



MANAGING QUALITY OF AROMATIZED WINE PREPARED BY CO-FERMENTATION OF GRAPE MUST AND BY-PRODUCTS OF ESSENTIAL ROSE OIL INDUSTRY

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ABSTRACT

Aromatized rosé wines with addition of essential rose oil industry wastes during fermentation of grape must were prepared. Six variants: W1-W6 with added 0.05%, 0.1%, 0.25%, 0.5%, 1%, and 2% *Rosa damascena* Mill. waste, respectively, and control wine were prepared. Slight differences in the color shades were observed: the lower the added rose waste, the more intensive peony color was obtained and this observation was confirmed with the increase of the hue angle value – 46.21±0.84 for the control and 54.95±0.70 for the W6. The polyphenol content increased significantly from 355.01±10.14 to 576.08±12.08 µmol GAE L⁻¹ for the control and W6, respectively. The major phenolic acids determined were 3,4-dihydroxy benzoic (up to 65.1±1.1 mg L⁻¹ in W6), gallic (up to 25.9±0.9 mg L⁻¹ in W6) and chlorogenic acid (up to 11.7±0.6 mg L⁻¹ in W5). The GC-FID analysis revealed slight increase of higher alcohols for W5 and W6. β-Caryophyllene, β-citronellol, phenethyl alcohol, rose oxide, and geraniol content increased significantly compared to control. The sensory evaluation revealed most of the panelists preferred W1 and W2 although some of the testers liked better the variants with higher amounts of added waste. The results suggested that rose waste successfully could be utilized for preparation of new aromatized wines with distinctive rose aroma.

1. Introduction

The wine is among the most popular and produced worldwide low alcoholic beverages. The fundamental factors determining the wine quality are geographical region, climate conditions, soils, grape variety, stage of ripeness, yeasts, as well as, vinification (Cioch-Skoneczny *et al.*, 2021; Nardi *et al.*, 2018). The wine aroma is among the most important factors for the wine quality and acceptance (Nardi *et al.*,

2018). The major contributors for the formation of aroma bouquet are the yeast fermentation of grape must and skin contact time (Cabaroglu and Canbas, 2002). Furthermore, the aroma could be modulated by addition of other flavoring substances and these beverages are categorized as aromatized wines. The aromatized wines, according to Regulation 251/2014 of the European Parliament and the Council, are

defined as wines with organoleptic characteristics achieved by addition of natural flavoring substances and/or herbs and spices, including their extracts, and/or flavor products, and combination thereof. Different flavoring materials were used: wormwood, dwarf gentians (*Gentianella* sp.), mint, cinnamon, green cardamom, elderberry, nutmeg, rosemary, juniper, *Hypericum* sp., clove, flat-leaved vanilla, etc. The utilization of agricultural by-products is a rare practice but some aromatized wines exist, i.e. St. Raphael's aperitif wine prepared with bitter orange peels (Buglass, 2011). By-products from the olive oil industry were used in an attempt for replacing sulfur dioxide in wine models (Ruiz-Moreno *et al.*, 2015), and overripe seeds from white grape by-products were added during red wine fermentation in order to investigate the effect on wine color and phenolic substances (Rivero *et al.*, 2017). Attempts for preparation of aromatized wines with addition of essential oil industry main products were made but problems with solubility and separation of the oils and wine during storage were observed. The literature survey suggested, to the best of our knowledge, that no attempts for preparation of aromatized wines with addition of by-products of the essential-oil industry, which is emblematic and widespread in some European and Asian countries (Bulgaria, France, Turkey, Iran, China, etc.), were described. By-products of the rose oil-industry are usually not further utilized and are discarded, although the waste could serve as a valuable raw material for obtaining of biologically active substances (Slavov *et al.*, 2017). For this reason, based on the above-mentioned observations, literature survey and experimental data, the present study aimed to investigate the possibility for preparation and managing quality of aromatized wines with addition of rose oil industry waste in the course of Mavrud must fermentation.

2. Materials and methods

2.1. Materials

2.1.1. Samples and reagents

The *Rosa Damascena* Mill. waste was provided by EKOMAAT Ltd. distillery (Mirково, region of Sofia, Bulgaria; 2016 harvest; waste obtained from certified bio roses was used). The grape used for wine preparation was *Vitis vinifera* L. cv Mavrud (Brestovica, region of Plovdiv, Bulgaria; 2016 harvest) with 23.8% sugars and 8.2% titratable acids. The Lallzyme cuvée blanc and the yeast strain Lalvin D47 were obtained from Lallemand (France). The Polymust press and bentonite were obtained from Laffort (France).

Acetonitrile, acetic acid, dichloromethane, sodium acetate, pyridine, N,O-Bis-(trimethylsilyl)-trifluoroacetamide, gallic acid, 3,4-dihydroxy benzoic acid, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, sinapic acid, rosmarinic acid, cichoric acid and cinnamic acid were obtained from Sigma-Aldrich (USA). The DPPH (2,2-diphenyl-1-picrylhydrazyl) was from Merck (Germany).

2.2. Methods

2.2.1. Preparative

The wines were prepared in the facilities of Villa Vinifera (Brestovitsa, Plovdiv, Bulgaria). The grape was pressed in a hydraulic press and 60 mg L⁻¹ SO₂ and 2g kg⁻¹ Lallzyme cuvée blanc was added. The must was cooled down to 8°C and when clarification occurred the precipitates were removed by filtration. The filtrated must was transferred to a fermentation vessel, warmed to 15°C and inoculated with LalvinD47 (0.25 g L⁻¹). The must was divided in seven vessels – one control and six variants (17 L each) and to each vessel (without the control one) was added rose waste: W1–8.5g (0.05%); W2–17g (0.1%), W3–42.5g (0.25%), W4–85g (0.5%), W5–170g (1%) and W6–340g (2%). The fermentation continued 22 days at 16±1°C and the solid substances were removed by filtration. A combined agent for wine treatment consisted of plant protein, bentonite and polyvinylpolypyrrolidone (Polymust press) was added (6 g per each). The wines were filtered,

bottled with cork stoppers and stored in dark at $18\pm 1^\circ\text{C}$.

2.2.2. Analytical

The ethanol content was determined by the pycnometric method (Cioch-Skoneczny *et al.*, 2021). Total polyphenols were determined according to Singleton and Rossi (1965) with Folin-Ciocalteu's reagent. Gallic acid was employed as calibration standard and the results were expressed as gallic acid equivalents (GAE) per liter of wine. The antioxidant activities were evaluated by [2,2-diphenyl-1-picrylhydrazyl] radical (DPPH) and Ferric Reducing Antioxidant Power (FRAP) methods as described by Slavov *et al.* (2017). The amount of total monomeric anthocyanins was determined by the pH-differential method (Giusti and Wrolstad, 2001). Briefly, the wine samples were diluted in parallel with two buffer solutions: 0.025 M KCl with pH 1.0 and 0.4 M sodium acetate with pH 4.5. After one hour at room temperature ($22\pm 1^\circ\text{C}$) absorption at 520 and 700 nm were measured (1 cm cuvette; spectrophotometer Helios Omega UV-Vis with VISIONlite software (Thermo Fisher Scientific, Madison, USA)). The results were calculated using molar absorption coefficient $26900\text{ L mol}^{-1}\text{ cm}^{-1}$, molecular mass of 449.2 g mol^{-1} and were expressed as equivalents cyaniding-3-glucoside per liter.

