fermentation

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The Effect of Different Fining Treatments on Phenolic and Aroma Composition of Grape Musts and Wines

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Abstract: The study evaluated the effect of different fining treatments such as oenological additives and the influence of oxygen and hyperoxygenation on the phenolic and aroma composition of grape musts and wines. The oxidative method, hyperoxygenation and fining agents polyvinyl polypyrrolidone (PVPP), pea protein and chitosan were used for the removal of phenolic compounds compared to the control experiment. The content of phenolic substances was determined by high performance liquid chromatography. A total of 24 volatiles—higher alcohols, ethyl esters and acetate esters were determined using gas chromatography-mass spectrometry. A lower concentration of caftaric acid was observed in hyperoxygenated (0.21 mg·L^{-1}), oxidated (0.37 mg·L^{-1}), PVPP-treated (35.50 mg·L^{-1}), pea protein-treated (42.56 mg·L^{-1}) and chitosan-treated variants (44.40 mg·L^{-1}), in contrast to the control must (caftaric acid 50.38 mg·L^{-1}). In the final wine, the results were similar with a lower concentration of caftaric acid in hyperoxygenated (7.10 mg·L^{-1}), oxidated (14.88 mg·L^{-1}), PVPP-treated (23.49 mg·L^{-1}), pea protein-treated (29.49 mg·L^{-1}) and chitosan-treated variant (30.02 mg·L^{-1}), in contrast to the control wine (caftaric acid 32.19 mg·L^{-1}).

Keywords: fining; polyphenolic compounds; volatile compounds; white wine

Citation: Rihak, Z.; Prusova, B.; Prokes, K.; Baron, M. The Effect of Different Fining Treatments on Phenolic and Aroma Composition of Grape Musts and Wines. *Fermentation* **2022**, *8*, 737. https://doi.org/10.3390/fermentation8120737

Academic Editor: Chih Yao Hou

Received: 2 November 2022
Accepted: 3 December 2022
Published: 13 December 2022

1. Introduction

White wines usually do not contain as many phenolics as red wines, but these compounds still strongly influence the quality of white wines [1].

Phenolic compounds are natural substances that are composed of one or more hydroxyl groups attached to aromatic or benzene rings. They can be categorised as non-flavonoids or flavonoids according to their benzoic ring structure [2]. The polyphenols in wine determine many sensory properties, such as appearance, colour, astringency, bitterness, flavour and also its stability through subsequent oxidative processes [3].

White wines, due to their polyphenolic composition, are more sensitive to oxidation and thus are more susceptible to degradation and oxidative browning [4]. The removal of phenolic compounds from wine with fining agents reduces the wine's capability to react with oxygen [5].

Fining is a widely used oenological operation that consists of adding fining agents to clarify, stabilise or modify grape musts and wines [6]. Thanks to the wide range of oenological additives (animal or vegetal protein base, synthetic polymers, polysaccharide based), there are many options to influence the winemaking process [7].

These interventions are almost always carried out in the grape must during clarification to reduce the negative effect of the fining agents on the quality of the resulting wine [8]. The primary purpose of fining is to reduce unwanted phenolic substances. The removal of oxidised phenols in the production of white wines has thus become a common practice. Proteins used as wine-fining agents present diverse physicochemical characteristics, mainly molecular weight distribution and surface charge density. Wine phenolic compounds interact with protein-fining agents in two main ways: hydrogen bonds and hydrophobic interactions.

The ratio of protein and phenolic concentration is very important. In the case of a small amount of protein, the polyphenols are spread over the surface in a single layer, reducing their hydrophilic character. Subsequently, phenolic compounds are removed by precipitation through self-association of the resulting complexes or by the formation of insoluble protein aggregates. When the protein concentration increases, the phenolic compounds spread over the protein surface. The surface hydrophobic layer then recombines and causes precipitation of the proteins [8]. Various proteins also have different numbers of potential hydrogen binding sites. These agents can be used to remove specific phenolic compounds [7,9].

The most commonly used fining agents are still animal-based proteins, such as pork gelatine, casein, egg albumin or isinglass made of fish. Unfortunately, these products have allergenic potential, so their usage is constantly decreasing. Moreover, vegan-friendly products are becoming more popular.

Another option is to use PVPP, which is a synthetic polymer used to adsorb phenols from beverages [10]. Adsorption of polyphenols by PVPP involves hydrogen bonding between the proton donor from the polyphenol and the carbonyl group from PVPP [11]. The main disadvantage of PVPP is its synthetic origin and its inability for use in production of bio-certified wines.

In 1999, the incidence of the bovine spongiform encephalopathy ('mad cow disease') led to grave concerns about the use of proteins derived from animal sources [12]. Issues with animal proteins have led to increasing interest in exploring alternative wine fining agents as proteins extracted from plant materials as well as non-proteinaceous plant-based materials [6]. One of these recently approved alternative fining agents is chitosan. It is a primary derivative of chitin, a natural polymer and the second most abundant polysaccharide on earth, after cellulose [13].

Moreover, there is an alternative method of how to remove undesirable phenolic substances from the grape must without using any external additives. Hyperoxygenation is a technique used to remove flavonoid phenols using the natural enzymatic equipment of the grape and to contribute to the sensory stability of white wine [14]. These techniques can also affect the expression of wine. With regard to the effect of hyperoxygenation treatment on white wine aroma, it greatly depends on variety, must composition and quantity of oxygen [15].

Original studies reported that maximum flavonoid precipitation took 30 to 60 min, [14] using 15 mg·L^{-1} of oxygen for hyperoxygenation due to the oxygen absorption capacity [16]. More recent studies have experimented with higher amount of oxygen (50–90 mg·L^{-1}) to demonstrate the influence of this technique to chemical parameters of the resulting wines [1,15].

The aim of this study was to assess the ability of reducing the phenolic content of grape must and the resulting wine with selected fining agents. In addition, the process of spontaneous oxidation and hyperoxygenation of must was used. The novelty of this study was to compare the effectiveness of hyperoxygenation, enzymatic oxidation and fining agents to reduce the concentration of phenolic compounds. In addition, the effect of fining agents on the aromatic profile of the resulting wine was investigated.

2. Materials and Methods

2.1. Experimental Design

The experiment was performed at Mendel University in the Czech Republic. The grapes were obtained from the Hibernal grape variety from vineyards of the Institute of Viticulture and Enology (Lednice, Czech Republic) 2021 harvest. The grapes were handpicked during the optimal ripening stage (pH 3.31, total acidity 9.13 g·L^{-1}, 25.4 °Brix). The destemmed and crushed grapes were pressed in a WOTTLE 1200 (Wottle, Austria) pneumatic press. The program selected for pressing was 0.3 to 1.3 bar.

After pressing, the must was divided into three parts: the control, oxidative and hyperoxygenated variants. The control variant was produced in reductive conditions.

Sulphur dioxide (SO_2) was used as an antioxidant at a dose of 50 mg·L^{-1} of SO_2 as potassium disulphate ($K_2S_2O_7$) in order to prevent enzymatic oxidation of phenolic compounds. The oxidative variant, representing enzymatic oxidation of phenolic compounds, was produced in oxidative conditions. The process of spontaneous oxidation was performed with no addition of any antioxidants. The hyperoxygenation variant was submitted to must hyperoxygenation. A micro-oxygenation device, the Oxy Genius Plus (Parsec s.r.l., Italy), was used for must oxidation. A silicon diffuser was connected to an oxygen bottle and placed in the bottom of the tank. The oxygen dose was set at 50 mg·L^{-1} for over five hours due to the device settings. Similar oxygen dose and oxygenation time procedure were already tested [4,15].

