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## THE IMPACT OF ENOLOGICAL PRODUCTS FOR TARTARIC STABILIZATION ON WINE FILTERABILITY

### Article Highlights

- The impact of enological products for tartaric stabilization on wine filterability was assessed
- The experiments were carried out using Quality Filterability Test
- Used products generally did not worsen the filterability of white and rosé wine
- Products containing mannoproteins and metatartaric acid reduced red wine filterability

### Abstract

*The aim of this research was to evaluate the impact of the most commonly used agents for tartaric stabilization of wine, such as metatartaric acid, carboxymethyl cellulose (CMC), mannoproteins and gum arabic, on the filterability of white, rosé and red wines previously pre-filtered and prepared for the final filtration and bottling. Wine filterability after sweetening with rectified concentrated must was also assessed. The experiments were carried out using the Quality Filterability Test and the obtained results were expressed through the filterability index (FI), and maximum filterable volume ( $V_{max}$ ). The results confirmed that the used enological agents for the inhibition of tartaric instabilities generally did not worsen the filterability of white and rosé wines ( $FI < 20$ ). However, it was evident that products containing mannoproteins and metatartaric acid considerably reduced red wine filterability ( $FI > 500$ ). The correlation between white and rosé wine turbidity and filterability was recorded in the trials but the same trend was not registered for the red wine. The results of this study are important since membrane (final) filtration of improperly prepared wine characterized by low filterability can both increase the costs and lead to holdup on the bottling line.*

*Keywords: wine, filtration efficiency, tartaric stabilization, filterability index, turbidity.*

Wine on the market must have an appropriate color, aroma and taste. The great majority of wines also need to be clear (without visible residues and/or turbidity). Over time, unstable wine compounds can create turbidity or crystalline sediment in bottles due to polymerization reactions. For that reason, modern wine production usually includes a clarification and stabilization phase whose goal is to remove unstable compounds and obtain a product that will be exempt

of any changes in clarity during aging. The clarification and stabilization procedures need to be carried out carefully and professionally in order to minimize the consequences on the wine quality, manifested by certain removal of aromatic and phenolic substances. One of the most common wine instabilities is related to the appearance of tartaric acid salts crystals (tartrates) during aging.

Nowadays, winemaking practice involves the application of two groups of procedures to achieve wine stability on tartrate salts: the removal of tartrate crystal precursors from wine by physical methods (cold stabilization, electro dialysis or ion exchange procedures) and addition of inhibiting enological stabilizers (carboxymethyl cellulose - CMC, manno-

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proteins or metatartaric acid) that prevent the formation (nucleation and growth) of crystals and their sedimentation, or by modifying their properties thus making them soluble at lower temperatures. Any treatment of wine that causes a change in pH (blending, malolactic fermentation, acidity correction) can affect the precipitation of tartrates and therefore, it is necessary to check the tartrate stability just before bottling.

The final step in wine clarification and stabilization almost always includes filtration. The purpose of filtration is to remove all suspended particles and to achieve microbiological and physical stability of wine, while, at the same time, avoiding the changes in chemical composition and sensory characteristics of wine. Wine filtration generally implies two possibilities - depth filtration and absolute (membrane) filtration. The racking of wine after clarification and a coarse filtration (pre-filtration) usually precede the final filtration prior to bottling. A coarse filtration can be carried out by the individual or combined use of diatomaceous earth, depth filter sheets or cross flow, depending on the degree of self-clarification of wine during storage [1]. The final filtration is mostly a microfiltration.

