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ORIGINAL PAPER

Changes in selected chemical and sensory parameters during aging of red wines produced in Poland

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Abstract
The study of the chemical composition of wines is of continuing interest to researchers, despite extensive knowledge about it. Many analyses focus on the chemical transformations that take place in this beverage as it ages. This study aimed to analyze changes in parameters such as free SO_2 concentration, acidity, pH, the concentration of selected organic acids, total polyphenol concentration, and FRAP in five red wine varieties: Rondo, Regent, Cabernet Cortis, Acolon, and Zweigelt produced in Poland which were aged for 3 years. The study also included a sensory analysis of wines. The results indicated that, despite the relatively short aging time of the observed wine, the changes were significantly noticeable. The flavors of the wines, as well as their aromas, evolved, resulting in higher scores in sensory evaluation. Parameters such as free sulfur oxide (IV) concentration, antioxidant activity, and tartaric acid, acetic acid, and citric acid concentrations changed significantly. In addition, an analysis of the results was carried out using the PLS method, in which the relationships between the parameters determined were revealed. Research carried out indicates for the first time that Polish red wines have a relatively high potential for long aging.

Keywords Red wines · Aging of wine · Organic acids · Sensory properties of wine · Wine parameters

Introduction

When wine is bottled, its composition may change over time as a consequence of chemical reactions between its ingredients [1]. The further development of the wine depends not only on its initial composition at the end of its maturation, but also on the bottling process: concentration of dissolved oxygen and free sulfur dioxide, bottle fill height, and gas components in the headspace [2] and type of closure [3, 4]. Moreover, the conditions under which the wine is stored may also affect the effectiveness of the chemical changes that take place in it. The most important parameters in this regard include bottle position [3], room temperature [4, 5], lighting, and humidity [6]. Changes in the profile and concentrations of individual wine components during aging will in turn affect its organoleptic properties. There are many studies in the subject literature in which changes in the chemical, physical, and sensory properties of wine during the aging process have been described. The group of compounds that are most frequently studied in terms of changing profile and concentration during wine aging (also under different aging conditions) are polyphenols. These compounds are involved in shaping the quality of the wine through their influence on sensory properties: color, bitterness, flavor or astringency, aging behavior, and antioxidant activities [7]. Most polyphenols are highly unstable. During the aging and maturation of wine, the concentration of monomeric anthocyanins decreases, and more complex and stable pigments are formed, mainly including proanthocyanins and polymeric anthocyanins, resulting from the condensation of anthocyanins and/or flavan-3-ols directly or in reactions mediated by aldehydes. Along with changes in the profile and concentration of polyphenol compounds in wine, its

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color changes from a purple-red color in young red wines to a brick-red hue in aged ones [8]. The same process brings about changes in wine attributes such as astringency and bitter flavors, which thus round out or soften the wine [9]. Organic acids are other ingredients in wine that influence its organoleptic properties and affect its color, flavor, and aroma. It should be emphasized that different organic acids in wine have different organoleptic properties, so not only the total acidity of the wine and its pH, but also the content of individual acids in it are important. Organic acids in wine come mainly directly from the fruit from which it was made (e.g., tartaric, malic, and citric acids) and are the result of processes taking place during vinification, e.g., alcoholic and malolactic fermentation (e.g., succinic acid, acetic acid or lactic acid). Furthermore, their presence stabilizes the wine's pH, which ensures its microbiological stability, preventing the development of microorganisms [10]. It is well known that wines with high acidity/low pH have greater aging potential.

Sulfur oxide (IV) has many functions in wine. First of all, due to its antiseptic properties, it limits the growth of bacteria and wild yeast organisms, the development of which can lead to microbial deterioration of the wine. Secondly, thanks to its antioxidant properties, it prevents oxidation of wine components, which is especially important for the red ones [11]. Furthermore, under the influence of SO_2 quinones, brown polymers are transformed into their phenolic form and the composition of polyphenols is also improved [12].

Reactions occurring in wine during its maturation modify its chemical composition, which affects its quality values, as its structure, taste, and aroma are transformed. Not all wines get better quality during many years of maturation. This largely depends on their acidity, alcohol level, tannins, and residual sugar. Furthermore, the storage conditions of the wine are of considerable importance (temperature, humidity, bottle positioning, etc.). Hence, the multitude of factors and their interrelationships make it difficult to predict how a wine will evolve sensorially over years of storage. This study aimed to analyze the effect of wine aging on selected parameters such as free SO_2 concentration, acidity, pH, and concentration of selected organic acids (namely: acetic acid, citric acid, lactic acid, malic acid, succinic acid, and tartaric acid), total polyphenol concentration, and FRAP. For this purpose, these parameters were determined 1 year after bottling the wine (2015) and then 3 years later (2018). In addition, a sensory evaluation of the same wines was carried out in 2015 and 2018. Five commercially produced red wines in Poland (Rondo, Regent, Cabernet Cortis, Acolon, Zwegelt) were analyzed.

