Volatile profile of white wines produced from grape varieties cultivated under the soil-climatic conditions of Kyustendil, Southwestern Bulgaria

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Abstract

Introduction. The aim of this study is to define the volatile composition of white wines from Slava, Droujba and Tamyanka grapevine varieties, grown in the terroir conditions of the town of Kyustendil, Southwestern Bulgaria.

Materials and methods. White wines from three varieties and several harvests were studied – Slava (three harvests – 2021, 2023 and 2024), Droujba (four harvests – 2021, 2022, 2023 and 2024), Tamyanka (three harvests – 2021, 2022 and 2024). Volatile compounds were analyzed by gas chromatography (GC-FID). Climatic characterization was performed for the study period. Descriptive statistics were applied to determine mean values and standard deviation of identified main volatile compounds.

Results and discussion. The highest amounts of total volatile compounds were found in wines from the last studied harvest (2024) – Slava (1879.92 mg/l), Droujba (1539.40 mg/l) and Tamyanka (1762.51 mg/l). The main aldehyde was acetaldehyde. Its levels in almost all studied wines were at the optimum for positive influence (up to 240.00 mg/l). The highest total content of higher alcohols was found in wines of Slava variety, where they ranged from 101.04 mg/l (harvest 2023) to 597.75 mg/l (harvest 2024). The main identified higher alcohols were 2-methyl-1-butanol, 3-methyl-1-butanol (the alcohol with the highest concentration in all experimental variants) and 1-butanol with the first two being identified in all the wines studied. The highest content of 3-methyl-1-butanol was found in Slava wines. In them, it varied between harvests from 42.95 mg/l (2023) to 250.63 mg/l (2024). In Droujba wine, this higher alcohol varied from 33.87 mg/l (2023) to 124.00 mg/l (2022), and in Tamyanka, a variation from 61.60 mg/l (2024) to 97.80 mg/l (2022) was found. The main ester was ethyl acetate. The highest total terpene content was found in the two harvests of Slava wines – 2021 (1.92 mg/l) and 2024 (1.45 mg/l). Slava also showed the greatest species diversity of terpenes with 5 individual representatives identified – linalool oxide, linalool, α-terpineol, β-citronellol and geraniol. Methanol was identified in all studied wines in normal concentrations, which ranged in Droujba - from 0.92 mg/l (2023) to 50.27 mg/l (2021), in Slava - from 1.90 mg/l (2023) to 60.23 mg/l (2021) and in Tamyanka - from 6.15 mg/l (2022) to 98.92 mg/l (2021).

Conclusions. The conducted comprehensive study proved that the terroir of the town of Kyustendil is promising for growing the studied varieties, providing favorable conditions for the development of their potential, which was reflected in the production of white wines with optimal and complex aromatic quality.

Introduction

Studies on the wines volatile profile determine chemical components of the composition, which are an indicator of quality. The accumulation of volatile compounds is strongly dependent from the terroir of the region where the vines are grown (Kazimova et al., 2024; Ostapenko, 2016). According to Clingeleffer (2014), the concept of terroir is based on the complex interactions between biological and physical environmental factors, specific to a particular region, with the additional participation of the applied agrotechnical measures. Thus, the terroir influence determines specific characteristics of grapes and wine, directly related to the region. Han et al. (2022) analyzed the volatile profiles of 12 white wines from various regions (France, Germany, Italy, New Zealand, Spain, USA) and vintages (2015–2018), identifying 33 volatile compounds, with the highest concentrations found for acetaldehyde (16.00–57.00 mg/L), 1-hexanol (3.75–23.88 µg/L), isoamyl acetate (1.86–39.70 µg/L), hexyl acetate (1.21–10.89 µg/L), phenylethanol (16.92–77.73 µg/L), octanoic acid (4.32–25.41 µg/L), ethyl octanoate (11.60–142.47 µg/L), and ethyl isopentyl succinate (13.01–100.02 µg/L). Bayram and Kayalar (2018) determined the volatile composition of white wines of the Narince variety grown in two different regions of Turkey – Emiseyit and Erbaa and found that wines from the second studied region accumulated higher amounts of volatile compounds (205605.32 µg/l) compared to those from the first one (179547.85 µg/l). Vilanova et al. (2013) identified 46 volatile compounds in white wines from the varieties Albariño, Treixadura, Torrones, Blanco lexitimo and Loureira, grown in Northwestern Spain and 20 of the identified volatile compounds varying significantly between variants and being determinants of the so-called "varietal aroma". Mikulikova et al. (2009) determined the volatile composition of 29 white wines (harvests 2003 – 2006) of the varieties Riesling, Pinot Gris, Sauvignon Blanc, Gewürztraminer and Grüner Valtiner from different regions of the Czech Republic and found that the highest content was presented by the higher alcohols 3-methyl-1-butanol (214.00±35.00 mg/l – 257.00±15.00 mg/l), followed by the ester ethyl lactate (17.80±6.90 mg/l – 29.80±5.60 mg/l), the aromatic alcohol 2-phenylethanol (5.80±1.20 mg/l – 19.20±8.90 mg/l) and octanoic acid (4.80±0.90 mg/l – 11.60±4.40 mg/l). The team (Mikulikova et al., 2009) identified 5 terpenes in the analyzed white wines – linalool, terpineol, citronellol, geraniol and nerol, with linalool and terpineol being present in the highest levels. Kalbach et al. (2024) studied the volatile profile of 5 white wines (harvest 2018) of the Riesling, Gewürztraminer, Sémillon, Sauvignon Blanc and Chardonnay varieties from the Campanha Gaúcha region, Brazil and in the higher alcohols fraction they found a quantitative dominance of 1-hexanol (897.30 – 1316.50 mg/l), 3-methyl-1-butanol (118.90 – 157.30 mg/l) and 1-propanol (21.30 – 43.50 mg/l); The dominant esters were isoamyl acetate (646.00 – 3000.90 mg/l), diethyl succinate (573.70 – 817.30 mg/l), ethyl decanoate (468.80 – 1177.80 mg/l), ethyl octanoate (238.70 – 1860.00 mg/l), ethyl hexanoate (346.90 – 1209.40 mg/l) and ethyl butanoate (332.30 – 720.80 mg/l). Barrio-Galán et al. (2021) investigated the volatile profile of white wines (varieties – Verdejo, Sauvignon Blanc, Godello, Malvasia castellana and Albillo) from several PDO (Protected Designation of Origin) regions of Spain (Rueda, Cigales, Toro, Ribera del Duero, Bierzo) and found that wines from the Ribera del Duero and Bierzo regions accumulated high levels of terpenes, while those from Cigales, Toro and Rueda were characterized by high concentrations of alcohol acetates and ethyl esters. Weldegegris et al. (2011) determined the volatile content of white wines from South Africa, obtained from the Sauvignon Blanc, Chardonnay and Pinotage varieties, harvest 2005, from the Paarl, Stellenbosch, Worcester, Robertson, Olifants River and Swartland regions and found that the quantitatively dominant higher alcohols were isoamyl alcohol (3-methyl-1-butanol) (123.00±14.5 mg/l – 133.00±29.20 mg/l), 1-propanol (17.70±13.10 mg/l – 31.80±18.30 mg/l) and isobutanol (2-butanol) (23.20±14.50 mg/l – 30.00±10.40 mg/l), and from the ester fraction, ethyl acetate was found in the highest concentration (115.00±29.50