Suspended and colloidal materials in wine are both responsible for membrane fouling. Suspended compounds include crystals, microorganisms, and large colloidal particles. Furthermore, colloids responsible for filter blockage are associative small molecules which are forming reversible aggregates through weak intermolecular bonds and macromolecules (polysaccharides, proteins and polyphenols) forming aggregates through stronger molecular bonds [2]. Smaller particles are more prone to blocking the membrane than the bigger ones which remain on the surface and usually just reduce the efficiency of filtration [3]. Some winemaking approaches such as prolonged maceration, ageing on lees etc., give wines with a higher content of macromolecules which can cause significant filter blockage [4]. Apart from these components, enological additives like arabic gum, tannins, mannoproteins and CMC can also contribute to filter blockage and a decrease in wine filterability [3]. The presence of these colloidal substances in wine and their potential negative impact on filterability cannot be evaluated only by the clarity assessment. A visual clarity of wine is assessed by determination of its turbidity (expressed in NTU units). This parameter is mostly used for assessment of a wine's suitability for bottling. A correlation between wine turbidity and its filterability does not always exist. The blocking of the filters during microfiltration can occur rapidly or slowly, regardless of the type of pre-filtration applied

and the efficiency achieved. For example, a wine with a NTU reading <1.0 is considered clear enough for bottling, however, this value does not necessarily guarantee lower fouling rates of filtration media and higher filterability. On the other hand, it is also possible that wine with turbidity higher than 1.0 NTU can be easily filtered by microfilter. The differences in wine filterability can also exist among wines demonstrating similar turbidity. This is a result of differences in the origin and composition of wine colloids and particles [3].

Unlike turbidity measurements, modern filterability tests consider and include the impacts of all compounds responsible for membrane blockage in the identification of wines prone to be problematic during sterile filtration. Requirements for obtaining the objective results of filterability tests include: a) wines used for testing must be protein-stable and prepared for bottling (pre-filtered); b) use of the same filtration medium as the one going to be used for final (sterile) filtration prior to bottling process; c) membrane porosities (0.45 or 0.65  $\mu\text{m}$ ) need to be the same in both cases; d) tests and real filtration must be carried at similar temperatures. Filterability tests are carried out on cellulose acetate, cellulose nitrate, polyethersulfone and polyvinylidene difluoride membrane filters.

Published research literature on wine filterability is very poor. In regard to this, the impact of enological agents for tartaric stabilization on wine filterability is mostly unknown. The aim of this research was to assess the influence of most commonly used stabilizing agents such as metatartaric acid, carboxymethyl cellulose (CMC), mannoproteins and gum arabic, on the filterability and turbidity of wines previously pre-filtered and prepared for final filtration and bottling. Moreover, the impact of white wine sweetening with rectified concentrated must on the same parameters was also assessed. The results of this study are important, since the exhaustion of depth medium or blocking of the membrane during final filtration can both increase the costs and lead to holdup on the bottling line.

## MATERIAL AND METHODS

### Wine samples

Wines used in this research were produced from grape varieties Sauvignon Blanc and Merlot grown in the Fruška Gora wine region, northern Serbia. The grape was hand-harvested at technological maturity during the 2019 harvest. White, rosé and red wines were produced in two small-scale Serbian wineries (annual production 10000-20000 L). Processing was

carried out immediately after harvest. The must was sulfited by the addition of potassium metabisulfite (0.1 g/L). Destemmed and crushed grapes were pressed in a pneumatic press for the production of white and rosé wine (3 h long maceration was applied in rosé wine production) and the must was transferred into 2000 L inox tanks for fermentation. The clarification of must was carried out by cold settling with the addition of pectolytic enzyme (Trenolin Frio, Erbslöh, Germany) in the amount of 5 mL/hL. In the case of red wine production, grape pomace was transferred into 2000 L casks after destemming and crushing. Maceration and fermentation were conducted using classical technology in open vessels with a “floating cap”. The cap was punched down by hand three times daily. Alcoholic fermentation in all three wines was carried out by the use of commercial *S. cerevisiae* strain Zymaflore Delta (Laffort, France), previously rehydrated and inoculated in a 0.25 g/kg dose. The yeast nutrient Complex vit (Essedielle, Italy) was added in all three vessels in the amount of 0.25 g/L. The moment of addition was after the one-third of sugar was fermented. The fermentation temperature was maintained at 16–18 °C for white and rosé wines, and at 25 °C for the red wine. After the end of fermentation, red pomace was pressed. All wines were racked from lees 7 days after the end of fermentation, and then sulfited (0.08 g/L potassium metabisulfite). The aging of all wines lasted 6 months, during which another racking and sulfiting as needed were performed. Clarification and stabilization on proteins followed. Appropriate doses of enological agents (bentonite for white and rosé wine and gelatin and bentonite for red wine), determined by small-batch lab tests, were applied. After additional racking, wines were filtered using depth filter sheets (Seitz K series, Pall). White and rosé wines were filtered through K100 (retention rating 1–3 µm) filter sheet while red wine was filtered through K200 (retention rating 3–6 µm) filter sheet.

