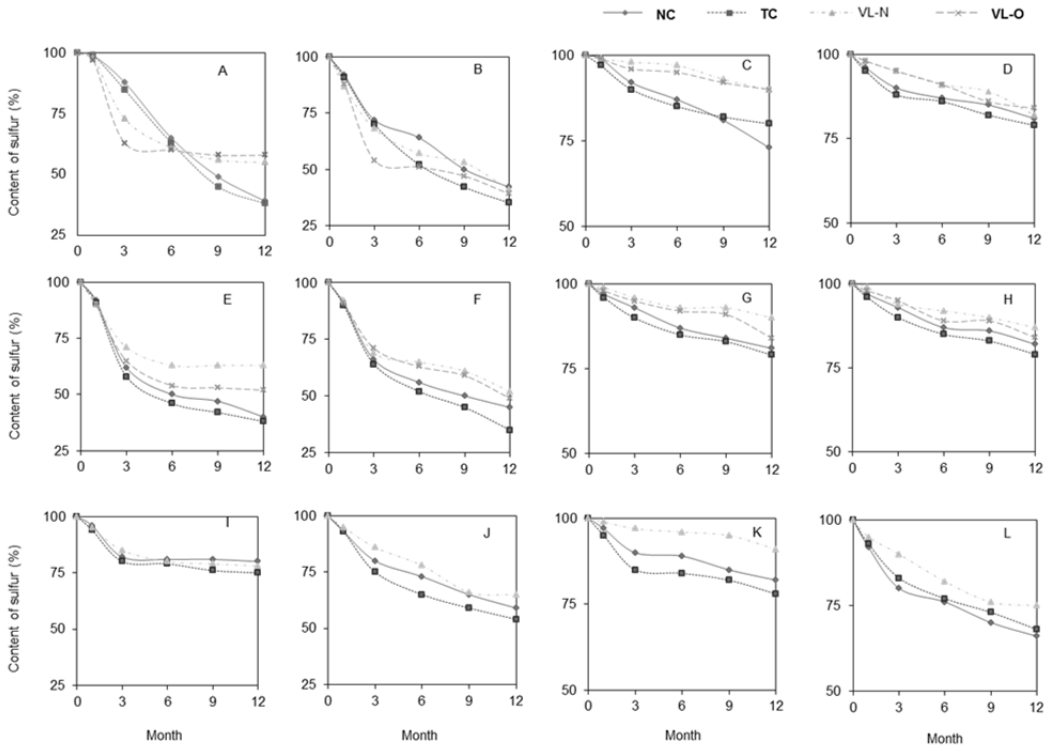

Fig. 2: Estimation of sulphur dioxide concentration in wine. A) Free SO_2 in white wine stored at 13 °C; B) Free SO_2 in white wine stored at 22 °C. C) Total SO_2 in white wine stored at 13 °C. D) Total SO_2 in white wine stored at 22 °C. E) Free SO_2 in rosé wine stored at 13 °C. F) Free SO_2 in rosé wine stored at 22 °C. G) Total SO_2 in rosé wine stored at 13 °C. H) Total SO_2 in rosé wine stored at 22 °C. I) Free SO2 in red wine stored at 13 °C. J) Free SO_2 in red wine stored at 22 °C. K) Total SO_2 in red wine stored at 13 °C. L) Total SO_2 in red wine stored at 22 °C; NC = Natural cork, TC = Technical cork, VL-N = Vino-Lok new type, VL-O = Vino-Lok old type

the period of 12 months). It was found out that the behaviour of sulphur dioxide was intensively influenced by the type of stopper already after a short period of storage; this observation was corroborated also by some other authors (He et al., 2013). A decrease in the SO2 concentration was observed in all experimental variants but in bottles with glass stoppers this decrease was lower than in bottles with cork closures (Fig. 2).

CONCENTRATIONS OF FREE SULPHUR DIOXIDE

Within a period of 12 months, the loss of free SO_2 recorded in white and rosé wine stored at 13 °C and 22 °C, respectively, in bottles with natural cork stoppers was about 60 % (Fig. 2). The highest losses (65 %) of sulphur dioxide were recorded in bottles with technical cork stoppers (Fig. 2). In bottles with natural cork stoppers, the loss of SO_2 was not significantly influenced by the temperature. When stored at different temperatures, bottles with glass stoppers showed a higher variability of SO_2 losses. In bottles with VL-O stoppers containing white wine and stored at 13 °C, the loss of free SO2 was about 45 % (Fig. 2A) while in bottles with VL-N glass stoppers, the corresponding loss was 29 % (Fig. 2A). In bottles with VL-O glass stoppers containing rosé wine and stored at the temperature of 13 °C, the loss of free SO2 was about 43 % (Fig. 2E) while in bottles with VL-N stoppers the corresponding loss was 36 % (Fig. 2E).