mg/l – 130.00±23.60 mg/l). Piras et al. (2020) studied the volatile profile of three white Portuguese wines from the Malvasia, Verdelho and Galego Dourado varieties (two harvests – 2017 and 2018) and found that the higher alcohols were quantitatively dominated by 3-methyl-1-butanol (isoamyl alcohol), which ranged from 46.035±6.510 mg/l to 168.256±0.706 mg/l and phenylethyl alcohol (phenyl ethanol) with a quantitative variation from 16.634±0.10 mg/l to 47.700±2.229 mg/l; the ester fraction was dominated by ethyl lactate (2.330±0.040 mg/l – 32.908±6.573 mg/l) and monomethyl succinate (6.375±0.419 mg/l – 19.743±6.290 mg/l), and 4 terpenes have also been identified – linalool, hotrineol, 2-terpineol and 2,6-dimethyl-3,7-octadiene-2,6-diol. Gómez-Miguez et al. (2007) studied the volatile composition of white wines from the autochthonous Zalema variety from the Huelva region (Southern Spain) and found that the strongest aromatic influence was exerted by fatty acids and ethyl esters.

The aim of this study is to define the volatile composition of white wines from the Slava, Droujba and Tamyanka varieties, grown in the terroir conditions of the town of Kyustendil, Southwestern Bulgaria.

Materials and methods

Varieties, plantation and agrotechnical measures. The study was conducted during the period 2021-2024 in an experimental vineyard plantation of the Institute of Agriculture - Kyustendil, Bulgaria. The terrain is located in the eastern part of Kyustendil Valley with an altitude of 450 m. The soil in the experimental plot is a highly leached, medium-textured sandy clay, slightly to moderately stony cinnamon forest soil (Chromic Luvisol) with a neutral pH reaction. The objects of the study were white wines produced from the grape varieties Slava and Droujba, developed at the Institute of Viticulture and Enology, Pleven (IVE) (Roychev, 2012; Simeonov et al., 2017), and Tamyanka, an autochthonous grapevine variety belonging to the Oriental ecogeographical group (Figure 1).

A B C

Figure 1. Grapevine varieties for white wines:
A – Slava; B – Droujba; C – Tamyanka

The vines were planted in the spring of 2015, grafted onto the Berlandieri x Riparia SO4 rootstock and trained using the Guyot stem system. The planting distances were 2.50 m between rows and 1.30 m within the row. The rows were oriented east west, in accordance with the prevailing southwestern and western winds in the area. The load during the study period was realized with 18 winter buds per vine (3 scions x 2 winter buds + 1 fruiting cane x 12 buds).

Climatic characteristics and vinification. The climate in the region is moderately continental, characterized by cold winters and conditions for late spring frosts. The average annual temperature is 10.5ºC (multi-annual norm). The highest average monthly temperature is in July (21.8ºC), and the lowest in January (- 0.8ºC). The annual temperature amplitude is relatively large (22.4ºC) and is an indicator of a prevailing moderately continental climate (Table 1).