Variants with fining agents: PVPP (dose 0.8 g·L^{-1}), pea protein (dose 0.3 g·L^{-1}) and chitosan (dose 0.1 g·L^{-1}) were added to must of each variant and preserved with SO_2 in the same concentration as control. The fining agents for the experiment were produced by the Institut Œnologique de Champagne (IOC) (Épernay, France), and the doses were set as the maximum recommended by the manufacturer. The samples of individual variants were analysed after 24 h of cold sedimentation.

The musts of the control, oxidative and hyperoxygenation variants were racked from the sludge and then inoculated with the active dry wine yeast *Saccharomyces cerevisiae* (IOC, France) at a dose of 20 g·hL^{-1}. After fermentation, all variants were racked from sludge.

Fermentation took place at a temperature of 15–18 °C in 50 L stainless steel tanks with three repetition. The fermentation time varied from 15 to 20 days when control variant wine stopped several days before two others.

After fermentation, the wine was racked and supplied with 40 mg·L^{-1} of SO_2 as $K_2S_2O_7$. The free SO_2 content was maintained at a level of 25–30 mg·L^{-1} during the entire winemaking process.

The samples were collected and analysed from all six variants of the wine after fining operations.

2.2. Basic Chemical Parameters

The sugar concentration of the grape must was analysed with an ATAGO PAL-1 (Atago, Tokyo, Japan) refractometer. The pH value of the must was estimated using a WTW 526 pH meter (WTW, Germany) with a SenTix 21 pH electrode.

The titratable acidity and assimilable nitrogen in the must was estimated using a TITROLINE EASY (SI Analytics GmbH, Mainz, Germany) automatic titrator. A 0.1 mol·L^{-1} solution of sodium hydroxide (NaOH) was used as a titration reagent. For the analyses, 10 mL wine samples diluted with 10 mL of distilled water were used. Individual samples were thereafter titrated up to pH 8.1, again using the SenTix 21 pH electrode. After titration, the consumption of the NaOH solution was read on the titrator display. This consumption was multiplied by the factor of the NaOH solution used for the titration with a coefficient of 0.75. The result was equal to the content of titratable acidity in the wine sample (g·L^{-1}). After titration, 5 mL of formaldehyde was added; the pH value declined, so the sample was again titrated to a pH of 8.1. The assimilable nitrogen was calculated from the second NaOH consumption value; the result was expressed in mg·L^{-1} [17].

The basic parameters of the resulting wine (alcohol, pH, residual sugar, titratable acidity, malic acid, lactic acid, tartaric acid, acetic acid and glycerol) were determined with an Alpha FTIR analyser (Bruker, Bremen, Germany) using the attenuated total reflection sampling technique. Before the first measurement, the spectrometer was thoroughly rinsed with deionized water and the background was determined using a blank sample (deionized water). For the analyses, 1 mL samples were taken with a syringe; 0.5 mL was used to rinse the system while the remaining volume of 0.5 mL was analysed three times. The measured values were evaluated automatically using the OpusWine software (Bruker, Bremen, Germany) [4].

2.3. Analysis of Volatile Organic Compounds

The concentration of the individual volatile compounds in the wine was determined according to the method of extraction using methyl *tert*-butyl ether (MTBE): 20 mL of wine was pipetted into a 25 mL volumetric flask along with 50 µL of 2-nonanol solution in ethanol. This compound was used as an internal standard (in a concentration of 400 mg/L) and 5 mL of a saturated ammonium sulphate ($(NH_4)_2SO_4$) solution. The flask's contents were thoroughly stirred; 0.75 mL of the extraction solvent (MTBE with an addition of 1% cyclohexane) was then added. After another thorough stirring and the separation of individual phases, the upper organic layer was placed into a micro-test tube along with the produced emulsion and then centrifuged. The clear organic phase was dried over anhydrous magnesium sulphate prior to the GCMS analysis. The extraction and subsequent GC analysis were performed three times. The average values and standard deviations were determined using Excel and Statistica 10. The determination was performed in a Shimadzu gas chromatograph (GC-17A) equipped with an autosampler (AOC-5000) and connected to a QP detector (QP-5050A).

Identification was performed using GCsolution software (LabSolutions, version 1.20, Kyoto, Japan). The analysis was performed under the following conditions of separation: column: DB-WAX 30 m × 0.25 mm; 0.25 µm stationary phase polyethylene glycol. The voltage of the detector was 1.5 kV. The sample injection volume was 1 µL with a split ratio of 1:5. The flow of the carrier gas (helium) was 1 mL/min (linear gas velocity 36 cm/s) and the temperature of the injection port was 180 °C. The initial column temperature was 45 °C maintained for 3.5 min, followed by temperature gradients: to 75 °C gradient 6 °C/min, to 126 °C gradient 3 °C/min, to 190 °C gradient 4 °C/min and to 250 °C gradient 5 °C/min. The final temperature was subsequently maintained for 6.5 min. The total length of the analysis was 60 min. The detector worked in SCAN mode in 0.25-second intervals with a range of 14–264. The individual compounds were identified by comparing the MS spectrum and the retention time with the NIST 107 library [18].

2.4. Determination of Total Polyphenols Concentration

The Folin–Ciocalteu method was used to determine the total polyphenolic compounds. All samples were analysed in triplicate; the resulting value was obtained as the average of these measurements.

A 40 µL sample was pipetted into a cuvette (3 mL) and diluted with 1960 µL of distilled water. Subsequently, 50 µL of Folin–Ciocalteu reagent was added to the cuvette. The mixture was then shaken thoroughly. After three minutes, 300 µL of 20% sodium carbonate (Na_2CO_3) decahydrate solution were added. The reaction mixture was shaken and incubated at 22 °C for 120 min. Absorbance was measured using a double-beam spectrophotometer (SPECORD 210, Carl-Zeiss, Jena, Germany) at λ = 750 nm against a blank sample. The results were expressed as a gallic acid equivalent [19,20].

2.5. Determination of Individual Phenolic Compounds by HPLC

The selected polyphenolic compounds were determined using HPLC-DADby the direct sample injection method. The prepared samples were diluted 10× with 100 mM perchloric acid ($HClO_4$) and then used for HPLC analysis.

The following were used in the study: instrumentation: Shimadzu LC-10A binary high-pressure system; controller system: SCL-10Avp; 2 pumps: LC-10ADvp, column thermostat with manual injection valve; Rheodyne: CTO-10ACvp; DAD: SPD-M10Avp; software: LCsolution. The separation was performed on an Alltech Alltima HP C18 3 µm column: 3 × 150 mm at 50 °C. The injection volume of the sample was 20 µL and the flow rate of the mobile phase was set at 0.9 mL min^{-1}. The composition of mobile phase A was 15 mM $HClO_4$; mobile phase B was composed of 15 mM $HClO_4$ and 80% acetonitrile. The gradient program was as follows: 0.00 min—3% B, 3.00 min—6% B, 15.00 min—24% B, 18.00 min—30% B, 19.50 min—36% B, 21.00 min—48% B, 21.50 min—60% B, 22.00 min—60% B, 22.01 min—0% B, 23.99 min—0% B and 24.00 min—3% B. The total analysis time

was 27 min. Data ranging from 200–520 nm was recorded for 24 min. The determination of individual phenolic compounds was performed using a calibration curve [19].