Filterability tests were carried out 7 days after filtration. Agents for tartaric stabilization of wine were added 48 h before the testing started. The following stabilizing agents were used: liquid gum arabic and mannoprotein (doses of 100, 200 and 300 mL/hL, commercial product Senso Ü, Erbslöh, Germany), carboxymethyl cellulose (doses 100, 150 and 200 mL/hL, commercial product EnartisStab Cellogum L, Enartis, Italy), mannoprotein (doses 50, 100 and 150 mL/hL, commercial product Claristar, Oenobrand, France) and metatartaric acid and gum arabic (doses 2, 5 and 10 g/hL, commercial product MetaGum, Erbslöh, Germany), as well as an oak tannin (doses

1, 5 and 10 g/hL, commercial product Tannivin Superb, Erbslöh, Germany) as a non-commonly used agent for tartaric stabilization. Moreover, white wine sweetening was carried out with rectified concentrated must (MCR 65° brix, Essedielle, Italy). The tests were carried out in duplicate.

## Methods

Reducing sugars, ethanol content, pH, total acidity (expressed as tartaric acid), volatile acidity (expressed as acetic acid), tartaric acid content, total SO<sub>2</sub> content and glycerol content of wine samples were determined using a specialized and calibrated wine device working on FTIR technology called WineScan (Foss Analytics, Denmark). Wine turbidity was determined using a portable nephelometer HI83749 (Hanna Instruments, Italy).

The filterability of used white, rosé and red wines after the addition of enological agents for tartaric stabilization was evaluated using Quality Filterability Test developed by Ju.Cla.S. and Vason Group. The instrument is capable of automatically assessing and calculating three factors: the filterability index (*FI*), the modified filterability index (*MFI*) and the maximum filterable volume (*V<sub>max</sub>*). The filterability index of a wine is an estimation of the time needed to block a specific filter medium during filtration [3]. Around 700 mL of a wine, free of carbon dioxide, at around 20 °C is used for conducting a filterability test. The device measures the time for which 200 mL (*T<sub>200</sub>*) and 400 mL (*T<sub>400</sub>*) of wine pass through a membrane (cellulose nitrate, 25 mm diameter, 0.65 µm absolute porosity) under 2 bar constant pressure (Eq. (1)):

$$FI = T_{400} - 2T_{200} \quad (1)$$

*V<sub>max</sub>* (mL) represents an assessment of the maximum volume that can pass through a 0.65 µm filter before fouling. This volume was not directly measured but was calculated automatically using the following formula (Eq. (2)):

$$V_{max} (mL) = \frac{T_{400} - T_{200}}{\frac{T_{400}}{400} - \frac{T_{200}}{200}} \quad (2)$$

The experts from scientific and industrial sectors gave different limit values which define if the wine is filterable enough. The most often used criterion under which wines could be marked as easily filterable is:

$$FK < 20$$

Wine stability on tartrates was assessed using the Mini Contact Test. In the first phase, the conductivity of wine samples after tempering at -4 °C for 45 min was measured. The measurement was followed

by the addition of potassium bitartrate crystals (1 g/100 ml) in all samples, which were then stored at -4 °C for 45 min for white and rosé wines, and 3 h for red wine. After the estimated time, the conductivity was measured again and the difference in conductivity ( $\Delta S$ ) was determined. The criterion by which wines achieved tartaric stability is  $\Delta S < 50 \mu S$ .