Table 1

Average monthly air temperature and monthly precipitation amounts in the Kyustendil region

Meteorological element	Month												Average
	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	
Average monthly air temperature, t, ºC	*Perennial norm for the Kyustendil region, 1904-2008.*												
	-0.8	1.2	6.0	11.2	15.4	19.1	21.8	21.2	12.2	11.2	6.2	0.9	10.5
	2009-2019												
	-0.5	2.6	6.3	11.1	15.4	19.1	21.5	21.1	16.6	10.4	6.1	1.0	10.9
Average monthly precipitation, mm	*Perennial norm for the Kyustendil region, 1904-200*												
	43	38	36	53	65	64	60	37	42	59	57	54	608
	2009-2019												
	44	41	54	43	57	61	49	32	38	56	44	47	566

During the study period, the main elements of the climate in the Kyustendil region were measured, which were directly related to the development of the vines and the quality of the grapes. The absolute minimum air temperature during the complete dormancy of the plants, in two of the years, decreased to the critical values for the regenerative organs (-18.0ºC), but due to the interspecific origin of the studied varieties, no damage to the buds in the winter eyes was recorded (Ivanov et al., 2016). The average starting date of stable retention of the air temperature above 10.0°C was April 11, which is 2 days later than the multi-annual norm for the period 1956-2020 (Table 1). In autumn, the values of the average daily temperature of +10.0°C were maintained on average until October 29. The duration of the period between the two dates was 197 days with a total temperature sum of 3530.0 °C

The average multi-year date of the last spring frost in Kyustendil was April 8. According to this criterion, in 82.0% of cases, the danger of late spring frosts in the region has passed. The average monthly temperature in July, which is decisive for the technological qualities of grapes, was 22.9 ºC. The established value was 2.0 ºC higher than that established for the period 1956 - 2020. The last two years of the study (2023 and 2024) were the warmest since meteorological measurements have been conducted in the Kyustendil region (1904). In 2024, 46 days with absolute maximum air temperatures between 35.1-40.0ºC and 6 days with above 40.0ºC were recorded. For comparison, the average values of these indicators for the period 1904-2020 are 6 and 0.3 days respectively. The hydrothermal coefficient (HTC) reflecting the heat and moisture security for June, July and August was on average 0.96, which characterized the period as well-moistened (Table 2).

Table 2

Agroclimatic parameters for the period 2021–2024

Kyustendil, year	t > 10ºC		Duration of period in days		Total temperature sum, t > 10ºC	Average air temperature for July, ºC	Average air temperature for August ,°C	Hydrothermal coefficient (HTC) for VI - VIII (months)	Precipitation in June, July and August	Absolute minimum air temperature (average), °C
	Average start date	Average end date	t > 10ºC	Frost-free						
1956-2020*	08.IV	23.X	198	192	3391	20.9	21.1	0.77	146.0	-16.7
2021	25.IV	23.X	182	179	3153	21.2	22.2	0.38	72.9	-18.0
2022	21.IV	28.X	187	186	3197	21.5	19.0	1.23	228.8	-18.0
2023	11.IV	07.XI	208	206	3827	24.0	23.2	1.60	324.5	-13.0
2024	27.III	21.X	209	208	3943	25.0	23.9	0.61	135.6	-13.0
Average	11.IV.	29.X.	197	195	3530	22.9	22.1	0.96	190.5	-15.5

** Perennial norm for the Kyustendil region of Bulgaria*

The established values of the individual climate elements are a prerequisite for successful harvesting of early and mid-ripening grape varieties (Stoev, 1960)

30 kg of grapes were used for each variety. The grapes were vinified in the Experimental Wine Cellar of IVE according to a classic technological scheme for the obtaining of dry white wines (Yankov, 1992).

GC-FID analysis for determination of the volatile compounds. The content of the main volatile compounds was determined based on a stock standard solution prepared in accordance with IS 3752:2005 method. The method describes the preparation of a standard solution of one congener, but the preparation step was followed to prepare a solution of more compounds. The standard solution in the present study included the following compounds (purity > 99.0%): acetaldehyde, ethyl acetate, methanol, 2-propanol, isopropyl acetate, 1-propanol, 2-butanol, propyl acetate, 1-butanol, isobutyl acetate, ethyl butyrate, 2-butyl acetate, 2-methyl-1-butanol, 3-methyl-1-butanol, 4-methyl-2-pentanol, 1-pentanol, pentyl acetate, 1-hexanol, ethyl hexanoate, hexyl acetate, 1-heptanol, linalool oxide, dimethyl succinate, phenyl acetate, linalool, ethyl caprylate, 2-phenylethanol, α-terpineol, nerol, ß-citronellol, geraniol, ethyl decanoate. The prepared standard solution containing all compounds was injected in an amount of 2 µl into a gas chromatograph Varian 3900 (Varian Analytical Instruments, Walnut Creek, California, USA) with a capillary column VF max MS (30 m, 0.25 mm ID, DF= 0.25 µm), equipped with flame ionization detector (FID). The carrier gas was helium. Hydrogen to support combustion was supplied to the chromatograph via a hydrogen bottle. The injection was manual, using a microsyringe. The gas chromatographic determination parameters were: injector temperature – 220 °C, detector temperature – 250 °C, initial oven temperature – 35 °C/1 min retention, rise to 55 °C with a step of 2 °C/min for 11 min, rise to 230 °C with a step of 15 °C/min for 3 min. Total chromatography time – 25.67 min. After the retention times of the compounds in the standard solution were determined, the identification and quantification of the volatile compounds in the wines were conducted. The volatile composition was determined based on the injection of wine distillates. Samples were injected in an amount of 2 µl into a gas chromatograph and identification and quantification of volatile compounds was performed.