2.6. Statistical Analysis

Statistical analysis and graphs were created using MS Excel 2010 (Microsoft Office 365, Redmond, WA, USA) and Statistica 10 (Copyright © StatSoft, Hamburg, Germany). A one-way analysis of variance (ANOVA) and Fischer's least significant difference (LSD) test were used to compare the means (n = 3) at the level of significance of $p < 0.05$.

3. Results and Discussion

3.1. Basic Analytical Parameters

Table 1 shows the general composition of the final wines from three base variants excluding the influence of fining products. It is evident that different grape must processing influenced the fermentation. Yeast cells need oxygen to produce sterols and unsaturated fatty acids that play a key role in the fluidity and activity of the membrane, which influences ethanol tolerance, fermentative capability and viability of yeast [5]. This explains the higher alcohol concentration in oxidation must variants. On the other hand, SO_2 can slow the onset of fermentation. The presence of SO_2 in concentration 15–20 ppm can reduce the viability of a yeast inoculum from 10^6 to 10^4 [21]—in this case, the control variant wine. For this reason, the must was not fermented to dryness.

Table 1. Basic analytical parameters of final wines.

Wine	Control	Oxidative	Hyperoxygenation
pH	3.31 ± 0.00 a	3.34 ± 0.00 b	3.38 ± 0.00 c
Alcohol % vol	13.8 ± 0.05 a	14.7 ± 0.02 b	14.7 ± 0.01 b
Total acidity g·L^{-1}	8.50 ± 0.00 a	8.74 ± 0.00 b	8.66 ± 0.01 c
Residual sugar g·L^{-1}	23.85 ± 0.02 a	12.15 ± 0.03 b	11.65 ± 0.09 c
Tartaric acid g·L^{-1}	2.85 ± 0.03 a	3.06 ± 0.01 b	2.99 ± 0.03 b
Malic acid g·L^{-1}	3.94 ± 0.11 a	4.40 ± 0.02 b	4.39 ± 0.05 b
Lactic acid g·L^{-1}	0.44 ± 0.01 a	0.63 ± 0.01 b	0.61 ± 0.01 b
Acetic acid g·L^{-1}	0.24 ± 0.00 a	0.26 ± 0.01 b	0.31 ± 0.00 c
Glycerol g·L^{-1}	7.33 ± 0.13 a	10.58 ± 0.11 b	10.26 ± 0.23 c

Note: The average values (n = 3) were combined by contribution to homogeneous groups according to Fisher's Least significant difference (LSD) test, where different letters in the same row indicate significant differences individual variants (α = 0.05).

3.2. Polyphenolic Compounds

Phenolic compounds are natural substances that are composed of one or more hydroxyl groups attached aromatic or benzene rings. Their content is greatly influenced by grape variety, its ripening stage and technological processes to which grapes are exposed [3]. The maceration stage during winemaking determines the phenolic profile [22].

Figures 1 and 2 demonstrate the concentration of total polyphenols in musts and wines of individual variants according to the Folin–Ciocalteu method. The control variant must and wine shows, as expected, the highest amount of total polyphenols. The oxidative and hyperoxygenation variants were quite effective in reducing phenols in the grape must, but unfortunately the concentration of phenols increased in the final wine in these variants so we can assume that the precipitation of the polyphenol by oxygen was not sufficient. A higher increase in polyphenolic substances in wine can be observed in the oxidative variant. The PVPP variant shows strong a affinity for reducing total polyphenols in both must and wine. The chitosan variant reduced total polyphenols better in the must than in the final wine and the pea protein variant was not quite as effective in the must but reduced total polyphenols effectively in the final wine.

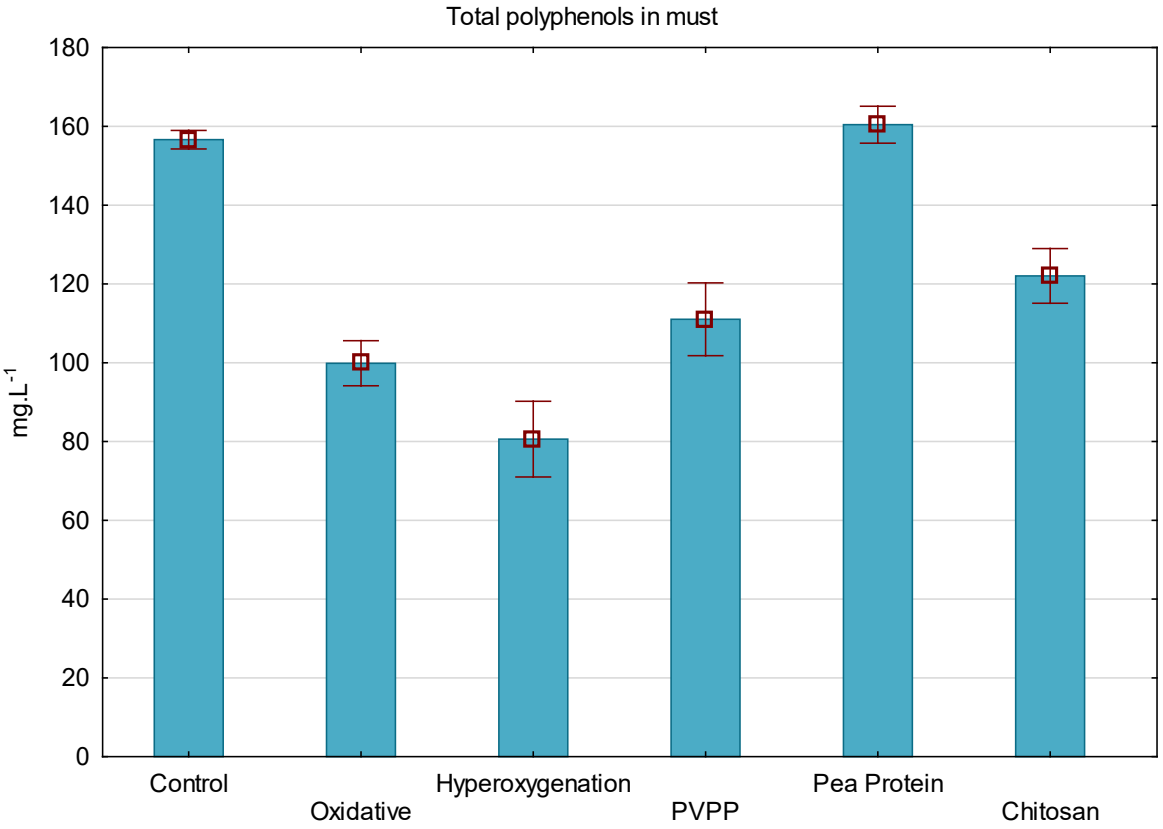

Figure 1. Concentration of total polyphenols in individual variants grape musts.