As far as stored samples of red wine were concerned, losses of free sulphur dioxide were generally lower than those from bottles containing white and rosé wine. In bottles with natural cork stoppers and stored at 13 and 22 °C, these losses were about 17 % (Fig. 2I) and 41 %

(Fig. 2J), respectively. Similarly as in variants with stored samples of white and rosé wine, losses of sulphur dioxide from bottles containing red wine and sealed with technical cork stoppers were higher than those from bottles with natural cork stoppers.
In the variant with red wine samples stored in bottles with technical cork stoppers at temperatures of 13 and 22 °C, the recorded losses of sulphur dioxide were about 24 % (Fig. 2I) and 47 % (Fig. 2J), respectively. Similarly as in variants with stored samples of white and rosé wine, the lowest losses of sulphur dioxide were recorded in bottles with glass stoppers. The loss of sulphur dioxide from bottles containing samples of red wine closed with glass stoppers and stored at 13 °C was only 11 % (Fig. 2I) while from bottles stored at 22 °C the corresponding loss was as much as 36 % (Fig. 2J).

CONCENTRATIONS OF TOTAL SULPHUR DIOXIDE

Sulphur dioxide added into wine shows a capability to be nearly completely bound and only its small amounts remain to be present in the form of free sulphur dioxide. In wine, SO_2 is bound predominantly on carbonyl compounds. Free and bound sulphur dioxide then represent the total concentration of SO_2 in stored wine (Kilmartin, 2010).
In samples of white wine stored in bottles with natural cork stoppers at temperatures of 13 and 22 °C, losses of total SO_2 were 27 % (Fig. 2C) and 20 % (Fig. 2D), respectively. In samples of rosé wine stored in bottles with natural cork stoppers at temperatures of 13 and 22 °C, losses of total SO_2 were similar, i.e. about 19 % (Fig 2G and 2H). In samples of red wine stored in bottles with natural cork stoppers at temperatures of 13 and 22 °C, losses of total SO_2 were 18 % (Fig. 2K) and 35 % (Fig. 2L). In bottles with technical cork stoppers containing samples of white and rosé wine and stored at 13 and 22 °C, losses of total SO2 were about 21 % in all variants (Figs. 2C, D, G and H). In bottles containing samples of red wine, sealed with technical cork stoppers and stored at temperatures of 13 and 22 °C, losses of total SO_2 were 21 % (Fig. 2K) and 31 % (Fig. 2L), respectively; these losses were lower than those of the total SO_2 from bottles with natural cork stoppers.
In all experimental variants, losses of total sulphur dioxide from wine stored in bottles with glass stoppers were lower than those from bottles with cork stoppers. In bottles with glass stoppers VL-O and VL-N that contained samples of white wine and were stored at the temperature of 13 °C, the recorded losses of free SO2 were equal to 10 % (Fig. 2C). In case of white wine samples stored in bottles with glass stoppers at the temperature of 22 °C, the recorded losses of free SO2 were higher and made about 17 % (Fig. 2D). There were differences between losses of total sulphur dioxide from rosé and white wine samples stored in bottles with glass stoppers. At the temperature of 13 °C, recorded losses of total SO_2 from bottles with VL-O and VL-N glass stoppers were 19 % and only 11 % (Fig. 2G), respectively. Also in samples of rosé wine stored at the temperature of 22 °C the losses of SO2 were recorded. In bottles with old and new types of glass stoppers, the losses of 16 % and 12 % were recorded (Fig. 2H). Samples of red wine were stored only in bottles with the new type of glass stopper (i.e. with VL-N). When comparing samples of red wine with those of white and rosé wine, the lowest losses of sulphur dioxide were recorded in bottles with glass stoppers. In bottles with VL-N stoppers stored at 13 °C, the recorded loss of SO2 was only 9 % (Fig. 2K). The loss of SO_2 from the same bottles stored at 22 °C was much higher, viz. 22 % (Fig. 2L).