Statistical analysis. Descriptive statistics were applied, with standard deviation (± SD) determined as the mean value of all harvests for the compounds found in the highest amount. The program Excel (MS Office, Microsoft Corporation, USA) was used for the analysis. Student's t-test was used to compare the values between grape varieties and five main identified volatile compounds (2-methyl-1-butanol, 3-methyl-1-butanol, Ethyl acetate, Acetaldehyde, Methanol).

Results and discussion

The results obtained for the identified volatile compounds are presented in Table 3. Wines of the Slava variety from three harvests – 2021, 2023 and 2024, Droujba, which includes four consecutive harvests – 2021 – 2024 and Tamyanka, for which wines from three harvests – 2021, 2022 and 2024 were analyzed.

Table 3

Volatile compounds (mg/l) identified in white wines of Slava, Droujba and Tamyanka grapevine varieties within different studied harvests

Identified compounds, mg/l	White wines									
	Slava			Droujba				Tamyanka		
	Harvests			Harvests				Harvests		
	2021	2023	2024	2021	2022	2023	2024	2021	2022	2024
Ethyl alcohol, vol.%	10.81	11.10	11.37	11.94	12.43	11.50	12.50	11.18	12.90	12.65
Acetaldehyde	57.78	225.22	219.87	81.81	297.66	200.00	304.00	14.56	147.28	1011.00
Methanol	60.23	1.90	8.84	50.27	36.18	0.92	8.04	98.92	6.15	7.94
2-methyl-1-butanol	62.71	7.46	150.70	52.22	38.68	5.28	26.52	47.54	21.42	13.10
3-methyl-1-butanol	120.25	42.95	250.63	121.06	124.00	33.87	110.69	74.92	97.80	61.60
4-methyl-2-pentanol	ND	ND	11.82	ND	1.29	0.46	ND	ND	ND	ND
1-propanol	ND	ND	2.24	8.22	ND	ND	ND	ND	ND	ND
2-propanol	13.10	ND	2.22	9.88	ND	ND	3.03	ND	ND	8.50
1-butanol	ND	34.92	18.63	ND	33.99	24.49	33.80	14.92	6.70	ND
1-pentanol	ND	ND	9.13	ND	ND	ND	0.87	ND	ND	7.34
2-butanol	30.30	6.32	77.16	ND	ND	ND	25.28	32.16	ND	5.55
1-hexanol	ND	9.39	75.22	ND	ND	9.05	5.00	ND	ND	8.23
2-henylethanol	ND	ND	ND	ND	ND	ND	41.32	ND	ND	65.57
Total content of higher alcohols	**226.36**	**101.04**	**597.75**	**191.38**	**197.96**	**73.15**	**246.51**	**169.54**	**125.92**	**169.89**
Ethyl acetate	33.07	22.85	495.82	142.85	46.83	19.26	759.49	211.82	71.25	127.81
Isobutyl acetate	ND	ND	75.47	7.02	ND	ND	218.88	ND	211.61	16.94
Butyl acetate	ND	ND	15.81	ND	ND	ND	ND	ND	11.32	ND
Propyl acetate	ND	ND	5.17	ND	3.94	ND	ND	ND	ND	5.42

Identified compounds, mg/l	White wines									
	Slava			Droujba				Tamyanka		
	Harvests			Harvests				Harvests		
	2021	2023	2024	2021	2022	2023	2024	2021	2022	2024
Isopropyl acetate	ND	ND	4.91	24.79	ND	ND	2.48	ND	ND	0.98
Isopentyl acetate	ND	ND	ND	ND	ND	ND	ND	39.56	ND	ND
Pentyl acetate	ND	ND	ND	ND	ND	15.74	ND	12.25	ND	307.14
Phenyl acetate	ND	ND	179.72	ND	40.84	ND	ND	ND	ND	66.39
Ethyl caprylate	6.99	ND	ND	15.82	ND	ND	ND	33.78	ND	ND
Hexyl acetate	ND	ND	265.62	ND	ND	ND	ND	ND	310.34	ND
Dimethyl succinate	ND	ND	ND	ND	ND	ND	ND	ND	ND	48.25
Ethyl butyrate	ND	ND	9.49	ND	15.63	ND	ND	ND	19.52	ND
Ethyl decanoate	ND	ND	ND	ND	ND	35.49	ND	ND	ND	ND
Total content of esters	**40.06**	**22.85**	**1052.01**	**190.48**	**107.24**	**70.49**	**980.85**	**297.41**	**624.04**	**572.93**
Linalool oxide	0.84	ND	0.20	ND	ND	ND	ND	ND	ND	0.43
Linalool	ND	ND	0.23	ND	ND	ND	ND	ND	ND	ND
α-terpineol	ND	ND	0.27	ND	ND	ND	ND	ND	ND	0.32
Nerol	0.37	ND	ND	0.36	ND	0.69	ND	ND	0.10	ND
β – citronellol	0.16	ND	0.17	0.11	ND	ND	ND	ND	ND	ND
Geraniol	0.55	ND	0.58	0.50	0.15	ND	ND	ND	0.39	ND
Total content of terpenes	**1.92**	**0.00**	**1.45**	**0.97**	**0.15**	**0.69**	**0.00**	**0.00**	**0.49**	**0.75**
Total content	**386.35**	**351.01**	**1879.92**	**514.91**	**639.19**	**345.25**	**1539.40**	**580.43**	**903.88**	**1762.51**