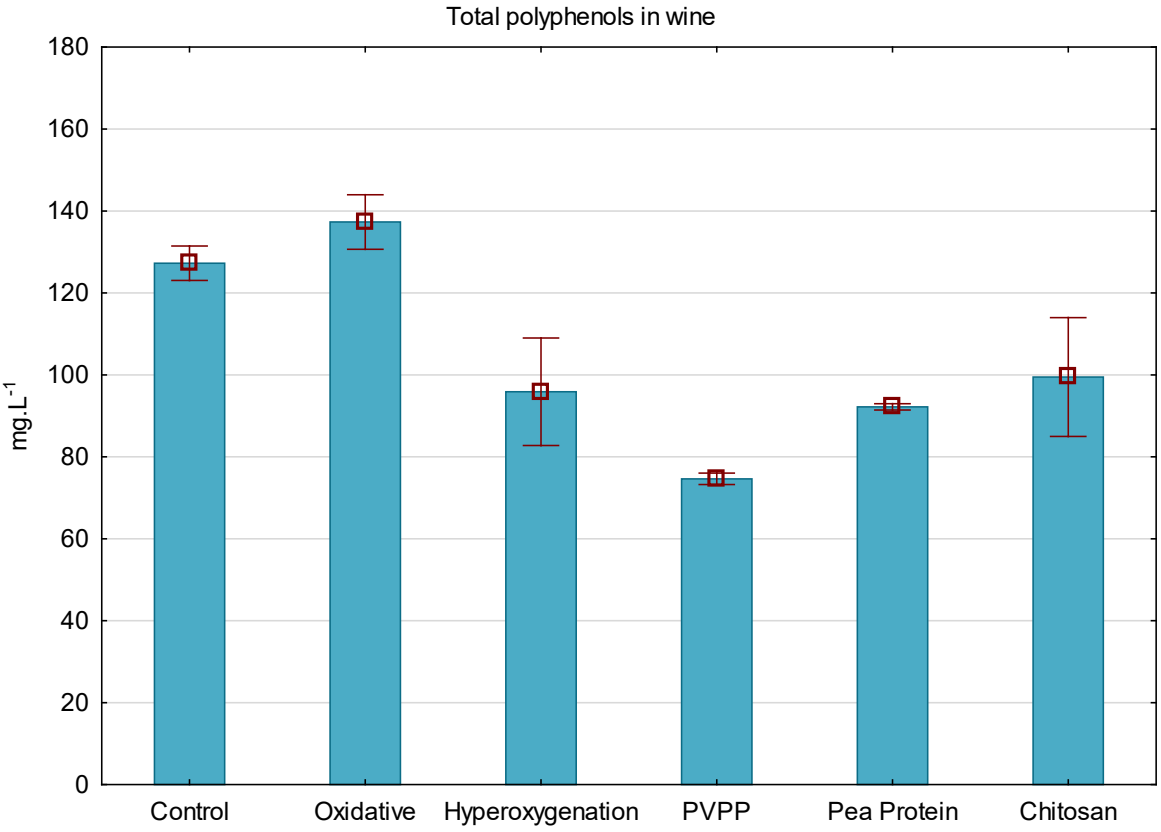

Figure 2. Concentration of total polyphenols in individual variants grape wines.

Hydroxybenzoic acids are a minor component in young wines. Gallic acid is released by the hydrolysis of gallate esters of hydrolysable tannins after at least a few months of maturation [23]. Comparing Tables 2 and 3, there was a slight increase in these compounds in the wine compared to the must. The concentration of gallic acid in the study of Cejudo-Bastante, Hermosín-Gutiérrez, Castro-Vázquez and Pérez-Coello [15] increased after one year of bottle storage.

Table 2. Mean value of concentration of selected phenolic compounds in musts (mg·L^{-1}).

Phenols in Must (mg·L^{-1})	Control	Oxidative	Hyperoxygenation	PVPP	Pea Protein	Chitosan
Hydroxybenzoic acids						
Gallic acid	1.58 ± 0.03 d	0.04 ± 0.01 a	0.08 ± 0.02 a	0.96 ± 0.01 c	1.26 ± 0.01 b	1.29 ± 0.02 b
Protocatechuic acid	1.39 ± 0.01 a	0.21 ± 0.01 b	0.24 ± 0.01 c	1.11 ± 0.00 d	1.90 ± 0.01 e	1.78 ± 0.01 f
4-Hydroxybenzoic acid	0.47 ± 0.01 e	0.51 ± 0.01 ab	0.49 ± 0.01 a	0.4 ± 0.00 d	0.55 ± 0.01 c	0.53 ± 0.01 bc
Vanillic acid	0.06 ± 0.02	0.04 ± 0.02	0.05 ± 0.02	0.03 ± 0.03	0.10 ± 0.03	0.11 ± 0.02
Sirring acid	0.01 ± 0.01 a	nd a	nd a	nd a	0 ± 0.00 a	0.04 ± 0.02 b
Hydroxycinnamic acids						
Caftaric acid	50.38 ± 0.37 e	0.37 ± 0.02 a	0.21 ± 0.02 a	35.50 ± 0.29 b	42.56 ± 1.05 c	44.40 ± 0.57 d
GRP	63.28 ± 0.63 a	0.62 ± 0.02 b	0.65 ± 0.11 b	63.10 ± 1.42 a	63.10 ± 0.49 a	64.51 ± 1.34 a
Caffeic acid	0.22 ± 0.01 a	0.09 ± 0.00 b	0.09 ± 0.01 b	0.22 ± 0.00 a	0.24 ± 0.01 a	0.23 ± 0.01 a
Coutaric acid	10.10 ± 0.07 b	2.78 ± 0.11 a	2.65 ± 0.06 a	8.93 ± 0.03 c	9.83 ± 0.07 b	10.48 ± 0.11 d
p-Coumaric acid	0.16 ± 0.00 b	0.08 ± 0.00 c	0.11 ± 0.01 c	0.20 ± 0.01 a	0.20 ± 0.01 a	0.18 ± 0.00 ab
Fertaric acid	7.88 ± 0.04 e	0.23 ± 0.01 a	0.22 ± 0.02 a	6.82 ± 0.07 b	7.29 ± 0.05 c	7.61 ± 0.02 d
Ferulic acid	0.04 ab	nd c	nd c	0.04 ± 0.00 b	0.03 ± 0.01 ab	0.03 ± 0.00 a
Flavanols						
Catechin	8.36 ± 0.03 e	0.03 ± 0.02 a	nd a	1.38 ± 0.04 b	7.15 ± 0.15 c	7.36 ± 0.07 d
Epicatechin	3.88 ± 0.03 e	0.01 ± 0.01 a	nd a	1.00 ± 0.01 b	3.22 ± 0.06 c	3.33 ± 0.05 d
Procyanidin B1	4.35 ± 0.03 e	nd a	nd a	0.34 ± 0.04 b	3.80 ± 0.04 c	4.11 ± 0.05 d
Procyanidin B2	1.20 ± 0.03 e	nd a	nd a	0.12 ± 0.01 b	0.95 ± 0.02 c	1.02 ± 0.02 d
Procyanidin C	0.65 ± 0.03 c	nd a	nd a	nd a	0.37 ± 0.05 b	0.36 ± 0.04 b

Note: The average values (n = 3) were combined by contribution into homogeneous groups according to Fisher´s Least significant difference (LSD) test, where different letters in the same row indicate significant differences between individual variants (α = 0.05). nd, not detected.

The oxidation variant had the lowest concentration of hydroxybenzoic acids due to the enzymatic oxidation and the hyperoxygenation variant was minimally higher in the must. In the wine, there was a larger increase in gallic acid in the oxidative variant against hyperoxygenation.