All measurements in the study have been done in triplicate.

### Statistical analysis

Statistical analysis in the present study was performed using Statistica 12.0 (StatSoft). The statistical difference between mean values of parameters was estimated by analyses of variance (ANOVA), at the 95% confidence level. Values detected as significantly different by the use of the Duncan multiple range test were marked with different letters (a, b, c ...).

## RESULTS AND DISCUSSION

The filterability of wine is an important factor which has a significant effect on the costs and efficiency of the bottling stage of wine production. Many winemakers have little awareness of its importance. Moreover, available methods for determination of this factor are limited and specific equipment is required.

Prior to filterability testing, the analysis of basic chemical parameters of wine samples had been carried out (Table 1). White, rosé and red wines were characterized by usual values for alcohol content, pH, acidity etc. It should be stated that only red wine was not dry (residual sugar 5.4 g/L) and it was characterized by significantly higher values for total acidity and glycerol content. Apart from basic quality parameters, turbidity, filterability index and  $V_{max}$  were also determined in control wine samples (without the addition of stabilizing agents). The values are given in Table 2 and used for comparison with the readings after the stabilizing agents' addition. According to the defined standards all wine samples could be considered clear

Table 1. Physicochemical parameters of wine samples

Parameter	White wine	Rosé wine	Red wine
Ethanol (%)	14.31	12.57	13.24
Reducing sugar (g/L)	1.8	0.6	5.4
pH	3.27	3.38	3.49
Total acidity (g/L)	5.9	5.2	8.1
Volatile acidity (g/L)	0.15	0.2	0.4
Tartaric acid (g/L)	3.4	2.1	3.6
Total SO <sub>2</sub> (mg/L)	115	60	40
Glycerol (g/L)	7.1	5.5	11.9

after pre-filtration (depth filter sheets) taking into account the turbidity measurements (NTU) for white, rosé and red wines (0.5, 1.0 and 1.2, respectively). Furthermore, the results for  $FI$  confirm that all control wines were characterized by good filterability ( $FI < 20$ ). This study also gives the values of  $V_{max}$  (expected maximal volume of wine filtered through a given filter surface area) for different wines, although there are different opinions on the accuracy and reliability of this factor. The more recent paper [5] on this subject reported that one should be very careful when making filtration decisions (questions regarding the longevity of the filter set *i.e.* the volume of filtrate obtained from one filter set etc.) based only on this factor since its calculation is based on the extrapolation and proportionality with  $FI$  value.  $V_{max}$  calculation uses the data from the standard filterability test (data obtained after 400 mL of wine has been filtered) to predict a maximum filtration volume. An error in calculation comes from the fact that the cleaner the wine is,  $FI$  is lower and extrapolation error is higher. On the other hand, wines with aggravated filterability exhibit higher  $FI$ , extrapolation is smaller and the error in  $V_{max}$  calculation tends to be smaller. Therefore, in this study we used  $V_{max}$  value only as an indicator of approximate maximal wine volume which can be filtered. Taking in account  $FI$  and  $V_{max}$  values for control wines, it is evident that rosé wine was characterized by the best filterability ( $FI = 2.4$  and  $V_{max} = 4185$ ).

The results of this study confirmed that enological agents used nowadays for the inhibition of potassium bitartrate crystallization have significant impact on wine filterability. In general, it can be seen that the addition of different products for tartaric stabilization did not cause significant changes in white wine turbidity (exception - the addition of 200 and 300 mL/hL of liquid gum arabic and mannoprotein) and filterability index (exception - the addition of 10 g/hL of metatartaric acid and gum arabic).  $V_{max}$  for white wines, which were characterized by good filterability ( $FI$  3.4–12.8), was in the very wide range (1243–4187) which can be misleading for wine producers. Furthermore, two higher used doses of enological products were efficient in achieving tartaric stabilization of white wine, except in the case of metatartaric acid and gum arabic, where only the highest dose (10 g/hL) was recommended.