The color characteristics of wines were determined with a Helios Omega UV-Vis spectrophotometer equipped with VISIONlite ColorCalc Basic software (Thermo Fisher Scientific, USA) using 1 cm cuvettes. Spectra were recorded in a 380–780 nm range at intervals $\Delta\lambda=2\text{ nm}$. CIELCh color coordinates were calculated using standard illuminant D 65 and 10° observer angle.

The relative proportion of red color from anthocyanes' flavylium cations, $dA(\%)$, was calculated using the equation (1), according to Azar *et al.* (1990):

$$dA(\%) = \left(1 - \frac{A_{420} - A_{620}}{2 \times A_{520}}\right) \times 100 \quad (1)$$

where, A_{420} , A_{520} и A_{620} are the values of absorption at 420, 520 and 620 nm, respectively.

Individual phenolic acids were determined as described by Terzieva *et al.* (2017) with an HPLC system ELITE LaChrome (Hitachi, Japan) equipped with diode array detector Elite LaChrome L-2455. The separation was performed on Supelco Discovery HS C_{18} column ($5\ \mu\text{m} \times 25\text{ cm} \times 4.6\text{ mm}$) operated at 30°C under gradient conditions with mobile phase consisting of 2% (v/v) acetic acid (mobile phase A) and acetonitrile (mobile phase B) at a flow rate 0.8 mL min^{-1} . The gradient used was: 0–1 min: 95% A and 5% B; 1–40 min: 50% A and 50% B; 40–45 min: 100% B; 46–50 min: 95% A and 5% B. The gallic, protocatechuic and cinnamic acids were detected at 280 nm and the chlorogenic, caffeic, ferulic, p-coumaric, sinapic, rosmarinic and chicoric acids – at 320 nm.

The composition of aromatized wines was investigated by gas chromatography with flame ionization detector (GC-FID) and gas chromatography with mass selective detector (GC-MS). The GC-FID analyses were performed on Shimadzu GC-17A (Shimadzu, Japan) equipped with TEKNOKROMA TRB-WAX column ($30\text{m} \times 0.32\text{mm} \times 0.25\ \mu\text{m}$) and software GC Solution (Shimadzu, Japan). Sample amount: $1\ \mu\text{L}$; injector temperature: 229°C ; carrier gas pressure: 32 kPa; carrier gas speed: 1 mL min^{-1} ; detector temperature: 250°C ; temperature regimen of the column: starting from 40°C , hold for 1 min, increase with 5°C min^{-1} until 100°C , hold for 10 minutes and increase with $15^\circ\text{C min}^{-1}$ until 220°C .

The GC-MS analyses were performed as follow:

1). Non-volatile polar substances: 0.2 mL ethanolic extract was lyophilized and 50 μL pyridine and 50 μL N,O-Bis-(trimethylsilyl)-trifluoroacetamide (BSTFA) were added. The sample was incubated at 70°C for 40 min. For analysis $1.0\ \mu\text{L}$ from the solution was injected on gas chromatograph Agilent GC 7890 (Agilent Technologies, Palo Alto, CA, USA) with mass-selective detector Agilent MD 5975 and column HP-5ms ($30\text{m} \times 0.32\text{mm} \times 0.25\ \mu\text{m}$ thicknesses). The following temperature regimen was used: initial temperature 100°C

(hold for 2 min) then increased to 180°C with 15°C min⁻¹ (hold for 1 min) and increase of the temperature to 300°C with 5°C min⁻¹ (hold for 10 min); injector and detector temperatures – 250°C, helium was used as carrier gas at flow rate 1.0 mL/min. The scanning range of mass-selective detector was $m/z = 50 - 550$ in split-split mode (10:1).

2). Volatile substances: The aroma substances were extracted according to the procedure described by Uekane et al. (2017). The analyses were performed with gas chromatograph Agilent GC 7890 with mass-selective detector Agilent MD 5975 and Agilent DB-5ms (30 m × 0.25 mm × 0.25 µm) column. The following temperature regimen was used – initial temperature was 40°C and then increase to 300°C with 5°C min⁻¹ (hold for 10 min); injector and detector temperatures – 250°C, helium was used as carrier gas at 1.0mL/min. The scanning range of mass-selective detector was $m/z = 40-400$ in splitless mode.

The individual compounds were identified comparing the retention times and the relative index (RI) with those of standard substances (linear n-alkanes (C₈–C₄₀) injected under the same conditions) and mass-spectral data from libraries of The Golm Metabolome Database (<http://csbdb.mpimg-golm.mpg.de/csbdb/gmd/gmd.html>) and NIST'08 (National Institute of Standards and Technology, USA).

2.2.3. Sensory analysis of aromatized wines

Sensory evaluation was performed according to ISO 13299:2016 with the following indicators: color intensity, aroma intensity, fruity nuances, flowery nuances, grassy nuances, taste intensity, acidity, and bitterness. Briefly, the bottles (15±0.5°C) were opened, poured in wineglasses and served coded to 21 (22-52 years old) untrained consumers. The degree of liking was based on an eleven-point scale (0: absence of the specified indicator, 10: extremely sensing the specified parameter). Organoleptic evaluation was done in three repetitions, and the values of individual attributes were averaged and added together.

2.2.4. Statistical analysis

The analyses were performed in triplicate and the data were given as mean values. Statistical significance was detected by analysis of variance (ANOVA, Tukey's HSD test; value of $p < 0.05$ indicated statistical difference).

3. Results and discussions

3.1. Preparation and characterization of aromatized wines

3.1.1. Preparation and physico-chemical characteristics

Mavrud is among the highly valued local grape varieties. It is specific for the Western Thrace wine region of Bulgaria and traditionally is used for production of red wines. During the last years a tendency for making rosé wines on the basis of Mavrud was observed. The wines are distinctive with well-balanced fruity aroma bouquet and elegant taste of wild forest berries. Six variants with different amounts of added waste and control rosé wine were prepared based on preliminary experiments. At the end of fermentation flavor intensity was amplified in the variants with higher amounts of added rose waste and grassy and incomplete nuances were sensed. After removal of the precipitations and clarification the flavor was significantly improved and more harmonious nuances were detected while the grassy notes significantly decreased. The alcoholic content was in the 14.4-14.6±0.2 % (v) range and pH 3.43–3.62±0.1.