The fining agent PVPP had the higher potential to reduce the concentration of hydroxybenzoic acids. Pea protein and chitosan showed similar affinity and their potential to reduce hydroxybenzoic acids was lower.

Hydroxycinnamic acids (HCAs) are the major group of phenolic compounds in grape juice and in white wine [23]. In grapes, HCAs are mainly found in the skins and pulps in the form of tartaric esters [24]. In grape must, enzymatic browning is largely correlated with their content, especially caftaric acid and coutaric acid [25].

The highest concentration was observed in the control variant treated by SO_2 to avoid the enzymatic oxidation. Figure 3 shows a graphic image of the effectiveness of enzymatic oxidation.

Table 3. Mean value of concentration of selected phenolic compounds in wines (mg·L^{-1}).

Phenols in Wine (mg·L^{-1})	Control	Oxidative	Hyperoxygenation	PVPP	Pea Protein	Chitosan
Hydroxybenzoic acids						
Gallic acid	1.71 ± 0.04 a	0.97 ± 0.16 b	0.27 ± 0.01 c	0.93 ± 0.01 b	1.64 ± 0.00 a	1.7 ± 0.00 a
Protocatechuic acid	2.55 ± 0.02 a	1.55 ± 0.08 c	1.83 ± 0.04 d	2.06 ± 0.02 e	2.64 ± 0.02 ab	2.72 ± 0.01 b
4-Hydroxybenzoic acid	0.66 ± 0.01 ab	0.54 ± 0.02 c	0.58 ± 0.01 d	0.63 ± 0.01 b	0.69 ± 0.01 a	0.70 ± 0.01 a
Vanillic acid	0.37 ± 0.02	0.32 ± 0.02	0.34 ± 0.01	0.36 ± 0.01	0.37 ± 0.03	0.34 ± 0.03
Sirring acid	0.13 ± 0.01 a	0.15 ± 0.01 b	0.12 ± 0.01 a	0.12 ± 0.00 a	0.14 ± 0.00 ab	0.13 ± 0.02 ab
Hydroxycinnamic acids						
Caftaric acid	32.19 ± 0.40 b	14.88 ± 1.32 d	7.10 ± 0.15 c	23.49 ± 0.29 e	29.49 ± 0.22 a	30.02 ± 0.31 ab
GRP	31.31 ± 0.27 b	26.21 ± 2.49 a	21.97 ± 0.65 c	26.88 ± 0.21 ab	29.41 ± 0.41 ab	28.56 ± 0.18 ab
Caffeic acid	0.81 ± 0.03 d	0.49 ± 0.05 a	0.39 ± 0.01 c	0.5 ± 0.00 a	0.67 ± 0.02 b	0.67 ± 0.01 b
Coutaric acid	7.94 ± 0.04 c	6.94 ± 0.08 a	4.50 ± 0.02 d	6.93 ± 0.03 a	7.69 ± 0.05 b	7.84 ± 0.04 bc
p-Coumaric acid	0.74 ± 0.00 ab	0.76 ± 0.02 a	0.71 ± 0.01 b	0.67 ± 0.00 c	0.78 ± 0.01 a	0.75 ± 0.00 a
Fertaric acid	7.11 ± 0.04 ac	7.22 ± 0.12 a	7.25 ± 0.07 a	6.21 ± 0.04 d	6.79 ± 0.02 b	6.97 ± 0.03 bc
Ferulic acid	0.32 ± 0.01 b	0.36 ± 0.02 c	0.32 ± 0.00 ab	0.28 ± 0.01 a	0.32 ± 0.00 ab	0.31 ± 0.01 abc
Flavanols						
Catechin	6.84 ± 0.04 a	3.87 ± 0.48 c	1.48 ± 0.04 b	1.24 ± 0.03 b	6.32 ± 0.06 a	6.20 ± 0.07 a
Epicatechin	3.17 ± 0.06 a	1.58 ± 0.28 b	0.37 ± 0.01 c	1.08 ± 0.02 b	2.92 ± 0.03 a	2.86 ± 0.07 a
Procyanidin B1	3.35 ± 0.06 d	1.43 ± 0.29 c	0.25 ± 0.01 a	nd a	2.70 ± 0.04 b	2.50 ± 0.03 b
Procyanidin B2	0.97 ± 0.01 d	0.44 ± 0.08 c	0.07 ± 0.01 a	nd a	0.78 ± 0.01 b	0.76 ± 0.01 b
Procyanidin C	0.12 ± 0.08	nd	nd	nd	0.10 ± 0.06	nd

Note: The average values (n = 3) were combined by contribution into homogeneous groups according to Fisher´s Least significant difference (LSD) test, where different letters in the same row indicate significant differences between individual variants (α = 0.05). nd, not detected.

The oxidative and hyperoxygenated variants have statistically the same level of HCAs with high differences against the control variant in the must, as shown in Table 2.

In the case of fining agents, PVPP reduced caftaric acid by 30%, pea protein by 15.5% and chitosan by almost 12%. The activity of fining agents in wine were similar for PVPP with a 27% decrease in caftaric acid, but the affinity of pea protein and chitosan were half. A higher response of PVPP compared to pea protein was also achieved in the study by [26].

Table 3 shows an increase in HCAs in the oxidative and hyperoxygenation variants in the final wine. This can be explained by the imperfect clarification of the must, because while the pigments from the oxidation variant are partially soluble in the alcohol medium, they are insoluble in the must [14]. Thus, an extensive amount of pigment from the must cause a release of HCAs in the medium with alcohol. In addition, the supplement with SO_2 after fermentation reduced the quinone product back to phenol [27].

On the other hand, the concentration of HCAs in the control variant decreased after fermentation. It is known that hydroxycinnamoyl tartaric acids suffer hydrolysis during the winemaking process; consequently, free HCAs from hydrolysis of the hydroxycinnamoyl tartaric acids were found in the wines [28]. The concentration of grape reaction product (GRP) in the must remained unchanged with no effect of any fining agents.

The conversion of caftaric acid into GRP limits must browning by trapping the caftaric acid quinones [29]. On the other hand, some instability of GRP following its formation has been noted in grape juice exposed to air and this shows that although GRP is not oxidised by grape oxidation enzymes, it can undergo other degradation reactions [30]. Consequently, the amount of GRP drops almost to zero in oxidative variants due to juice air exposure.