**ND – Not detected*

Total volatile compounds, identified aldehydes and higher alcohols

It was found that the highest total content of volatile compounds was accumulated in the wines of the last harvest (2024). The quantitative variation of total volatile compounds in the wines of each individual variety between harvests was as follows: Slava (351.01 mg/l – 1879.92 mg/l), Droujba (345.25 mg/l – 1539.40 mg/l) and Tamyanka (580.43 mg/l – 1762.51 mg/l). According to Jackson (2020) the aromatic matrix of wines consists of over 1000 identified volatile compounds, which occupy average concentrations up to 800.00 – 1200.00 mg/l.

The aldehyde fraction was represented by acetaldehyde. This compound occupies 90% of all fatty aldehydes in wines by concentration (Chobanova, 2012). Its variation in the wines of the Slava variety ranged from 57.78 mg/l (harvest 2021) to 225.22 mg/l (harvest 2023), followed by those of the Droujba variety – 81.81 mg/l (harvest 2021) to 297.66 mg/l (harvest 2022), and Tamyanka showed a variation of the aldehyde from 14.56 mg/l (harvest 2021) to 1011.00 mg/l (harvest 2024). In dry white wines, aldehyde could vary from 7.00 to 240.00 mg/l, but could also reach 800.00 mg/l (Coetzee and du Toit, 2015; Lachenmeier and Sohnuis, 2008). Its formation is related to the yeasts activity, which synthesize it metabolically, but also to its chemical formation, which is related to oxidation processes (Nykanen, 1986). When the must is presulphated, before fermentation, the yeast synthesizes it in significant quantities, which increases its final concentration in the wine. At low concentrations (within the norm) it gives a pleasant fruity aroma, and above the permissible levels it forms a sharp, irritating and oxidized nuance (Guittin et al., 2025).

In the present study, high levels of this aldehyde were found in the variants of white wines of the Droujba variety - harvest 2022 (297.66 mg/l) and harvest 2024 (304.00 mg/l) and Tamyanka - harvest 2024 (1011.00 mg/l), where the amount of the aldehyde was very high, indicating a strong oxidative process or presulfited must in the initial phases of processing, before fermentation.

The total content of higher alcohols in the analyzed wines did not show any distinguishable disproportions. Their content was the highest in Slava wines - 101.04 mg/l (harvest 2023) - 597.75 mg/l (harvest 2024), followed by Droujba, where they varied from 73.15 mg/l (2023) to 246.51 mg/l (2024), and for this variety it was striking that the 2021 and 2022 harvests showed an almost similar total amount of higher alcohols. The lowest levels of this fraction were found in the wines of the Tamyanka variety, where the levels of higher alcohols between its harvests were extremely similar, and in the 2021 and 2024 harvests they were almost identical. The variation in this variety was from 125.92 mg/l (2022) to 169.89 mg/l (2024).

According to Chobanova (2012), the content of higher alcohols in white wines varied in the range of 150.00 – 400.00 mg/l. The data in the present study were in correlation with these levels, with only exception of the Slava wine variant from the 2024 harvest which accumulated a slightly higher amount (597.75 mg/l).

The main identified higher alcohols were 3-methyl-1-butanol, 2-methyl-1-butanol and 1-butanol, with the first two being identified in all the wines studied.

The highest content of 3-methyl-1-butanol was found in Slava wines. In them, it varied between harvests from 42.95 mg/l (2023) to 250.63 mg/l (2024). In Droujba wine, this higher alcohol varied from 33.87 mg/l (2023) to 124.00 mg/l (2022), and in Tamyanka, a variation from 61.60 mg/l (2024) to 97.80 mg/l (2022) was found. The data correlated with other studies that found it as a major component in various white wines from regions of the Czech Republic, Brazil, South Africa and Portugal with a variation from 46.03±6.51 mg/l to 257.00±15.00 mg/l (Mikulikova et al., 2009; Kalbach et al., 2024; Weldegegris et al., 2011; Piras et al., 2020). Characteristic aromas that this alcohol had the potential to impart to wines were green, grassy, alcoholic, cheesy (Costello et al., 2012; Genovese et al., 2007; Torreus et al., 2010).

The next major higher alcohol was 2-methyl-1-butanol. It was found in lower concentrations. It was identified in the highest amount in Slava wines, where it varied between harvests from 7.46 mg/l (2023) to 150.70 mg/l (2024). Its variation in Droujba wines ranged from 5.28 mg/l (2023) to 52.22 mg/l (2021), and in Tamyanka, it ranged from 13.10 mg/l (2024) to 47.54 mg/l (2021). This compound is a major part of the wine aromatic matrix and present in wines in concentrations from 12.00 mg/l to 311.00 mg/l (Haenel, 1989). Our data correlated with this range. It was found that 2-methyl-1-butanol could impart citrus, green and malty aromas (Karabagias et al., 2011; Nistor et al., 2017).

The third main higher alcohol was 1-butanol. It was identified in the wines of two of the Slava harvests – 2023 (34.92 mg/l) and 2024 (18.63 mg/l), in three of the Droujba harvests (2022 to 2024), where it ranged from 24.49 mg/l to 33.99 mg/l, and in two of the Tamyanka harvests – 2021 (14.92 mg/l) and 2022 (6.70 mg/l). It was characterized by a floral aromatic influence (Alises et al., 2023).