Contrary to white wine samples, the addition of stabilizing agents in rosé wine caused significant increase in the turbidity. It was evident that 48 h (the time between addition and measurement) was not long enough for stabilization of turbidity. NTU values 2.0–2.5 are a bit higher for rosé wine, however that

Table 2. The effects of enological products for tartaric stabilization application on the filterability of white, rosé and red wine samples; mark (-) means sample not stable on tartrates; mark (+) means sample stable on tartrates. Different letters in the same column for each stabilizing product (including the value for control sample) indicate significant differences between values ( $p < 0.05$ ). n/a: determination was not possible

Product	Dosage	Wine turbidity (NTU)	Filterability index FI (s)	Vmax (mL)	Tartaric stability
White wine					
Control white wine sample		0.5 <sup>a</sup>	4.3 <sup>a</sup>	2412 <sup>b</sup>	-
Mannoprotein	50 (mL/hL)	0.52 <sup>a</sup>	4.5 <sup>a</sup>	2382 <sup>b</sup>	-
Mannoprotein	100 (mL/hL)	0.55 <sup>a</sup>	5 <sup>a</sup>	2135 <sup>b</sup>	+
Mannoprotein	150 (mL/hL)	0.56 <sup>a</sup>	6 <sup>a</sup>	1960 <sup>a</sup>	+
Liquid gum arabic and mannoprotein	100 (mL/hL)	0.92 <sup>a</sup>	3.4 <sup>a</sup>	4187 <sup>c</sup>	-
Liquid gum arabic and mannoprotein	200 (mL/hL)	1.29 <sup>b</sup>	4.3 <sup>ab</sup>	2586 <sup>b</sup>	+
Liquid gum arabic and mannoprotein	300 (mL/hL)	1.30 <sup>b</sup>	5.4 <sup>b</sup>	2074 <sup>a</sup>	+
Carboxymethyl cellulose (CMC)	100 (mL/hL)	0.61 <sup>a</sup>	5.6 <sup>a</sup>	2000 <sup>b</sup>	-
Carboxymethyl cellulose (CMC)	150 (mL/hL)	0.63 <sup>a</sup>	8.7 <sup>b</sup>	1544 <sup>a</sup>	+
Carboxymethyl cellulose (CMC)	200 (mL/hL)	0.62 <sup>a</sup>	8.9 <sup>b</sup>	1483 <sup>a</sup>	+
Metatartaric acid and gum arabic	2 (g/hL)	0.71 <sup>a</sup>	7.4 <sup>a</sup>	1659 <sup>c</sup>	-
Metatartaric acid and gum arabic	5 (g/hL)	0.74 <sup>a</sup>	12.8 <sup>b</sup>	1243 <sup>b</sup>	-
Metatartaric acid and gum arabic	10 (g/hL)	0.78 <sup>a</sup>	30.5 <sup>c</sup>	814 <sup>a</sup>	+
Rosé wine					
Control rosé wine sample		1.0 <sup>a</sup>	2.4 <sup>a</sup>	4185 <sup>a</sup>	-
Liquid gum arabic and mannoprotein	100 (mL/hL)	2.45 <sup>b</sup>	2.4 <sup>a</sup>	4183 <sup>a</sup>	-
Liquid gum arabic and mannoprotein	200 (mL/hL)	2.32 <sup>b</sup>	2.3 <sup>a</sup>	4295 <sup>a</sup>	-
Liquid gum arabic and mannoprotein	300 (mL/hL)	2.10 <sup>b</sup>	2.1 <sup>a</sup>	4704 <sup>b</sup>	+
Carboxymethyl cellulose (CMC)	100 (mL/hL)	2.40 <sup>b</sup>	2.9 <sup>a</sup>	3655 <sup>a</sup>	-
Carboxymethyl cellulose (CMC)	150 (mL/hL)	1.70 <sup>b</sup>	1.9 <sup>a</sup>	4688 <sup>b</sup>	-
Carboxymethyl cellulose (CMC)	200 (mL/hL)	2.15 <sup>b</sup>	2.1 <sup>a</sup>	4590 <sup>b</sup>	+
Metatartaric acid and gum arabic	2 (g/hL)	2.14 <sup>b</sup>	2.1 <sup>a</sup>	4552 <sup>b</sup>	-
Metatartaric acid and gum arabic	5 (g/hL)	2.61 <sup>b</sup>	2.6 <sup>a</sup>	3938 <sup>a</sup>	-
Metatartaric acid and gum arabic	10 (g/hL)	2.34 <sup>b</sup>	2.3 <sup>a</sup>	4086 <sup>a</sup>	+
Red wine					
Control red wine sample		1.2 <sup>a</sup>	6.5 <sup>a</sup>	1910 <sup>b</sup>	-
Liquid gum arabic and mannoprotein	100 (mL/hL)	1.40 <sup>a</sup>	509	549 <sup>a</sup>	+
Liquid gum arabic and mannoprotein	200 (mL/hL)	1.48 <sup>a</sup>	n/a	n/a	+
Liquid gum arabic and mannoprotein	300 (mL/hL)	1.42 <sup>a</sup>	n/a	n/a	+
Metatartaric acid and gum Arabic	2 (g/hL)	1.51 <sup>a</sup>	830.3	438	-
Metatartaric acid and gum arabic	5 (g/hL)	1.70 <sup>b</sup>	n/a	n/a	+
Metatartaric acid and gum arabic	10 (g/hL)	1.66 <sup>b</sup>	n/a	n/a	+
Tannin	1 (g/hL)	1.70 <sup>a</sup>	14 <sup>a</sup>	1200 <sup>b</sup>	-
Tannin	5 (g/hL)	1.92 <sup>a</sup>	74 <sup>b</sup>	707 <sup>a</sup>	-
Tannin	10 (g/hL)	2.45 <sup>b</sup>	145 <sup>c</sup>	668 <sup>a</sup>	-