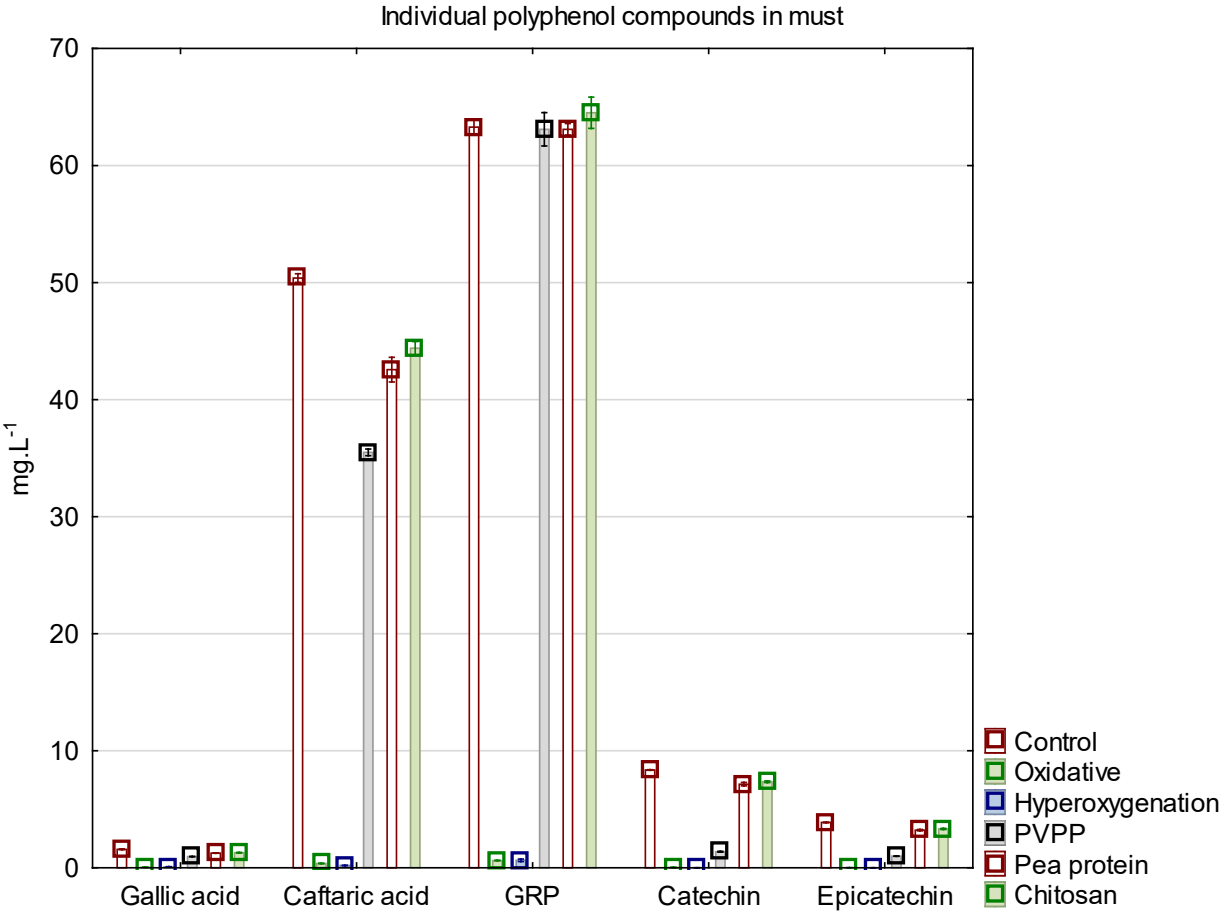

Figure 3. Comparison of selected phenolic compounds in individual variants of must from. The average values ($n = 3$) were combined by contribution into homogeneous groups according to Fisher´s Least significant difference (LSD) test, where different letters in the same row indicate significant differences between individual variants ($\alpha = 0.05$) (Table 2).

Flavonoids are found mainly in grape seeds and skins [24] evolving to polymeric flavanols, also known as tannins, which are responsible for astringency [22]. In must treated by SO_2 or other antioxidants, winemakers reduce redundant phenols with fining agents. Generally, PVPP is very effective in the case of flavanol compounds because it can bind small phenols, such as monomeric and oligomeric flavan-3-ols [31], thus reducing catechin in must by more than 83% and achieving similar efficiency in wine fining. The efficiency of PVPP depends on the pH. For instance, at neutral pH, the adsorption percentage of catechin reached nearly 100% [32].

The alternative product base on pea protein and chitosan was not so effective in reducing catechin. Pea protein was more effective in must with a 14% drop and chitosan during wine fining with a reduction of 9.3% [9]. A reduction of flavanol fraction with pea protein was about 10% in a red wine fining experiment.

As with the previous phenolic groups, enzymatic oxidation showed its potency when both the oxidative and hyperoxygenation variants removed all flavanol substances from the must. When juice and wine contain higher concentrations of these flavonoids, they also become more susceptible to oxidation and subsequent browning [5].

In the final wine, the concentration of flavanols was a little higher in the oxidative variants, but deeply below the control, pea protein and chitosan variants. Only PVPP could reduce flavanols under the concentration of the oxidative and hyperoxygenation variants (Table 3). The comparison of selected phenolic compounds from different groups in Figure 4 shows a comprehensive view of the effectiveness of these individual fining practices.

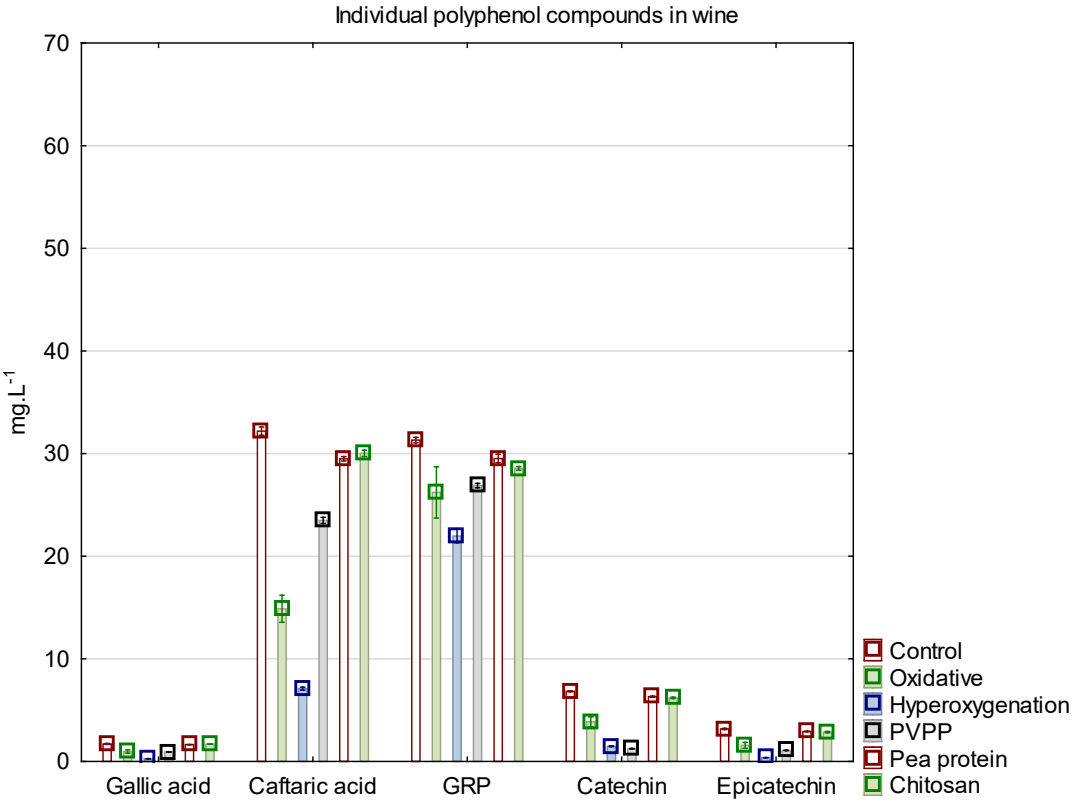

Figure 4. Comparison of selected phenolic compounds in order to individual variants in wine. The average values (n = 3) were combined by contribution into homogeneous groups according to Fisher´s Least significant difference (LSD) test, where different letters in the same row indicate significant differences between individual variants (α = 0.05) (Table 3).