The remaining higher alcohols were not present in all the studied wines, but were identified in individual harvests, which proved the significant influence of the climatic factor in the formation of the wine aromatic complex. The highest species diversity of higher alcohols was recorded in very hot and dry vegetation (2024). 4-methyl-2-pentanol was identified in Slava's wine in the 2024 harvest variant (11.82 mg/l), in Droujba it was present in the 2022 harvest (1.29 mg/l) and 2023 (0.46 mg/l), and in Tamyanka's wines it was not identified; 1-propanol was identified in Slava's wines only in the 2024 harvest (2.24 mg/l), and in Droujba it was present only in the 2021 harvest (8.22 mg/l), while in Tamyanka it was not detected; 2-propanol was identified in two harvests of Slava – 2021 (13.10 mg/l) and 2024 (2.22 mg/l), in two harvests of Droujba – 2021 (9.88 mg/l) and 2024 (3.03 mg/l) and in one harvest of Tamyanka – 2024 (8.50 mg/l); 1-pentanol was found only in the 2024 harvest but in the wines of the all three varieties in concentrations respectively: Slava (9.13 mg/l), Droujba (0.87 mg/l) and Tamyanka (7.34 mg/l); 2-butanol was identified in all three Slava harvests, where it varied from 6.32 mg/l (2023) to 77.16 mg/l (2024), in Droujba it was found only in one harvest – 2024 (25.28 mg/l), and in Tamyanka it was present in two harvests – 2021 (32.16 mg/l) and 2024 (5.55 mg/l); 1-hexanol was identified in two of the Slava's harvests – 2023 (9.39 mg/l) and 2024 (75.22 mg/l), in two of the Droujba's harvests – 2023 (9.05 mg/l) and 2024 (5.00 mg/l) and in one of the Tamyanka's harvests – 2024 (8.23 mg/l); 2-phenylethanol – identified only in the 2024 harvest of Droujba (41.32 mg/l) and Tamyanka (65.57 mg/l).

Total ester content and identified individual representatives

The highest total ester levels were found in the 2024 harvest in all three studied wines. In Slava, esters varied between the three harvests from 22.85 mg/l (2023) to 1052.01 mg/l (2024). The variation in Droujba ranged from 70.49 mg/l (2023) to 980.85 mg/l (2024), and in Tamyanka the total ester content ranged from 297.41 mg/l (2021) to 624.04 mg/l (2022). According to Chobanova (2012), the total ester content in young wines varied from 200.00 to 500.00 mg/l. In the present study, the majority of the experimental variants covered this range, with the exceptions of Slava variants from the 2024 harvest (1052.01 mg/l), Druzhba variant from the 2024 harvest (980.85 mg/l) and Tamyanka variant from 2022 harvest (624.00 mg/l). The disproportion in the first two variants was due to the higher levels of ethyl acetate found, while in the third variant the increased total amount was due to the higher levels of hexyl acetate found.

The main ester that was identified in all the studied wines was ethyl acetate. High levels of this ester were found in two of the experimental variants – Slava – harvest 2024 (495.82 mg/l) and Droujba – harvest 2024 (759.49 mg/l). In the remaining wines, it was found in normal concentrations. In the other two harvests of Slava, it was present in an amount of 33.07 mg/l (2021) and 22.85 mg/l (2023), in Droujba it varied between the three harvests from 19.26 mg/l (2023) to 142.85 mg/l (2021), and in Tamyanka it varied from 71.25 mg/l (2022) to 211.82 mg/l (2021). According to Chobanova (2012), its concentration varies from 30.00 to 300.00 mg/l. The main characteristic of the ethyl acetate is its dual nature of aromatic influence. At concentrations up to 100.00 mg/l, it enhances the fruity nuances in the wine aroma, while above these values it can form aromas from the chemical group (Lasik-Kurdyś et al., 2018). Its presence in high concentrations indicates acidification processes. Its content in the three experimental variants listed above was above the permissible limits, which created the possibility of generating unpleasant aromatic notes in the wines. In the remaining studied variants, its concentration was in correlation with other studies – that of Miranda et al. (2017), who found a variation of ethyl acetate from 26.10±0.36 mg/l to 110.31±0.17 mg/l in young Madeira wines, the data were also in agreement with the study of Lasik-Kurdyś et al. (2018), who found ethyl acetate in young Chardonnay wines with a variation from 58.12±5.37 mg/l to 110.26±4.93 mg/l. It was found as the main ester in white wines from

South Africa, in which it varied from 115.00±29.50 mg/l to 130.00±23.60 mg/l (Weldegegris et al., 2011).