As far as the wine quality is concerned, the estimation of sulphur dioxide plays a significant role. It is generally known that in bottles the concentration of this antioxidant decreases during the period of storage. However, the extent of differences in SO_2 losses in samples of white, rosé and red wine is not quite obvious. It is also not known how much the temperature and the type of stopper contribute to the extent of these losses. Differences between white (and rosé) wine on the one hand and the red wine on the other are more obvious. Regarding the fact that the technologies of making white and rosé wine are similar, the differences in SO_2 loss are not so marked. In samples of white and rosé wine the decrease in the level of sulphur dioxide is bigger than in red wine.
Experimental data indicate that there are also differences between individual types of bottle stoppers. There is a general rule that in bottles with cork stoppers there are

more intensive losses of sulphur dioxide than in those with glass stoppers. The highest losses of sulphur dioxide can be observed in bottles with technical cork closures, probably due to their greater porosity.
The storage temperature did not influence the losses of sulphur dioxide from wine in bottles with cork stoppers too much. Nevertheless, a more significant effect of the storage temperature was observed in bottles with glass stoppers. In this variant, losses of sulphur dioxide were lower at 13 °C.

SENSORY EVALUATION

The sensory evaluation is an integral part of the appraisal of wine quality. This information is very important and plays a decisive role in the process of wine development, aging and also in the field marketing (Stone and Sidel, 2004). This is one of methods that are used most frequently in winemaking practice and it gives precise, exact and consistent results (Meilgaard et al., 1999).

WHITE WINE

As far as samples of white wine were concerned, the best average result was obtained in samples stored in bottles with the new type of glass stopper at the temperature of 22 °C; in this case, the average score was 79.3 OIV points (Fig. 3B). The new glass stopper gave better results also at lower temperatures; in this case, the average score was 78.2 OIV points (Fig. 3A). The worst results of sensory evaluation were recorded in the variant with natural cork; in this case, one sample had to be eliminated because of cork taint (2,4,6-trichloranisol). When comparing variants with different temperatures of storage, the lower variance of results was recorded among wine samples stored at 22 °C.

ROSÉ WINE

As far as samples of rosé wine were concerned, the best average result was obtained in samples stored in bottles with the new type of glass stopper at the temperature of 22 °C; in this case, the average score was 77.9 OIV points (Fig. 4B). When stored at the lower temperature, the best result was recorded in the variant with the old type of glass stopper (VL-O); the average score of these samples was 77.1 OIV points (Fig. 4A). As compared with samples of white wine, better results of sensory evaluation were more consistent in the variant with the low temperature of storage (Fig. 4A).

RED WINE

When evaluating samples of red wine, only variants with the new type of glass stoppers, natural cork and technical cork were stored and evaluated. The sensory analysis identified cork taint in one bottle with natural cork stopper and for that reason this variant was not evaluated and was eliminated. The best average score was recorded in the variant with samples stored in bottles with the new type of glass stopper at the temperature of 22 °C; in this case, the average score was 82.0 OIV points (Fig. 5B). The lowest average result was recorded in the variant with red wine samples stored in bottles with technical cork at the higher temperature (Fig. 5B).

The sensory analysis indicated that the better overall results were recorded in the variant with red wine samples stored in bottles with the new type of the glass stopper. It was found out that the new type of glass stopper was the best variant for all samples of wine under study.
The development of bottled wine quality is very dependent on the amount of oxygen that is absorbed in the course of its making and aging (Godden et al., 2005). It is generally known that an excessive amount of oxygen in wine results in its oxidative aroma (Lopes et al., 2006). It can be therefore concluded that stoppers that inhibit the penetration of oxygen into the bottle may show a positive effect on the final aroma of stored and aged wine.

STATISTICAL EVALUATION

After the analysis of variance on the significance level of 0.05, after 12 months of storage, statistically significant differences were found in white wine stored at 13 °C, both for free and for total SO_2; for rosé wines in both storage variants and also for free and even total SO_2; for red wine in both storage variants, but only for total SO_2. In the analysis of variance for sensory analysis, statistically significant differences were found only in the storage variant at 22 °C of white wine.

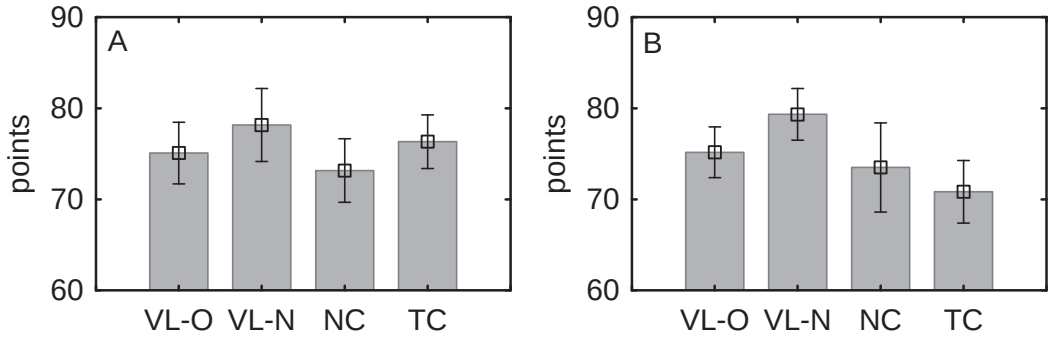

Fig. 3: Means with 0.95 conf. interval of the sensory assessment of white wine; NC = Natural cork, TC = Technical cork, VL-N = Vino-Lok new type, VL-O = Vino-Lok old type; A) Stored at 13 °C, B) Stored at 22 °C