3.3. Volatile Aroma Compounds

The concentrations of volatile aroma compounds are presented in Table 4. There was a total of 25 chemical substances belonging to different groups, such as higher alcohols, C6 compounds, volatile phenols or esters.

Alcohols with more than two carbon atoms are known as higher alcohols. Several of these are produced during fermentation and their esters have intense odours that play a role in wine aromas [8]. Higher alcohols can be anabolically synthesised from intermediates of sugar metabolism or catabolically synthesised from branched-chain amino acids through the Ehrlich pathway by yeast metabolism [33]. Amino acid deamination is especially important in the generation of longer-chain higher alcohols. Synthesis is supported by the presence of oxygen, high fermentation temperatures and higher turbidity of the fermenting juice [21]. The presence of oxygen during prefermentative grape must treatment and its influence on the concentration of volatile compounds have already been confirmed by many studies [1,15,34]. The effect of treatment by oxygen resulted in an increase in C6 compounds, primarily hexan-1-ol and 2-phenylethanol [35]. Table 4 shows an increase in 1-hexanol in the oxidative and hyperoxygenation variants.

The impact of several fining agents on the composition of volatile compounds was investigated. Fining agent addition is a process used to modulate and protect the organoleptic properties of the wines. Unfortunately, fining can also remove some soluble substances including aroma components [36]. The interaction between fining agents and free or bound aroma compounds depends on several factors, such as the physical–chemical characteristics of the fining agent, the chemical features of the target compound and the possible interactions between volatiles and other macromolecules previously linked to the fining agent [37]. Table 4 shows statistical differences in a decrease in isoamyl alcohol with pea protein fining and a decrease of 1-propanol with the PVPP fining variant against the control

variant. According to [38], pea protein has been shown to be effective in reducing phenyl ethyl alcohol in the fining of red wine.

The two main groups of fermentation-derived esters that have long been associated with wine fruitiness are acetate esters and ethyl esters derived from fatty acids and ethanol [39].

Esters are produced naturally by yeast metabolism during alcoholic fermentation. Several esters give pleasurable aromas, such as fruity or floral aromas, and improve the quality of wines made from neutral grape varieties with low varietal aroma characteristics [40]. Oxidation and hyperoxygenation of the must can cause both favourable and undesirable changes in aroma depending on the grape variety. Oxygen affects the formation of volatile compounds, such as esters (acetates and ethyl esters), higher alcohols and medium-chain fatty acids [41]. A low concentration of acetate esters was noted by both oxidative and hyperoxygenation variants, except ethyl acetate and 2-phenyl ethyl acetate, whose concentrations were higher. This was noted also by [34].

Using fining agents in wine shows relevant decline in acetate esters. Isoamyl acetate concentration decreased by about 25%, comparable to the level of the hyperoxygenated variant. Generally, all three fining agents show a similar affinity to acetate esters volatile compounds. The study of [42] compared volatile compounds determined in the wines produced from the differently clarified must and proved that also prefermentative fining influences the composition of volatile compounds in final wines.

Short-chain esters produced fruity characters, while longer-chain esters were responsible for soap-like character. Ethyl hexanoate has an odour reminiscent of apple and violets, ethyl octanoate an odour reminiscent of pineapple and pear, and ethyl decanoate has a floral odour [43].

According to [37], lentil and pea proteins had the highest affinity toward ethyl esters of mid-chained fatty acids (ethyl heptanoate, ethyl octanoate, ethyl decanoate). Similar results are also evident in Table 4. The addition of oxygen to the must also had a significant influence on the formation of ethyl hexanoate, ethyl octanoate and ethyl decanoate. Differences between the control and hyperoxygenated variants were around 16%. These results differ from previous studies [44] that report higher amounts of these compounds in the treatments with added SO_2.

Volatile phenols greatly influence the aroma of wine. The most important molecules in this class are 4-vinylphenol, 4-vinylguaiacol, 4-ethylphenol and 4-ethylguaiacol. The origin of volatile phenols involves the sequential action of two enzymes on a free hydroxycinnamic acid (ferulic, p-coumaric or caffeic acid) substrate. Cinnamate decarboxylase first turns these hydroxycinnamic acids into vinyl phenols. which are then reduced to ethyl derivatives by vinyl phenol reductase [45]. *Saccharomyces cerevisiae* can produce vinyl phenols, but it lacks enzyme vinyl phenol reductase to reduction into ethyl phenols. Therefore, white wines contain variable quantities of vinyl phenols but no ethyl phenols, depending on the concentration of free HCAs. The most unpleasant smelling are vinyl-4-phenol (reminiscent of pharmaceuticals, gouache paint and 'Band Aids') and vinyl-4-guaiacol (carnations) [8]. Higher concentrations of ethyl phenols in wines are related to the activity of *Brettanomyces* yeast, which contains a depsidase enzyme capable of cleaving hydroxycinnamoyl tartaric acid derivatives into free HCAs. These are then enzymatically converted to the sensory active ethyl phenols [45]. The concentration of volatile phenols can thus be affected by the removal of precursors by fining procedures. Using fining agents (Table 3) to the final wine reduces volatile phenols around 30% with PVPP, 25% for chitosan and 20% for pea protein. On the other hand, enzymatic oxidation of hydroxycinnamic acids results in an average 36% decrease in volatile phenols for the oxidation variant and 56% in the hyperoxygenation variant. 4-vinylguaiacol concentration differences between the hyperoxygenation variant and the control variant is almost 90%. A reduction in 4-vinylguaiacol was also observed by [34] in their hyperoxygenated wine.

Table 4. Concentrations of volatile organic compounds in final wines.