The total monomeric anthocyanins (Table 1) increased in W2 compared to control but then decreased in W4-W6 and the same trend was observed for the relative part of the red color due to flavilium cations of anthocyanins. In general the process of distillation of roses led to destruction of anthocyanins and the waste could not contribute to the final wine anthocyanin content. Slight differences in the color shades were observed: the lower the added rose waste, the more intensive peony color was obtained and this observation was confirmed with the increase of hue angle value – 46.21±0.84 for the control wine and 54.95±0.70 for the W6. From the other side the higher content of polyphenols in the

rose waste could probably contribute to stabilization of the anthocyanins in the beverage due to co-pigmentation (Shikov *et al.*, 2012). The total polyphenol content (TPP) increased significantly from $355.01 \pm 10.14 \mu\text{mol GAE L}^{-1}$ for the control wine to $576.08 \pm 12.08 \mu\text{mol GAE L}^{-1}$ for W6 which is due to extraction of

polyphenols during maceration and co-fermentation. The increased TPP resulted in significant augmentation of antioxidant activity: for control wine 668.33 ± 21.28 (by DPPH) and $918.33 \pm 15.64 \text{ mg TE L}^{-1}$ (by FRAP) compared to W6 – 1991.67 ± 23.95 (by DPPH) and $2850.00 \pm 24.85 \text{ mg TE L}^{-1}$ (by FRAP).

Table 1. Physico-chemical characteristics of control and aromatized wines

	C	W1	W2	W3	W4	W5	W6
TMA, mg CG L⁻¹	2.62± 0.10 ^a	2.57± 0.12 ^a	2.94± 0.11 ^b	2.52± 0.10 ^a	2.38± 0.11 ^{a,c}	2.24± 0.12 ^c	2.06± 0.11 ^c
TPP, μmol GAE L⁻¹	355.01± 10.14 ^{a,b}	344.98± 15.21 ^{a,b}	355.27± 11.18 ^{a,b}	375.32± 12.08 ^{b,c}	395.44± 14.51 ^{c,d}	412.12± 10.14 ^d	576.08± 12.08 ^e
DPPH, mg TE L⁻¹	668.33± 21.28 ^a	558.33± 19.84 ^b	712.50± 18.30 ^c	885.00± 21.35 ^d	1016.67± 17.84 ^e	1423.33± 26.34 ^f	1991.67± 23.95 ^g
FRAP, mg TE L⁻¹	918.33± 15.64 ^a	941.67± 18.41 ^a	1085.00± 17.74 ^b	1288.33± 16.95 ^c	1488.33± 21.54 ^d	2091.67± 19.62 ^e	2850.00± 24.85 ^f
C (Chroma)	23.10± 0.68 ^{a,c}	22.81± 0.88 ^{a,c}	25.43± 0.72 ^{b,c}	22.81± 0.81 ^{a,c}	21.83± 0.95 ^a	23.80± 0.81 ^{a,c}	23.94± 0.72 ^c
h (Hue angle)	46.21± 0.84 ^a	45.34± 1.01 ^a	46.32± 0.74 ^a	44.33± 0.83 ^a	46.78± 0.84 ^a	49.56± 0.68 ^b	54.95± 0.70 ^c
L (Lightness)	82.10± 0.88 ^a	77.07± 1.15 ^{b,c}	79.96± 1.08 ^{b,a}	77.88± 1.17 ^{b,c}	76.06± 1.21 ^c	80.34± 1.35 ^{b,a}	79.86± 1.41 ^{b,a}
a	17.22± 0.45 ^a	17.11± 0.61 ^a	18.84± 0.48 ^b	17.25± 0.51 ^a	15.74± 0.49 ^c	16.42± 0.54 ^{a,c}	14.78± 0.57 ^d
b	15.51± 0.41 ^a	15.12± 0.61 ^a	17.17± 0.48 ^b	15.04± 0.74 ^a	15.12± 0.46 ^a	17.37± 0.50 ^b	18.92± 0.49 ^c
CI, dA %	62.53± 1.05 ^a	65.28± 1.16 ^b	53.32± 1.21 ^c	52.15± 1.30 ^c	56.21± 1.50 ^d	46.34± 1.17 ^e	41.02± 1.24 ^f

TMA – total monomeric anthocyanins; CG – cyanind-3-glucoside; TPP – total polyphenolic content; GAE – gallic acid equivalents; TE – Trolox equivalents; CI, dA% – relative part of the red color due to flavilium cations of anthocyanins; a, b, c, d, e, f, g Values with different letters in a row are statistically different (Tukey's HSD test, $p < 0.05$)

3.1.2. HPLC determination of phenolic acids

Individual phenolic acids were determined by HPLC (Table 2). The highest increase was detected for gallic acid and 3,4-dihydroxy benzoic acid: from $1.2 \pm 0.9 \text{ mg L}^{-1}$ and $5.4 \pm 0.2 \text{ mg L}^{-1}$ in the control wine to $25.9 \pm 0.9 \text{ mg L}^{-1}$ and $65.1 \pm 1.1 \text{ mg L}^{-1}$ in W6, respectively. The increase of phenolic acids quantity in W3-W6 compared to control and W1-W2 could be explained with the addition of higher amounts of rose waste and subsequent extraction during

fermentation, having in mind that the rose wastes are rich source of polyphenols (Shikov *et al.*, 2012). The higher amounts of phenolic acids determined was also related to increase in the antioxidant capacity of the aromatized wines from W1 to W6 (Table 1) and this could be explained with the higher amounts of total polyphenols extracted from the rose waste but not the anthocyanins (no significant difference in the TMA amounts in all wines).

Table 2. Phenolic acids in control and aromatized wines

Compound, mg L ⁻¹	C	W1	W2	W3	W4	W5	W6
Gallic acid	1.2±0.9 ^a	3.3±0.9 ^b	4.1±0.8 ^b	8.8±0.7 ^c	17.9±0.5 ^d	19.2±0.7 ^d	25.9±0.9 ^c
3,4-dihydroxy benzoic acid	5.4±0.2 ^a	9.8±0.4 ^b	15.0±0.6 ^c	22.4±0.3 ^d	24.2±0.9 ^d	32.4±1.0 ^e	65.1±1.1 ^f
Chlorogenic acid	traces	0.1±0.0 ^a	traces	traces	10.8±0.5 ^b	11.7±0.6 ^b	10.2±0.8 ^b
Caffeic acid	0.7±0.2 ^a	0.7±0.2 ^a	0.7±0.2 ^a	1.8±0.1 ^b	1.9±0.1 ^b	2.3±0.2 ^b	3.4±0.2 ^c
Ferulic acid	traces	nd	traces	0.1±0.0 ^a	traces	traces	0.2±0.0 ^a
p-Coumaric acid	2.7±0.5 ^a	2.7±0.5 ^a	2.7±0.5 ^a	2.5±0.5 ^a	3.2±0.4 ^{a,b}	4.0±0.7 ^{b,c}	4.5±0.3 ^c
Sinapic acid	1.0±0.3 ^a	0.9±0.3 ^a	1.0±0.2 ^a	1.1±0.4 ^a	1.2±0.2 ^a	1.5±0.3 ^{a,b}	1.8±0.3 ^b
Rosmarinic acid	traces	traces	traces	traces	traces	traces	0.9±0.2
Cinnamic acid	traces	traces	traces	traces	traces	0.1±0.0 ^a	0.2±0.0 ^a