The remaining esters were found in separate variants and harvests. Isobutyl acetate was identified only in one harvest (2024) of Slava (75.47 mg/l), in two of the Droujba's harvests – 2021 (7.02 mg/l) and 2024 (218.88 mg/l) and in two of the Tamyanka's harvests – 2022 (211.61 mg/l) and 2024 (16.94 mg/l); Butyl acetate was found in one of the Slava's harvests – 2024 (15.81 mg/l) and in one of the Tamyanka's harvests – 2022 (11.32 mg/l); Propyl acetate was detected in one of the Slava's harvests – 2024 (5.17 mg/l), in one of the Droujba's harvests – 2022 (3.94 mg/l) and in one of the Tamyanka's harvests – 2024 (5.42 mg/l); Isopropyl acetate was detected only in the 2024 harvest (4.91 mg/l) of Slava, in the 2021 harvest (24.79 mg/l) and 2024 (2.48 mg/l) of Droujba, and also in the 2024 harvest (0.98 mg/l) of Tamyanka; Isopentyl acetate was detected only in the 2021 harvest (39.56 mg/l) of Tamyanka; Pentyl acetate was detected in the 2023 harvest (15.74 mg/l) of Droujba and in the 2021 (12.25 mg/l) and 2024 (307.14 mg/l) of Tamyanka; Phenyl acetate was detected in the 2024 harvest (179.72 mg/l) of Slava, the 2022 harvest (40.84 mg/l) of Droujba and the 2024 harvest (66.39 mg/l) of Tamyanka; Ethyl caprylate was identified only in one of the harvests of Slava – 2021 (6.99 mg/l) and in the 2021 harvests of Druzhba and Tamyanka in concentrations of 15.82 mg/l and 33.78 mg/l, respectively; Hexyl acetate was identified only in the 2024 harvest (265.62 mg/l) of Slava and in the 2022 harvest (310.34 mg/l) of Tamyanka; Diethyl succinate was identified only in the 2024 harvest (48.25 mg/l) of Tamyanka; Ethyl butyrate was identified in the 2024 harvest (9.49 mg/l) of Slava, in the 2022 harvests of Droujba (15.63 mg/l) and Tamyanka (19.52 mg/l); Ethyl decanoate was detected only in the 2023 harvest (35.49 mg/l) of Droujba.

Total terpene content, individual representatives, and methanol

Terpenes were not identified in all the wines studied. The highest total terpene content was found in Slava's wines, as in the 2023 harvest they were not present, in the 2021 harvest they were present in an amount of 1.92 mg/l, and in the last harvest (2024) their total content was 1.45 mg/l. Droujba's wines rank second in terms of concentration presence. Terpenes were not detected in them only in the 2024 harvest, while in the other three harvests their content varied from 0.15 mg/l (2022) to 0.97 mg/l (2021). Tamyanka showed the lowest levels of terpenes, as in the 2021 harvest they were not identified, while in the 2022 harvest they occupied an amount of 0.49 mg/l, and in the 2024 harvest – 0.75 mg/l.

Terpenes are a major factor in the aroma formation of Muscat varieties, where they occupy concentrations of up to 2.00 mg/l on average (Chobanova, 2012). Based on the terpene content, varieties can be classified as Muscat - with a total terpene content higher than 6.00 mg/l, aromatic non-Muscat, which contains terpenes in the range of 1.00 to 4.00 mg/l and neutral, in which terpenes are present in amounts lower than 1.00 mg/l (Mateo and Jiménez, 2000). Based on this, wines of the Slava variety could be categorized as aromatic non-Muscat, and those of Droujba and Tamyanka as neutral. Terpenes are not a fermentation product, but pass from the grapes to the wine after processing and fermentation. Celik et al. (2015) investigated the total terpene content of the white aromatic variety Muscat of Bornova from three different terroirs – Kemaliye, Halibeyli and Menderes from Izmir-Manisa province, Turkey, for two harvests (2013 and 2014) and found a variation in total terpene content between the three regions for the first harvest from 1.3 mg/l (Kemaliye) to 2.1 mg/l (Menderes), and for the second harvest – from 1.3 mg/l (Halibeyli) to 2.6 mg/l (Kemaliye). The data for Slava in our study correlated with this range.

Linalool oxide was identified in two of Slava's wines – harvest 2021 (0.84 mg/l) and harvest 2024 (0.20 mg/l) and in one of Tamyanka's harvest – 2024 (0.43 mg/l). Linalool was identified in only one of Slava's harvests – 2024 (0.23 mg/l). α-terpineol was identified in Slava's 2024 harvest (0.27 mg/l) and Tamyanka's 2024 harvest (0.32 mg/l). Nerol was identified in Slava's 2021 harvest (0.37 mg/l), Druzhba's 2021 (0.36 mg/l) and 2023 (0.69 mg/l) and Tamyanka's 2022 harvest (0.10 mg/l). β-citronellol was detected in the 2021 (0.16 mg/l) and 2024 (0.17 mg/l) harvests of Slava and in the 2021 (0.11) harvest of Droujba. Geraniol was detected in the 2021 (0.55 mg/l) and 2022 (0.15 mg/l) harvests of Droujba and in the 2022 (0.39 mg/l) harvest of Tamyanka. The wine of the Slava variety from the 2024 harvest was the richest in individual terpenes.