would not represent a problem for wines scheduled for wine filtration. Just in case, the turbidity of the samples was measured again after additional 48 hours and the NTU values declined to 1.1-1.4, which is evidently more acceptable (results not shown). On the other hand, the addition of products based on mannoproteins, CMC and metatartaric acid in this study did not cause significant changes in wine filterability ( $FI$  was in a rather narrow range 1.9-2.9, reg-

ardless of the product used).  $V_{max}$  values for rosé wine were fairly uniform (3600-4600 mL). Regarding the tartaric stability, only the highest doses of all three used products were able to stabilize rosé wine.

Unlike white and rosé, the red wine was characterized by much worse filterability after the addition of stabilizing products based on mannoproteins and metatartaric acid (CMC is not recommended for use in red wines). The determination of  $FI$  was possible

only in the wines where the smallest doses of these products were added, however they were far higher than acceptable ( $F > 500$ ). The turbidity readings were below 4 which can be considered suitable for bottling according to the industry. Almost all applied doses of mannoproteins and metatartaric acid in combination with gum arabic were efficient in stabilization of the wine. Similar results and problems with fast membrane fouling during red wine filtration were also previously reported [6]. The authors reported that treated red wine (supplemented with phenolic compounds and yeast extract) showed rapid and complete blockage of the membrane (cellulose acetate, 0.2  $\mu\text{m}$ , and dead-end mode under 2 bar pressure). The drop in flux was evident in the first minutes of filtration. It should be kept in mind that in practice, red wine filtration is almost never done on a membrane of such low porosity (0.2  $\mu\text{m}$ ). The possible solution for more efficient red wine microfiltration could be in the use of different hydrolytic enzymes (pectinase,  $\beta$ -glucanase). For instance, a method for determination of the (1,3)- $\beta$ -glucanase specific activity (releasing of only glucose units) in wines was developed and through these trials the filterability of wines (taking into account the impact of  $\beta$ -glucans) was evaluated [7]. The authors reported the increase in the volume of wines being filtered when enzymes with glucanase activity were used (increase in  $V_{max}$  for 37-55% after 24 h at 23 °C). Also, wine filterability did not change during 24 h in the absence of enzyme addition.