^{a, b, c, d, e} Values with different letters in a row are statistically different (Tukey's HSD test, $p < 0.05$)

Table 3. GC-FID analysis of control and aromatized wines

Compound, mg L ⁻¹	C	W1	W2	W3	W4	W5	W6
Acetaldehyde	17.1±1.2 ^a	19.9±0.9 ^a	20.3±1.3 ^{a,b}	22.2±1.0 ^{b,c}	24.1±0.9 ^c	25.2±1.1 ^{c,d}	27.0±0.9 ^d
Ethyl acetate	32.7±2.1 ^a	33.8±2.3 ^a	34.7±2.0 ^a	33.3±2.3 ^a	36.0±1.9 ^{a,b}	37.6±1.8 ^b	37.7±1.9 ^b
Methanol	34.2±2.1 ^a	35.1±2.0 ^a	35.2±2.3 ^a	36.4±2.1 ^{a,b}	37.2±2.5 ^{a,b}	38.7±2.6 ^b	39.1±1.8 ^b
2-butanol	29.4±0.9 ^a	30.2±1.2 ^{a,b}	32.8±1.2 ^{a,b}	33.3±1.1 ^b	29.0±1.1 ^a	33.7±1.2 ^b	34.0±1.2 ^b
1-propanol	45.8±0.9 ^a	45.9±1.0 ^a	44.9±1.1 ^a	46.4±1.0 ^{a,b}	46.1±1.1 ^{a,b}	47.7±1.1 ^{a,b}	48.0±1.0 ^b
i-butanol	27.3±1.5 ^{a,b}	24.2±1.4 ^{b,c}	28.1±1.6 ^{a,b,d}	23.8±1.5 ^c	25.6±1.7 ^{a,b,c,d}	26.2±1.7 ^{a,b,c,d}	28.3±1.3 ^d
1-butanol	7.0±0.3 ^a	7.2±0.3 ^a	8.0±0.2 ^b	8.6±0.4 ^{b,c}	8.7±0.2 ^{b,c}	9.0±0.3 ^{c,d}	9.8±0.3 ^d
i-amyl alcohols	7.9±0.4 ^a	8.7±0.5 ^a	8.7±0.4 ^a	8.6±0.3 ^a	8.6±0.4 ^a	8.7±0.5 ^a	8.9±0.4 ^a
Sum of higher alcohols	117.4±1.5 ^a	116.2±1.4 ^a	122.5±1.6 ^b	120.7±1.5 ^{a,b}	118.0±1.7 ^a	125.3±1.7 ^b	129.0±1.3 ^c

^{a, b, c, d} Values with different letters in a row are statistically different (Tukey's HSD test, $p < 0.05$)

3.1.3. GC-FID analyses

The GC-FID analysis (Table 3) revealed slight increase of acetaldehyde, ethyl acetate and methanol from W1 to W6 compared to control. The increased methanol content could be explained with the presence of pectic substance in the rose waste (Slavov *et al.*, 2017). Nevertheless, the amounts determined (even in W6) were within the permissible limits: for example the methanol limit is 250 mg L⁻¹ for white and rosé wines and the higher amount observed in W6 was 39.1±1.8 mg L⁻¹ (Compendium of international methods of analysis – OIV, 2018). The quantity of ethyl acetate determined was in the 32.7±2.1 – 37.7±1.9 mg L⁻¹ range. An aroma similar to acetone is sensed if the concentration of ethyl acetate exceeds a threshold reported most often as being between 100-200 mg L⁻¹ (Cliff and Pickering, 2006). Ethyl acetate concentrations below the threshold can contribute to the depth of body, richness and sweetness of wine and between 30-80 mg L⁻¹ ethyl acetate can add to the wine character and be a part of the pleasant wine bouquet (Plata *et al.*, 2003). The amounts of higher alcohols increased in the W1-W6 variants (except for i-amyl alcohols) compared to the control wine. Higher alcohols, also known

as fusels, plays an important role in the formation of wine aroma and at concentrations below 300 mg L⁻¹ positively influence aroma. The higher amounts negatively affect the proper bouquet of the wine. The aromatized wines and the control rosé had a total amount of fusels in the 117.4-129.0±1.5 mg L⁻¹ range and it could be concluded that addition of rose waste during fermentation did not affect negatively formation of higher alcohols.

3.1.4. GC-MS analyses – determination of polar volatile and non-volatile compounds

Furthermore the aromatized wines were subjected to GC-MS analysis. The preparation of rosé wines with Mavrud grape is a rare practice since this regional grape variety is mostly used for red wine preparation and to the best of our knowledge this is the first report for GC-MS profiling of Mavrud rosé. As a result of the analysis thirty nine polar non-volatile metabolites (amino acids, sugars, acids, sugar alcohols and sterols) were tentatively detected in the control and aromatized wines. In general increase in the content of most of the detected substances from control to W6 was observed (Table 4 and 5).

Table 4. Polar non-volatile metabolites in control and aromatized wines

Compound	RI	C	W1	W2	W3	W4	W5	W6
		% of TIC						
Lactic acid	1066	25.1± 0.7 ^{a, b}	24.2± 0.6 ^a	24.8± 0.8 ^a	26.1± 0.5 ^b	24.3± 0.9 ^{a, b}	27.5± 0.4 ^{b, c}	29.2± 0.6 ^c
L-Valine	1228	10.6± 0.8 ^a	11.4± 0.9 ^{a, b}	12.3± 0.8 ^{a, b, c}	13.2± 0.7 ^{b, c, d}	13.9± 0.9 ^{c, d, e}	15.2± 0.7 ^{d, e}	15.9± 0.9 ^e
Glycerol	1266	426.3± 1.6 ^a	398.8± 1.4 ^b	431.7± 1.6 ^c	532.9± 2.1 ^d	586.8± 2.5 ^e	576.7± 2.0 ^f	592.4± 1.8 ^e
L-Leucine	1272	11.0± 0.7 ^a	11.9± 0.8 ^{a, b}	12.7± 0.9 ^{a, b, c}	13.8± 0.6 ^{b, c}	14.5± 0.7 ^c	12.9± 0.9 ^{a, b, c}	15.2± 0.8 ^c
Phosphoric acid	1278	88.0± 1.0 ^{a, b}	85.2± 0.9 ^a	90.3± 1.2 ^b	92.9± 1.1 ^{b, c}	94.2± 1.0 ^c	93.3± 1.5 ^{b, c}	95.4± 1.2 ^c
L-Isoleucine	1299	10.1± 0.5 ^a	10.8± 0.6 ^a	11.4± 0.5 ^{a, b}	12.6± 0.6 ^b	14.3± 0.7 ^c	14.9± 0.5 ^c	15.6± 0.7 ^c
L-Proline	1307	42.0± 0.9 ^a	41.5± 0.7 ^a	49.3± 0.8 ^b	52.5± 1.0 ^c	58.4± 0.9 ^d	57.5± 0.8 ^d	59.9± 0.9 ^d