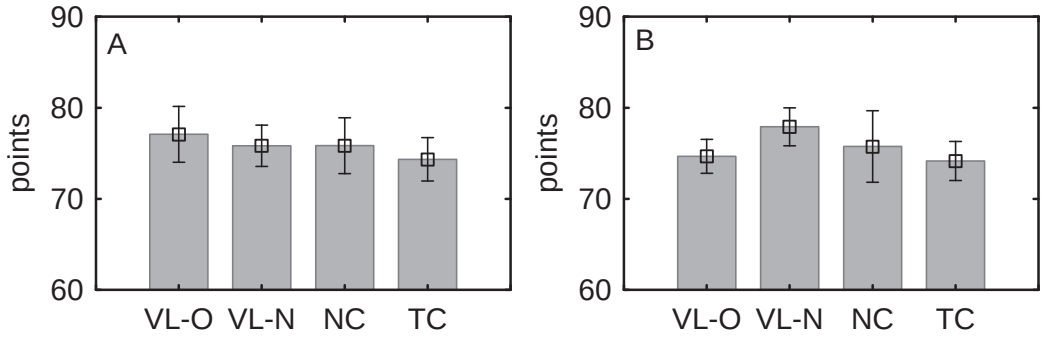

Fig. 4: Means with 0.95 conf. interval of the sensory assessment of rosé wine; NC = Natural cork, TC = Technical cork, VL-N = Vino-Lok new type, VL-O = Vino-Lok old type; A) Stored at 13 °C, B) Stored at 22 °C

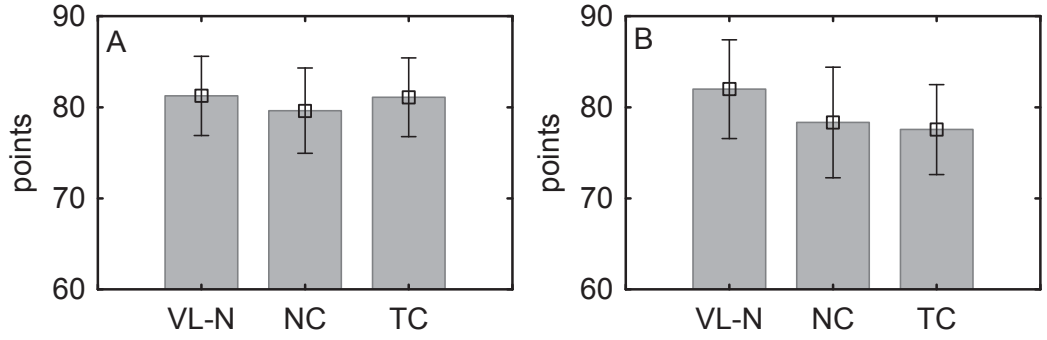

Fig. 5: Means with 0.95 conf. interval of the sensory assessment of red wine; NC = Natural cork, TC = Technical cork, VL-N = Vino-Lok new type, VL-O = Vino-Lok old type; A) Stored at 13 °C, B) Stored at 22 °C

CONCLUSIONS

It was found out that in the course of wine storage and aging there were very different trends in losses of sulphur dioxide. In bottles with cork stoppers, the decrease in the concentration of SO_2 was bigger than in those with glass stoppers. The highest losses of sulphur dioxide from stored wine were observed in bottles with technical cork stoppers. In bottles with glass stoppers, the loss of SO_2 was lower by approximately 50 percent than in bottles with cork stoppers. The best result was recorded in bottles with the new type of glass stoppers because in this case the loss of sulphur dioxide was the lowest. Also, the sensory analysis of all wine samples indicated that the new type of glass stoppers was the most suitable for the storage of wine in bottles.

In general, cork stoppers are more permeable for oxygen and for that reason they are more suitable for the longer period of red wine storage. They are not suitable for longer periods of storage of white and rosé wine. It is recommended to store these wines in bottles with glass stoppers because they protect the content against undesirable oxidation. If wine bottles are produced for immediate consumption (i.e. within the period of one year) it is recommended to use glass stoppers.

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