The effect of oak tannins on red wine filterability was also assessed in this study (Table 2), although these compounds are not non-commonly used agents for tartaric stabilization - they are widely used in red winemaking. Only the addition of the lowest dose of tannin was connected with the acceptable filterability ( $F = 14$ ), while none of the three tannin doses provided tartaric stability. The impact of tannin treatment on the wine filtration which was carried out immediately, after three days, and after eight days after the tannin addition was previously investigated [1]. The filtration was carried out through 0.45, 0.65 and 0.8  $\mu\text{m}$  polyvinylidene difluoride membranes (diameter 20 mm). The addition of tannin caused an increase in the filterability index regardless of the membrane porosity used, so the wines were more difficult to filter compared to the control sample ( $F$  around 15). The increase of the filterability index of a wine filtered through a 0.45  $\mu\text{m}$  membrane was the most pronounced when the filtration was performed immediately and after three days ( $F$  values 15 and 27, respectively). Similar results were also obtained for trials using a 0.65  $\mu\text{m}$  membrane, while when a 0.8  $\mu\text{m}$  membrane was

used,  $F$  values for control and tannin supplemented samples begin to converge. Also, it was reported that prolongation of the moment of filtration through the 0.8  $\mu\text{m}$  membrane (until up to eight days from tannin addition) caused the increase in  $F$  compared to the measurement immediately after the tannin addition.

As already stated, available literature on wine filterability is very poor and the impact of enological agents for tartaric stabilization on wine filterability has not been examined in detail. Previously published studies [8] stated that CMC addition can contribute to a decrease in a wine's filterability, especially if the manufacturer's recommendations (addition to a wine stable on proteins at least 48 h before the final filtration) were not followed. Moreover, CMC could be partially eliminated during filtration. For instance, different doses of CMC (50, 100 and 300 mg/l) caused the increase in the filterability index ( $F$  5, 6 and 24, respectively) in comparison with a highly filterable control wine ( $F = 0.7$ ) [3]. This trend can be explained by the increase of colloidal compounds content coming from CMC use. At the end, the same authors [3] emphasized the importance of checking the amount of CMC remaining after microfiltration, as well as its efficiency in achieving tartaric stability. Furthermore, the effects of both wine components (yeast extract in doses up to 0.5 g/L and phenolic compounds such as anthocyanins and tannins in doses up to 1.5 g/L) and physicochemical parameters (pH 3.3 and 3.7) on the blockage of a micro-filter membrane (cellulose acetate, 0.2  $\mu\text{m}$ ) were investigated [6]. The filtration was carried out with both synthetic and real wine samples, in dead-end mode under 2 bar pressure. They reported that the simultaneous presence of two types of phenolic compounds (anthocyanins and tannins) had a synergistic and more pronounced contribution to membrane fouling, compared to the presence of only one compound. For instance, the presence of proteins from yeast extract (300 mg/g of proteins, free of mannoproteins) caused the increase in membrane fouling. Moreover, it was shown that a decrease in pH value of the sample facilitates the filtration. The given explanation was that with decrease of pH in wine, an increase in the representation of charged functional groups on the protein surface occurs. This is followed by the increase of repulsive forces which prevent protein aggregation and enhance flow rate of the wine during filtration. Furthermore, apart from pH, temperature also has an impact on filtration effectiveness. An increase in wine temperature causes the enhancement in its filterability as a consequence of a viscosity decrease [9].

In the case of white and rosé wine used in this study, wine turbidity was also a good indicator of wine filterability, due to good correlation between wine turbidity and *FI* measurements. On the other hand, this was not the case with the red wine used. The literature and the practice usually report cases when wines with appropriate turbidity (suitable for bottling) are not easily filterable ( $FI > 20$ ), however there are also the opposite cases where turbidity measurements are above 5 NTU while the *FI* values are acceptable ( $FI < 1.5$ ) [3].