Succinic acid	1310	188.2± 1.9 ^a	187.2± 2.5 ^a	190.1± 2.0 ^{a, b}	191.2± 1.8 ^{a, b}	195.4± 1.9 ^b	196.8± 1.6 ^b	195.1± 1.7 ^b
Glyceric acid	1339	55.7± 1.8 ^a	57.6± 1.2 ^a	62.4± 1.0 ^b	69.7± 1.4 ^c	72.9± 1.0 ^d	74.0± 1.1 ^{d, e}	75.4± 1.2 ^e
Fumaric acid	1355	28.0± 1.1 ^a	27.9± 0.9 ^a	30.3± 0.8 ^a	35.0± 1.0 ^b	36.9± 0.8 ^{b, c}	38.3± 0.9 ^{c, d}	39.9± 1.0 ^d
Serine	1362	12.4± 0.6 ^a	13.5± 0.4 ^a	12.4± 0.8 ^a	15.5± 0.5 ^{b, d}	16.8± 0.4 ^{c, d}	16.0± 0.5 ^d	17.1± 0.6 ^{c, d}
L-Threonine	1390	14.8± 0.8 ^a	16.0± 0.7 ^a	17.3± 0.5 ^b	18.4± 0.4 ^c	20.3± 0.7 ^d	21.4± 0.5 ^{d, e}	22.5± 0.6 ^e
L-Malic acid	1488	297.0± 2.0 ^a	298.8± 1.1 ^a	299.9± 1.0 ^a	301.3± 1.9 ^{a, b}	298.4± 1.8 ^a	302.3± 1.9 ^{a, b}	305.6± 1.8 ^b
Pyroglutamic acid	1512	65.6± 0.9 ^a	69.8± 0.8 ^b	74.2± 0.7 ^c	82.0± 0.6 ^d	85.8± 0.8 ^e	86.7± 0.8 ^{e, f}	88.3± 0.9 ^f
Salicylic acid	1516	24.0± 0.4 ^a	25.9± 0.8 ^{a, b}	26.3± 0.7 ^b	27.0± 0.8 ^b	25.2± 0.7 ^{a, b}	26.9± 0.5 ^b	25.8± 0.4 ^{a, b}
L-Aspartic acid	1531	11.0± 0.8 ^a	12.1± 0.9 ^a	12.8± 0.8 ^{a, b, c}	13.7± 0.9 ^{b, c, d}	14.1± 0.7 ^{c, d}	15.2± 0.8 ^d	15.9± 0.7 ^d
L-Threonic acid	1528	88.0± 1.2 ^a	95.2± 1.0 ^b	104.3± 1.1 ^c	110.0± 1.3 ^d	109.2± 1.1 ^d	111.4± 1.0 ^d	112.9± 1.1 ^d
L-(+)-Tartaric acid	1612	224.3± 1.6 ^a	228.4± 1.7 ^b	222.1± 1.4 ^a	230.8± 1.5 ^b	231.2± 1.1 ^b	229.9± 1.2 ^b	235.0± 1.3 ^c
L-Phenylalanine	1646	13.4± 0.9 ^a	14.4± 0.8 ^{a, b}	15.7± 0.7 ^{a, b, c}	16.8± 1.0 ^{b, c, d}	17.3± 0.8 ^{c, d}	17.8± 0.7 ^d	18.0± 0.5 ^d
Vanillic acid	1758	16.1± 1.0 ^a	16.9± 0.7 ^a	19.5± 0.5 ^b	20.2± 0.7 ^{b, c}	21.1± 0.6 ^{c, d}	22.0± 0.7 ^d	22.8± 0.9 ^d
Protocatechuic acid	1813	18.6± 0.6 ^a	19.5± 0.5 ^a	21.2± 0.8 ^b	23.3± 0.5 ^c	24.9± 0.8 ^d	25.3± 0.9 ^d	26.1± 0.5 ^d
Quinic acid	1843	22.5± 0.8 ^a	24.3± 0.9 ^a	27.8± 0.8 ^b	28.1± 0.7 ^b	29.8± 0.9 ^{b, c, d}	30.5± 0.6 ^{c, d}	30.4± 0.7 ^d
Fructose	1862	66.5± 1.4 ^a	70.1± 0.9 ^b	78.3± 1.0 ^c	83.1± 1.3 ^d	85.0± 1.0 ^d	88.2± 1.1 ^e	90.7± 0.9 ^e
Galactose	1884	62.3± 0.8 ^a	66.7± 0.9 ^b	69.2± 1.1 ^c	77.9± 0.9 ^d	79.3± 0.8 ^{d, e}	80.4± 0.9 ^{d, e}	80.8± 0.8 ^e
Syringic acid	1888	16.1± 0.6 ^a	17.2± 0.7 ^a	19.5± 0.8 ^b	20.2± 0.8 ^{b, c}	19.1± 0.7 ^b	20.5± 0.6 ^{b, c}	21.4± 0.7 ^c
Glucose	1896	169.0± 1.9 ^a	181.5± 1.4 ^b	208.4± 1.5 ^c	211.3± 1.8 ^c	219.4± 1.9 ^d	215.8± 1.4 ^e	218.7± 1.5 ^{d, e}
Glucitol	1930	60.9± 0.8 ^a	65.4± 0.7 ^b	66.9± 0.5 ^c	76.1± 0.9 ^d	77.8± 0.7 ^d	75.9± 0.9 ^d	78.3± 0.8 ^d
Gluconic acid	1991	36.8± 0.9 ^a	37.9± 0.8 ^a	41.2± 0.7 ^b	46.0± 0.7 ^c	45.8± 0.8 ^c	46.9± 1.0 ^c	47.8± 1.1 ^c
Palmitic acid	2039	54.7± 1.0 ^a	59.8± 1.1 ^b	63.5± 1.0 ^c	68.4± 0.9 ^d	66.3± 0.8 ^d	67.5± 0.9 ^d	67.6± 0.7 ^d
Glucaric acid	2013	27.7± 0.7 ^a	28.5± 0.8 ^{a, b}	30.8± 0.8 ^b	34.6± 0.9 ^c	35.7± 1.0 ^c	33.9± 1.1 ^c	36.8± 1.0 ^c