According to Resolution OENO 19/2004 of the OIV (International Organisation of Vine and Wine), the maximum permissible levels of methanol in white wines must be up to 250.00 mg/l. In practice, the formation of methanol is a normal process in the production of alcoholic beverages, which is due to the breakdown of fruit pectin by the pectolytic enzyme complex, also present in the fruit (Han and Du, 2022). All established levels of methanol in the studied white wines of the three varieties were well below the limit of its permissible amount. Thus, in the three harvests of Slava, the amounts of methanol varied from 1.90 mg/l (2023) to 60.23 mg/l (2021). In the four harvests of Droujba, the methanol variation occupied concentrations from 0.92 mg/l (2023) to 50.27 mg/l (2021), and in the three harvests of Tamyanka, methanol ranged from 6.15 mg/l (2022) to 98.92 mg/l (2021). Our data correlated with the established methanol levels in Australian white wines, where it varied from 40.00 – 120.00 mg/l (Hodson et al., 2017), in Turkish white wines from different regions – 30.50 – 121.40 mg/l (Cabaroglu, 2005), in Georgian wines, where it varied from 85.90 mg/l to 111.30 mg/l (Khomasuridze et al., 2005).

Average value and statistical variation of the main volatile compounds in the analyzed wines

The data for the mean value (Slava – three harvests, Droujba – four harvests and Tamyanka – three harvests) and standard deviation of the main identified compounds (2-methyl-1-butanol, 3-methyl-1-butanol, ethyl acetate, acetaldehyde and methanol) are presented in Table 4.

Table 4

Content of main identified volatile compounds in white wines (mg/l)

Identified compounds, mg/l	White wines from		
	Slava*	**Droujba****	**Tamyanka***
2-methyl-1-butanol	73.62±72.24	30.67±19.92	27.35±17.97
3-methyl-1-butanol	137.94±104.96	97.40 ±42.73	78.10 ±18.30
Ethyl acetate	183.91±270.16	242.10±348.96	136.92±70.73
Acetaldehyde	167.62±95.16	220.86±104.21	390.94 ±541.06
Methanol	23.65±31.86	23.85±23.27	37.67 ±53.05

** Data for three harvests, Average±SD*
*** Data for four harvests, Average±SD*
**** Student's t-test was used to compare the values between grape varieties for each of the compounds. No statistically significant differences were found (p>0.05).*

2-methyl-1-butanol was present (as an average amount of all studied harvests) in the highest concentration in Slava (73.62±72.24 mg/l). The other two wines – Droujba and Tamyanka showed very close average content of this higher alcohol, respectively 30.37±19.92 mg/l and 27.35±17.97 mg/l.

3-methyl-1-butanol, which was the higher alcohol with the highest concentration, showed an average value of all studied harvests, which is the highest (137.94±104.96 mg/l) again in Slava. In second place by average accumulation it was found in Droujba (97.40±42.73 mg/l), and its average levels were the lowest in Tamyanka (78.10±18.30 mg/l).

The highest average levels of ethyl acetate were established in the wine of the Droujba variety (242.10±348.96 mg/l), followed by Slava (183.91±270.16 mg/l), and in Tamyanka its levels were the lowest (136.92±70.73 mg/l). The large ranges of standard deviation of this compound in Slava and Droujba were due to its very high levels found in the last studied harvest – 2024.

The highest average levels of acetaldehyde were found in the wine of the Tamyanka variety (390.94±541.06 mg/l), followed by Droujba (220.86±104.21 mg/l) and the lowest, but optimal levels, were found in Slava (167.62±95.16 mg/l). Here too, a very high standard variation was reported in the wine of the Tamyanka variety, which was again due to a very high accumulated amount of acetaldehyde in the 2024 harvest (1011.00 mg/l).

In the case of methanol, the average levels for the wines of all three varieties were very close. The highest average amount was identified in Tamyanka (37.67±53.05 mg/l), and the other two wines showed almost similar average methanol concentration, respectively: Slava (23.65±31.86 mg/l) and Droujba (23.85±23.27 mg/l).

The average values of volatile compounds varied, since each harvest exerted the individual influence of many factors (soil, climate, agricultural techniques, cultivation technology, vinification technology, and yeast cultures used), which had complex and heterogeneous effects on the accumulation of each volatile compound in the wine.

Conclusions

The study demonstrated clear evidence of the influence of climatic conditions on the aromatic composition of white wines from the Kyustendil region. The 2024 harvest showed the highest total content of volatile compounds, confirming that hotter and drier seasons favor the quantitative and qualitative enrichment of wine aroma. This reflects the impact of recent climate changes marked by frequent droughts and elevated temperatures. Acetaldehyde levels were generally within acceptable limits, contributing positively to wine aroma, except in Droujba (2022 and 2024) and Tamyanka (2024) wines, where higher concentrations likely caused slight oxidative notes. The predominant higher alcohols were 2-methyl-1-butanol, 3-methyl-1-butanol, and 1-butanol, while ethyl acetate was the main ester. Excessive ethyl acetate levels in Slava (2024) and Druzhba (2024) may have produced acetic tones, whereas normal levels in other wines enhanced fruity notes. The Slava wine (2024) exhibited the most diverse terpene profile, with five identified compounds (linalool oxide, linalool, α-terpineol, β-citronellol, and geraniol), indicating that drought conditions promote terpene biosynthesis in grapevines. Statistical analysis of key volatiles (2-methyl-1-butanol, 3-methyl-1-butanol, ethyl acetate, acetaldehyde, and methanol) revealed considerable variability linked to the complex interaction of soil, climate, viticultural and vinification practices, and yeast strains.

Overall, the results confirm that the Kyustendil terroir provides favorable conditions for the studied varieties, supporting the production of white wines with complex and balanced aromatic profiles. The findings enrich current knowledge on terroir-specific effects and highlight the growing impact of climate change on grapevine metabolism and wine quality.

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