At the end of this study, the impact of white wine sweetening with rectified concentrated must on wine turbidity and filterability was assessed 48 h after the supplementation (Table 3). Only the highest dose of rectified concentrated must cause significant increase in wine turbidity and the recorded value (1.42 NTU) is a bit out of the range recommended for bottling ( $< 1$ ). On the other hand, all three added doses caused a significant increase in the *FI* (30-95) which means that the obtained samples are not suitable for effective realization of final membrane filtration (required  $FI < 20$ ).

Table 3. The effects of sweetening on white wine filterability; different letters in the same column for each stabilizing product (including the value for control sample) indicate significant differences between values ( $p < 0.05$ )

Sample	Dosage (g/L)	Wine turbidity (NTU)	<i>FI</i> (s)	<i>Vmax</i> (mL)
Control white wine sample		0.5 <sup>a</sup>	4.3 <sup>a</sup>	2412 <sup>c</sup>
Rectified concentrated must doses	30	0.85 <sup>a</sup>	30.2 <sup>b</sup>	907 <sup>b</sup>
	90	1.06 <sup>ab</sup>	73.7 <sup>c</sup>	806 <sup>b</sup>
	150	1.42 <sup>b</sup>	94.2 <sup>d</sup>	665 <sup>a</sup>

## CONCLUSION

The use of NTU values representing wine turbidity as a parameter for assessing wine filterability is not always reliable. This data by itself cannot be used for the prediction of wine interaction with filter media during microfiltration. Modern filterability tests used for obtaining of the wine filterability index have proven to be simple, rapid and reliable. The results of this

study confirmed that enological agents used nowadays for the inhibition of potassium bitartrate crystallization generally do not worsen the filterability of white and rosé wines. On the other hand, the addition of products containing mannoproteins and metatartaric acid significantly declines red wine filterability. Luckily, the tartaric stabilization of red wines was generally being achieved with small doses of these products. The quantification of wine filterability is important for one making decisions in wineries since the exhaustion of depth medium or blocking of the membrane during final filtration can both increase the costs and lead to holdup on the bottling line.

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NAUČNI RAD

## UTICAJ PRIMENE ENOLOŠKIH PREPARATA ZA STABILIZACIJU NA TARTARATE NA FILTRABILNOST VINA

*Cilj ovog istraživanja bio je procena uticaja najčešće korišćenih enoloških sredstava za stabilizaciju vina na tartarate, na filtrabilnost belog, roze i crvenog vina. U eksperimentima su korištena sledeća enološka sredstva: metavinske kiseline, karboksimetilceluloza (CMC), manoproteini i gumiarabika. Uzorci korištenih vina su prethodno grubo filtrirani i pripremljeni za završnu filtraciju i flaširanje. Procenjena je takođe i filtrabilnost vina nakon doslađivanja rektifikovanom koncentrovanom širom. Rezultati ocene filtrabilnosti tretiranih vina izraženi su preko indeksa filtrabilnosti (FI) i maksimalne zapremine koja se može filtrirati pod datim sulovima ( $V_{max}$ ). Rezultati su potvrdili da korišćeni enološki preparati za inhibiciju nestabilnosti vina izazvanu taloženjem tartarata, uglavnom nisu negativno uticali na filtrabilnost belog i roze vina ( $FI < 20$ ). Međutim, bilo je evidentno da preparati koji sadrže manoproteine i metavinsku kiselinu značajno smanjuju filtrabilnost crvenog vina ( $FI > 500$ ). Utvrđena je korelacija između mutnoće i filtrabilnosti belog i roze vina, a što nije bio slučaj kada je u ogleđima korišteno crveno vino. Rezultati ove studije su važni jer membranska (završna) filtracija neadekvatno pripremljenog vina koje karakteriše niska filtrabilnost (visok FI), može povećati troškove proizvodnje i dovesti do zastoja na liniji za flaširanje.*

*Ključne reči: vino, filtracija, stabilizacija na tartarate, indeks filtrabilnosti, mutnoća.*