Myo-Inositol	2090	8.4± 0.6 ^a	8.9± 0.5 ^{a, b}	9.2± 0.6 ^{a, b, c}	10.5± 0.7 ^{b, c}	10.9± 0.5 ^c	9.9± 0.8 ^{a, b, c}	10.8± 0.6 ^{b, c}
Stearic acid	2132	32.7± 1.1 ^a	31.5± 1.2 ^a	35.7± 1.3 ^b	40.9± 1.0 ^c	42.4± 0.8 ^{c, d}	43.1± 0.9 ^d	43.8± 0.9 ^d
Caffeic acid	2140	14.5± 0.9 ^a	15.2± 0.7 ^a	16.9± 0.8 ^{a, b}	18.2± 0.8 ^{b, c}	18.9± 0.5 ^c	18.5± 0.7 ^c	19.3± 0.6 ^c
Linoleic acid	2209	48.3± 1.0 ^a	51.9± 0.8 ^b	55.5± 0.9 ^c	60.3± 1.1 ^d	62.5± 1.0 ^{d, e}	61.9± 1.1 ^{d, e}	64.1± 1.0 ^e
α-Linolenic acid	2217	23.9± 0.8 ^a	24.8± 0.7 ^a	28.1± 0.4 ^b	29.8± 0.9 ^c	31.2± 0.8 ^{c, d}	32.4± 0.7 ^d	30.3± 0.8 ^{c, d}
Sucrose	2649	63.4± 1.1 ^a	66.8± 1.0 ^b	68.9± 1.1 ^b	79.2± 1.0 ^c	77.8± 1.2 ^c	78.9± 1.1 ^c	79.9± 1.0 ^c
Turanose	2742	35.1± 1.3 ^a	39.8± 1.0 ^b	42.7± 1.2 ^c	43.9± 1.1 ^c	42.3± 1.2 ^c	41.9± 1.0 ^{b, c}	40.8± 1.1 ^{b, c}
Stigmasterol	3315	12.3± 0.7 ^a	14.1± 0.8 ^{a, b}	13.3± 0.9 ^{a, b}	15.4± 0.8 ^{b, c}	16.0± 0.7 ^c	15.8± 0.9 ^c	16.7± 0.8 ^c
β-Sitosterol	3355	11.7± 0.8 ^a	12.9± 0.7 ^{a, b}	12.7± 0.5 ^a	14.6± 0.7 ^{b, c}	15.7± 0.8 ^c	15.4± 0.7 ^c	15.8± 0.8 ^c

RI: relative index (Kovats retention index)

TIC: total ion current

The results are presented as mean ± SD (n=3)

a, b, c, d, e, f, g Values with different letters in a row are statistically different (Tukey's HSD test, $p < 0.05$)

Table 5. Polar volatile (aroma) substances in control and aromatized wines

Compound	RI	C	W1	W2	W3	W4	W5	W6
		% of TIC						
Alcohols								
Propan-1-ol	599	0.36± 0.08 ^a	0.35± 0.05 ^a	0.36± 0.06 ^a	0.37± 0.07 ^a	0.38± 0.05 ^a	0.38± 0.04 ^a	0.39± 0.08 ^a
Butan-1-ol	660	0.30± 0.08 ^a	0.30± 0.07 ^a	0.31± 0.04 ^a	0.31± 0.05 ^a	0.30± 0.06 ^a	0.30± 0.06 ^a	0.30± 0.05 ^a
Pentan-1-ol	768	0.40± 0.04 ^a	0.41± 0.02 ^a	0.40± 0.04 ^a	0.40± 0.05 ^a	0.41± 0.03 ^a	0.42± 0.04 ^a	0.42± 0.02 ^a
Hexan-1-ol	867	1.80± 0.10 ^a	1.79± 0.10 ^a	1.84± 0.09 ^a	1.85± 0.09 ^a	1.86± 0.07 ^a	1.86± 0.08 ^a	1.87± 0.06 ^a
Heptan-1-ol	912	0.48± 0.07 ^a	0.49± 0.06 ^a	0.49± 0.05 ^a	0.50± 0.05 ^a	0.50± 0.07 ^a	0.50± 0.06 ^a	0.50± 0.05 ^a
Octan-1-ol	993	0.60± 0.06 ^a	0.59± 0.06 ^a	0.61± 0.07 ^a	0.61± 0.06 ^a	0.61± 0.06 ^a	0.61± 0.07 ^a	0.62± 0.06 ^a
Nonan-1-ol	1170	0.21± 0.01 ^a	0.21± 0.01 ^a	0.21± 0.01 ^a	0.21± 0.02 ^a	0.21± 0.03 ^a	0.22± 0.02 ^a	0.23± 0.03 ^a
Decan-1-ol	1272	0.25± 0.04 ^a	0.26± 0.03 ^a	0.27± 0.04 ^a	0.26± 0.03 ^a	0.27± 0.04 ^a	0.27± 0.04 ^a	0.28± 0.06 ^a
Acids								
Acetic acid	640	0.41± 0.06 ^a	0.41± 0.05 ^a	0.42± 0.06 ^a	0.42± 0.07 ^a	0.41± 0.05 ^a	0.42± 0.06 ^a	0.43± 0.04 ^a