Volatile Compounds		Aroma Descriptor *	Control	Oxidative	Hyperoxygenation	PVPP	Pea Protein	Chitosan
Higher alcohols								
Isoamyl alcohol	mg·L^{-1}	ripe fruit	163.26 ± 3.61 a	174.65 ± 2.39 c	166.35 ± 2.85 a	160.44 ± 0.38 ab	152.10 ± 2.32 b	170.26 ± 3.66 ac
Isobutanol	mg·L^{-1}	Ether. fruits	21.05 ± 0.23 abc	21.67 ± 0.28 c	21.03 ±0.17 ab	20.38 ± 0.12 ab	20.72 ± 0.36 a	21.32 ± 0.23 bc
2-Phenylethanol	mg·L^{-1}	rose. talc. honey	8.49 ± 0.23 a	9.45 ± 0.13 c	9.05 ± 0.16 bc	8.99 ± 0.25 abc	8.62 ± 0.11 ab	9.00 ± 0.28 abc
1-Propanol	mg·L^{-1}	Fruits. alcohol	9.45 ± 0.08 a	14.16 ± 0.30 c	14.33 ± 0.26 c	8.24 ± 0.06 b	9.12 ± 0.18 ab	9.67 ± 0.11 a
1-Hexanol	mg·L^{-1}	fresh cut grass	1.08 ± 0.02 a	1.42 ± 0.02 b	1.45 ± 0.02 b	1.15 ± 0.02 a	1.15 ± 0.02 a	1.09 ± 0.03 a
C6 unsaturated alcohols								
(E)−3-Hexen-1-ol	µg·L^{-1}	grass	48.74 ± 1.93 a	33.61 ± 4.87 b	40.05 ± 1.27 ab	42.88 ± 0.46 ab	47.84 ± 1.31 a	39.88 ± 1.28 ab
(Z)-3-Hexen-1-ol	µg·L^{-1}	grass	47.89 ± 2.74 a	41.05 ± 5.24 b	28.42 ± 2.60 ab	35.21 ± 2.33 ab	35.29 ± 1.79 a	38.04 ± 1.11 ab
Acetate esters								
Ethyl acetate	mg·L^{-1}	fruity. nail polish	36.60 ± 0.70 a	46.58 ± 0.49 d	44.04 ± 0.19 c	41.50 ± 1.85 b	38.15 ± 0.36 a	38.42 ± 0.37 a
Isoamyl acetate	µg·L^{-1}	banana	2782.57 ± 13.73 c	2223.62 ± 40.63 b	2052.77 ± 44.24 a	2090.89 ± 56.90 a	2061.34 ± 19.59 a	2069.96 ± 18.70 a
Hexyl acetate	µg·L^{-1}	pear	289.93 ± 7.25 c	216.73 ± 3.58 a	229.29 ± 2.57 b	209.05 ± 3.37 a	210.72 ± 3.51 a	211.24 ± 1.95 a
2-Phenylethyl acetate	µg·L^{-1}	Peaches. honey. roses	285.42 ± 3.70 c	308.54 ± 4.39 b	310.24 ± 2.64 b	217.54 ± 4.46 a	226.56 ± 2.77 a	229.76 ± 3.50 a
Isobutyl acetate	µg·L^{-1}	Fruits	158.97 ± 4.55 e	103.82 ± 1.51 c	91.41 ± 2.26 b	133.94 ± 3.68 a	130.17 ± 3.14 a	118.79 ± 4.13 d
Ethyl esters								
Ethyl butyrate	µg·L^{-1}	Fruits	310.67 ± 7.57 b	391.03 ± 8.14 c	381.64 ± 12.19 c	296.40 ± 8.32 ab	290.21 ± 6.42 ab	274.04 ± 6.53 a
Ethyl hexanoate	µg·L^{-1}	Flowers. green apple	675.99 ± 6.74 b	775.58 ± 13.09 c	804.93 ± 5.43 d	612.16 ± 13.95 a	608.10 ± 8.88 a	639.67 ± 1.63 a
Ethyl octanoate	µg·L^{-1}	Raisins	1057.14 ± 28.46 ab	1053.88 ± 8.43 ab	1080.04 ± 10.04 b	993.63 ± 6.83 c	958.66 ± 11.81 cd	1035.02 ± 5.28 ad
Ethyl decanoate	µg·L^{-1}	flowers. soap-like	356.94 ± 1.74 a	363.36 ± 5.12 ab	372.63 ± 5.87 b	362.36 ± 3.35 ab	300.32 ± 4.00 c	366.23 ± 0.86 ab
Ethyl dodecanoate	µg·L^{-1}		27.26 ± 0.56 a	25.53 ± 0.78 a	26.03 ± 0.81 a	24.80 ± 1.57 a	15.59 ± 0.34 b	21.40 ± 0.09 c
Ethyl lactate	mg·L^{-1}		2.13 ± 0.06 a	2.34 ± 0.20 a	2.10 ± 0.13 a	4.59 ± 0.18 b	4.12 ± 0.51 b	4.36 ± 0.06 b
Diethyl succinate	mg·L^{-1}	melon. vinous	0.17 ± 0.01 ab	0.20 ± 0.01 b	0.14 ± 0.01 a	0.63 ± 0.02 c	0.65 ± 0.01 c	0.57 ± 0.02 d
Diethylmalate	mg·L^{-1}		0.45 ± 0.06 a	0.50 ± 0.03 a	0.45 ± 0.04 a	1.27 ± 0.04 bc	1.33 ± 0.02 c	1.18 ± 0.03 b
Volatile phenols								
4-Vinylguaiacol	µg·L^{-1}	carnations	275.81 ± 15.13 a	113.50 ± 33.38 c	29.69 ± 1.16 b	212.48 ± 3.25 a	226.24 ± 0.39 a	210.37 ± 4.98 a
4-Vinylfenol	µg·L^{-1}	almond shell	1457.49 ± 38.84 c	1253.40 ± 66.83 b	1127.29 ± 32.00 a	1065.47 ± 11.11 a	1127.42 ± 19.01 ab	1073.45 ± 6.49 a
Others								
Acetoin	mg·L^{-1}	buttery. cream	2.24 ± 0.26 a	1.30 ± 0.10 b	1.09 ± 0.06 b	2.13 ± 0.07 a	2.10 ± 0.10 a	2.17 ± 0.03 a
2,3-Butandiol	mg·L^{-1}		577.93 ± 16.75 ab	736.26 ± 11.90 c	725.21 ± 12.43 c	543.94 ± 3.60 a	589.06 ± 5.83 b	587.13 ± 10.23 ab
1,1-Diethoxyethan (Acetal)	µg·L^{-1}		84.26 ± 5.81 a	44.67 ± 2.70 d	25.57 ± 2.02 c	96.82 ± 2.03 b	74.29 ± 3.58 a	97.38 ± 6.60 b

Note: The average values (n = 3) were combined by contribution to homogeneous groups according to Fisher's LSD test, where different letters in the same row indicate significant differences between individual variants (α = 0.05). * Aroma descriptors reported in the literature [1,42].

Moreover, interaction between aroma compounds and polyphenols has received more attention for their influences on odorant volatility [46]. Wine volatile organic compounds–polyphenols interactions and their effects on aroma release and sensory perception represent an current research topic in oenology [47]. The study of [48] investigated the effect of polyphenols on the release of aroma-active compounds in a real apple cider matrix. The presence of (−) epicatechin suppressed the volatility of the esters. An increase in the concentration of hydrocaffeic acid showed a rapid decrease in ethyl hexanoate, ethyl octanoate and ethyl decanoate. The volatility of 2-phenylethanol was decreased slowly when the (−) epicatechin concentration was gradually increased.

4. Conclusions

The study was focused on comparing enzymatic oxidation represented by spontaneous oxidation and hyperoxygenation and using of selected fining agents in order to reduce phenolic compounds. Nowadays, fining agents are often used to reduce unwanted phenols. Fining products based on vegetable proteins or polysaccharides are becoming more popular as alternatives to animal-based proteins. However, their effectiveness is generally lower.

Enzymatic oxidation can serve as a suitable tool in pre-fermentative grape must processing to removed most of undesirable phenolic compounds. On the other hand, the grapes for must oxidation must be in good health quality. The effect on varietal character also needs to be considered. The structure of aromatic substances is partially influenced by oxygen, but also by the structure of phenolic substances itself. Thus, using fining agents in wine also affect the aromatic structure of the wine.

In this direction, more work is needed to understand the relationship between phenolics and aroma.

Author Contributions: Conceptualization, Z.R. and B.P.; methodology, M.B.; validation, M.B.; formal analysis, Z.R.; writing—original draft preparation, Z.R.; writing—review and editing, B.P. and K.P.; visualization, Z.R. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by project IGA-ZF/2022-SI1-007 (Options for removing phenolic substances from must and wine) and by project CZ.02.1.01/0.0/0.0/16_017/0002334 Research Infrastructure for Young Scientists.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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