Butanoic acid	785	0.32± 0.07 ^a	0.31± 0.06 ^a	0.31± 0.05 ^a	0.33± 0.04 ^a	0.33± 0.04 ^a	0.33± 0.05 ^a	0.33± 0.06 ^a
Octanoic acid	1192	5.88± 0.09 ^a	5.89± 0.08 ^a	5.95± 0.06 ^a	6.07± 0.09 ^a	6.08± 0.08 ^a	6.11± 0.05 ^a	6.13± 0.04 ^a
Nonanoic acid	1281	0.76± 0.10 ^a	0.77± 0.08 ^a	0.78± 0.06 ^a	0.78± 0.09 ^a	0.79± 0.07 ^a	0.78± 0.08 ^a	0.79± 0.07 ^a
Decanoic acid	1388	6.90± 0.11 ^a	6.95± 0.08 ^a	7.03± 0.09 ^a	7.12± 0.12 ^a	7.09± 0.09 ^a	7.13± 0.06 ^a	7.15± 0.05 ^a
Dodecanoic acid	1573	1.48± 0.08 ^a	1.49± 0.05 ^a	1.50± 0.06 ^a	1.53± 0.09 ^a	1.54± 0.08 ^a	1.55± 0.07 ^a	1.56± 0.05 ^a
Tetradecanoic acid	1774	0.30± 0.06 ^a	0.30± 0.04 ^a	0.31± 0.03 ^a	0.31± 0.04 ^a	0.32± 0.05 ^a	0.32± 0.04 ^a	0.33± 0.03 ^a
Aldehydes								
Acetaldehyde	400	0.27± 0.06 ^a	0.30± 0.05 ^a	0.30± 0.05 ^a	0.28± 0.05 ^a	0.34± 0.04 ^b	0.38± 0.06 ^c	0.42± 0.05 ^d
Hexanal	800	1.72± 0.09 ^a	1.73± 0.05 ^a	1.74± 0.08 ^a	1.77± 0.10 ^a	1.78± 0.09 ^a	1.79± 0.07 ^a	1.78± 0.08 ^a
Decanal	1205	0.87± 0.08 ^a	0.87± 0.07 ^a	0.88± 0.06 ^a	0.90± 0.09 ^a	0.90± 0.07 ^a	0.91± 0.06 ^a	0.92± 0.07 ^a
Hydrocarbons								
Hexadecane	1600	2.81± 0.10 ^a	2.83± 0.09 ^a	2.86± 0.08 ^a	2.91± 0.11 ^a	2.92± 0.08 ^a	2.93± 0.09 ^a	2.95± 0.10 ^a
Octadecane	1800	3.34± 0.11 ^a	3.36± 0.08 ^a	3.41± 0.09 ^a	3.45± 0.12 ^a	3.44± 0.09 ^a	3.46± 0.10 ^a	3.48± 0.09 ^a
Nonadecane	1900	2.68± 0.09 ^a	2.69± 0.07 ^a	2.71± 0.08 ^a	2.76± 0.14 ^a	2.77± 0.08 ^a	2.75± 0.10 ^a	2.79± 0.08 ^a
Eicosane	2000	2.01± 0.12 ^a	2.02± 0.08 ^a	2.05± 0.06 ^a	2.08± 0.09 ^a	2.08± 0.09 ^a	2.10± 0.08 ^a	2.12± 0.07 ^a
Heneicosane	2100	2.10± 0.10 ^a	2.11± 0.08 ^a	2.15± 0.07 ^a	2.17± 0.09 ^a	2.18± 0.08 ^a	2.19± 0.09 ^a	2.22± 0.10 ^a
Docosane	2200	1.76± 0.08 ^a	1.78± 0.06 ^a	1.77± 0.09 ^a	1.82± 0.11 ^a	1.85± 0.10 ^a	1.87± 0.08 ^a	1.89± 0.11 ^a
Terpenes								
Linalool	1097	1.53± 0.10 ^a	1.54± 0.09 ^a	1.57± 0.08 ^a	1.58± 0.14 ^a	1.63± 0.09 ^a	1.69± 0.09 ^a	1.75± 0.08 ^a
Phenethyl alcohol	1110	nd	1.48± 0.11 ^a	1.95± 0.08 ^b	2.18± 0.14 ^b	2.68± 0.11 ^c	2.99± 0.10 ^d	3.44± 0.12 ^c
Cis-Rose oxide	1112	0.08± 0.05 ^a	0.49± 0.11 ^b	0.57± 0.10 ^b	0.69± 0.13 ^{b,c}	0.86± 0.14 ^c	1.24± 0.09 ^d	1.68± 0.08 ^c
Trans-Rose oxide	1127	0.10± 0.06 ^a	0.57± 0.05 ^b	0.61± 0.08 ^b	0.59± 0.07 ^b	0.89± 0.11 ^b	1.11± 0.09 ^c	1.79± 0.08 ^d
β-Citronellol	1228	nd	0.12± 0.02 ^a	0.15± 0.03 ^{a,b}	0.16± 0.01 ^{a,b}	0.18± 0.02 ^b	0.19± 0.01 ^b	0.25± 0.02 ^c
Geraniol	1255	nd	0.63± 0.11 ^a	0.69± 0.10 ^a	0.78± 0.09 ^a	1.25± 0.11 ^b	1.68± 0.12 ^c	2.19± 0.14 ^d
Eugenol	1356	2.10± 0.09 ^a	2.15± 0.10 ^a	2.18± 0.07 ^a	2.17± 0.08 ^a	2.19± 0.09 ^a	2.22± 0.08 ^a	2.28± 0.08 ^a
β-Bourbonene	1383	1.26± 0.11 ^a	1.27± 0.09 ^a	1.29± 0.08 ^a	1.30± 0.10 ^a	1.33± 0.07 ^a	1.34± 0.06 ^a	1.38± 0.05 ^a

β-Elemene	1390	2.54± 0.12 ^a	2.57± 0.09 ^a	2.63± 0.10 ^a	2.62± 0.08 ^a	2.64± 0.06 ^a	2.66± 0.05 ^a	2.69± 0.04 ^a
β-Caryophyllene	1419	2.93± 0.06 ^a	2.95± 0.07 ^{a,b}	2.98± 0.08 ^{a,b}	3.02± 0.09 ^{a,b}	3.03± 0.08 ^{a,b}	3.08± 0.06 ^{a,b}	3.10± 0.07 ^b
β-Cubebene	1389	0.76± 0.05 ^a	0.76± 0.07 ^a	0.77± 0.08 ^a	0.78± 0.10 ^a	0.80± 0.05 ^a	0.85± 0.04 ^a	0.89± 0.03 ^a
α-Guaiene	1438	1.08± 0.11 ^a	1.08± 0.10 ^a	1.09± 0.06 ^a	1.11± 0.10 ^a	1.15± 0.12 ^a	1.18± 0.07 ^a	1.22± 0.06 ^a
α-Humulene	1454	1.51± 0.09 ^a	1.53± 0.08 ^a	1.55± 0.08 ^a	1.56± 0.08 ^a	1.55± 0.07 ^a	1.59± 0.08 ^a	1.67± 0.07 ^a
(Z)-β-Farnesene	1459	1.81± 0.08 ^a	1.82± 0.09 ^a	1.86± 0.07 ^a	1.87± 0.09 ^a	1.96± 0.08 ^{a,b}	2.05± 0.06 ^b	2.15± 0.05 ^b
Germacrene D	1479	3.18± 0.14 ^a	3.20± 0.08 ^a	3.24± 0.09 ^a	3.28± 0.11 ^a	3.29± 0.10 ^a	3.33± 0.09 ^a	3.39± 0.08 ^a
δ-Guaiene	1508	1.88± 0.10 ^a	1.89± 0.09 ^a	1.92± 0.07 ^a	1.94± 0.12 ^a	1.95± 0.11 ^a	1.99± 0.08 ^a	2.12± 0.09 ^a
δ-Cadinene	1524	2.42± 0.10 ^a	2.45± 0.08 ^a	2.49± 0.06 ^a	2.50± 0.09 ^a	2.54± 0.08 ^a	2.59± 0.08 ^a	2.66± 0.07 ^a

RI: relative index (Kovats retention index); TIC: total ion current; nd – not determined; The results are presented as mean \pm SD (n=3)

^{a, b, c, d, e} Values with different letters in a row are statistically different (Tukey's HSD test, $p < 0.05$)

The major primary acids in wine grapes and subsequently in wines are tartaric, malic and depending from the grape variety but usually in minor amounts, citric acid (Bellman and Gallander, 1979). During the winemaking process and mainly fermentation, lactic, succinic, acetic and other acids could be formed and they play significant role in the final wine quality. The amounts of tentatively determined tartaric and malic acid are comparable for all variants of aromatized vines with control rosé with slight significant increase in W5 and W6. Malic acid is an important precursor of lactic acid through malolactic fermentation and the lactic acid formed is giving milder acidic taste (Bellman and Gallander, 1979). The amount of lactic acid found in all the variants is comparable which suggested that the added rose wastes of grape must did not influenced substantially the fermentation process.

A total of 41 volatile substances were tentatively detected (alcohols, acids, aldehydes, hydrocarbons and terpenes). Significant effect on the aroma substances formation/extraction in the aromatized wines and control were observed (Table 5) for β -citronellol, phenethyl alcohol, rose oxides, and geraniol. These substances

were absent or present in the control in low amounts and appeared in the aromatized wines due to addition of rose waste. Phenethyl alcohol is among the compounds which contribute significantly to the favorable aroma of white and rosé wines (Cabaroglu and Canbas, 2002). β -caryophyllene, β -citronellol, phenethyl alcohol, rose oxides, and geraniol increased significantly and distinctive rose aroma in W2-W6 variants was sensed. The amounts of alcohols increased in the aromatized wines compared to control but this increase was insignificant. The amount of acetaldehyde increased significantly for W4-W6 variants which confirm the GC-FID analysis results.

3.2. Sensory analysis of aromatized wines

An important attribute of every new or modified food system is the consumers' opinion and for this reason in the subsequent experiments sensory analysis of aromatized wines was conducted (Figure 1). The results of the sensory tests revealed most of the panelists preferred W1 and W2 variants as wines with characteristics closer to the control rosé. This could be explained with the more traditionally oriented taste of the Bulgarian consumers

concerning wines. The variants W3 to W6 were characterized with more pronounced rose aftertaste, the grassy nuances became more intense, the bitterness increased (along with the astringency, although astringency was not included in the indicators of the sensory analysis but most of the panelists expressed such sensations), as well as the flowery nuances increased. The W5 and W6 were disliked by most of the panelists (Overall acceptability 2.1 ± 0.8 and 2.0 ± 0.8 , respectively, compared with 6.1 ± 1.2 for the control).

In general the panelists divided wines in three groups: 1). Control, W1 and W2; 2). W3 and W4; and 3). W5 and W6. The group one was preferred mostly by the traditionally oriented consumers. It is interesting to note the opinion of some of the consumers towards group 3: they gave highest marks to these variants based on their personal preferences for aromatized (especially with rose notes) low-alcoholic beverages.

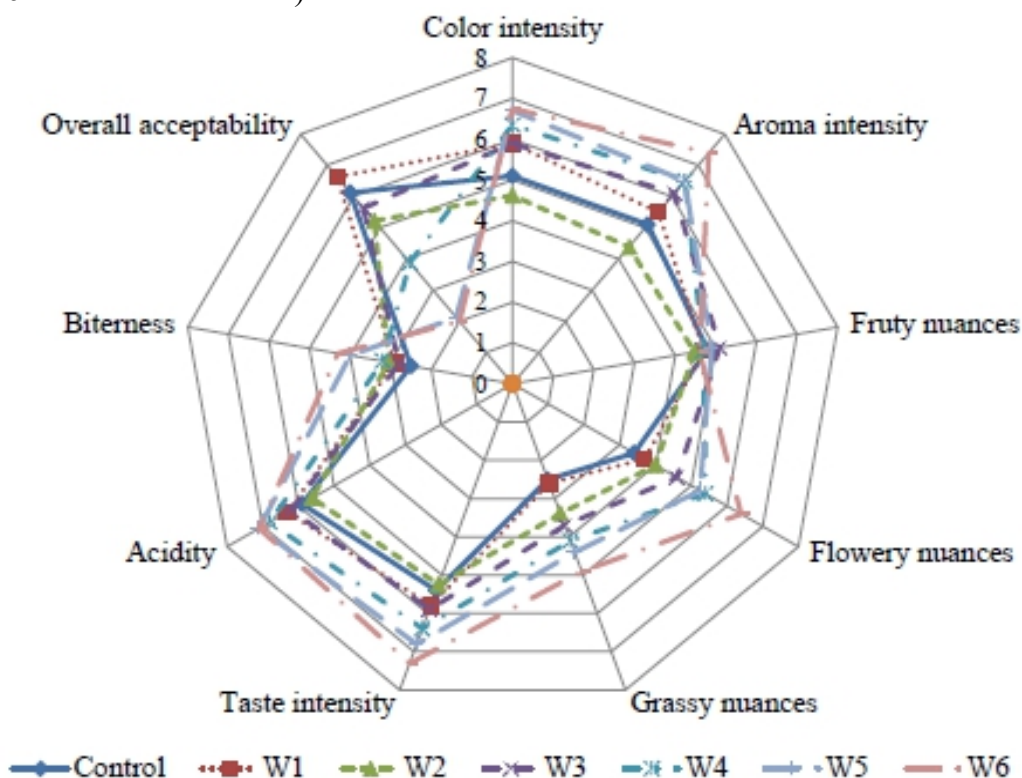


Figure 1. Sensory profile of wines

4. Conclusions

The present work explored the possibility for preparation and managing the quality of aromatized wines with addition of essential rose oil industry waste during grape must fermentation. To the best of our knowledge this is the first attempt for manufacturing of aromatized wines with addition of essential rose oil industry waste and the first experimental data for GC-MS profiling of Mavrud rosé. Control rosé and six variants with addition of different amounts of dry rose wastes (from 0.05% to 2%)

were obtained. The aromatized wines and the control had alcoholic content in the $14.4\text{--}14.6\pm 0.2\%$ range and $\text{pH } 3.43\text{--}3.59\pm 0.1$. The polyphenol content increased significantly from 355.01 ± 10.14 to $576.08\pm 12.08 \mu\text{mol GAE L}^{-1}$ for the control and W6, respectively. The higher amounts of phenolic acids (mainly gallic acid and 3,4-dihydroxy benzoic acid: from $1.2\pm 0.9 \text{ mg L}^{-1}$ and $5.4\pm 0.2 \text{ mg L}^{-1}$ in the control to 25.9 ± 0.9 and $65.1\pm 1.1 \text{ mg L}^{-1}$ in W6, respectively) is also related to increase in the antioxidant capacity of aromatized wines from

W1 to W6 and this could be explained with the higher amounts of total polyphenols extracted from the rose waste but not the anthocyanins (no significant difference in the TMA amounts). The aromatized wines and the control rosé had a total amounts of fusels in the 117.4-129.0±1.5 mg L⁻¹ range and it could be concluded that addition of rose waste during fermentation did not affect negatively formation of higher alcohols. β-caryophyllene, β-citronellol, phenethyl alcohol, rose oxides, and geraniol increased significantly and rose aroma in W1-W6 was achieved. The sensory analysis revealed W1 and W2 (overall acceptability 6.6±1.0 and 5.9±0.9, respectively) were considered with closer characteristics to control wine (overall acceptability 6.1±1.2) and more appropriate for consumption by the consumers. The overall interpretation of experimental data suggested that added rose wastes in the grape must during its fermentation, did not influenced substantially the fermentation process. The results of the present study confirmed the main hypothesis that rose oil industry by-products successfully could be utilized for preparation of aromatized wine and contributed for augmentation of total polyphenol content, antioxidant capacity, and new aroma profile of the final product was obtained.

5. References

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