Materials and methods

Chemicals

18 MΩcm deionized water was obtained with the Milli Ro & Q water purification system. 36% hydrochloric acid and ferric chloride ($FeCl_3$) were purchased from Fluka (Steinheim, Germany), 2,4,6-tris (2 pyridyl)-1,3,5-triazine (TPTZ), gallic acid, acetonitrile (ACN), acetic acid, citric acid, lactic acid, malic acid, succinic acid, and tartaric acid from Sigma-Aldrich Chemie GmbH (Steinheim, Germany), Folin–Ciocalteu reagent from Fluka (Steinheim, Germany), sodium carbonate from Avantor Performance Materials (Gliwice, Poland), and 98% diethylamine and 85% orthophosphoric acid from Merck (Germany). All the chemicals were of analytical purity.

Samples

The wines for analysis were obtained from the Srebrna Góra vineyard (Poland). From 2015 to 2018, they were stored in a wine cooler (EuroCave Multi Temp S083) at a temperature of 12 ℃ and humidity of 60–70%. The cork that was used to close the wines is a Twin Top A type with dimensions of 44 × 23.5 mm. As for the material, it was mixed, a combination of a conglomerate and two discs of natural cork located at its ends. The use of the Twin Top cork allows the wine to undergo gas exchange and micro-oxidation, but also limits it only to the first period after closure. The manufacturer used Bordeaux-type bottles made of dark green glass, which limited the impact of light. These wines were available commercially. Three samples were taken from each type of wine for analysis. The grape cultivars from which the wines were made are listed in Table 1**.**

The grapevine varieties selected for research have confirmed higher than classic *Vitis vinifera* resistance to fungal and oomycete diseases such as downy mildew, powdery mildew, and gray mold [13], enjoying interest among winemakers also due to their ability to produce grapes of high health in cooler climate vineyards.

Selected varieties description

Rondo is a red grape variety that is a hybrid between Saperawi Siewiernyj × Saint Laurent. It was developed in Germany and is now widely grown in Europe, particularly in Germany, Poland, and the UK. Rondo is known for its resistance to cold weather. The bushes can withstand temperatures as low as −23 °C during the period of deep winter dormancy. The fruit ripening season is early, typically they are harvested for wine production in mid-September.

Table 1 Grape varieties from which the analyzed wines were made

Breeding number	Synonyms	Derivation	Origin	Wine color
Rondo	GM (Geisenheim) 6494–5	Saperawi Siewiernyj × Saint Laurent	Germany	Red
Regent	GF (Geilweilerhof) 67–198-3	Sylvaner × Müller Thurgau × Chambourcin	Germany	Red
Cabernet Cortis	FR (Freiburg) 437–82 r	Cabernet Sauvignon × (Merzling × (Zarya Severa × Muscat Ottonel))	Germany	Red
Acolon	We (Weinsberg) 71–816-102	Blauer Limberger (Blaufränkisch) × Dornfelder	Germany	Red
Zweigelt	Klosterneuburg 181–2-71	Blaufränkisch × St. Laurent	Austria	Red

The resulting wine is of high quality, with an intensive ruby color and a pleasant cherry aroma, Regent is another red grape variety that was developed in Germany. It is a cross between Sylvaner × Müller Thurgau × Chambourcin grape varieties. Regent is a more disease-resistant variety than Rondo, as well as more tolerant to frost. The shrubs can withstand temperatures as low as − 24 °C during deep winter dormancy. The resulting wine is of very good quality, with a southern character, an intensive red color, medium tannin content, and extractive. Cabernet Cortis is a red grape variety that was developed in Germany. It is a hybrid of Cabernet Sauvignon and a disease-resistant grape variety (Merzling × (Zarya Severa × Muscat Ottonel)). The shoots become woody early and are quite resistant to winter freezing, and can withstand temperatures of about − 22 °C. The variety produces full-bodied red wines that are characterized by their fruity aroma, high tannin content, and good aging potential. Cabernet Cortis may prove to can be useful in the warm regions of Poland [14].

Acolon is a red grape variety that was developed in Germany. It is a cross between Blaufränkisch and Dornfelder grape varieties. Acolon reaches maturity early and attains significant must weights. It is a relatively new variety that has gained popularity in Germany and some other parts of Europe for its fruity, aromatic, and full-bodied red wines. Acolon grapes have a thin skin and a moderate resistance to fungal diseases, which makes them suitable for organic and sustainable viticulture in a controlled and healthy condition. The wine made from Acolon grapes typically has a deep red color, with flavors and aromas of dark berries, black cherry, and spice, and it can be enjoyed young or aged [15]. Zweigelt is a red grape variety that was developed in Austria. It is a cross between Blaufränkisch and St. Laurent grape varieties. Zweigelt is a grape variety that is extensively cultivated across all wine regions of Austria, producing wines that are capable of aging well [16]. Its ability to produce high-quality wines has also caught the attention of wine growers in warmer regions of Poland. It produces medium-bodied red wines that are characterized by their fruity aroma, low tannin content, and good aging potential [17]. Upon consideration of wine characteristics, it can be concluded that Zweigelt represents a suitable alternative to Malbec and Merlot, which are not amenable for cultivation in cool climate conditions [18]. All of these grape varieties are well suited to cool climate regions, particularly in Europe and North America. They are resistant to disease and frost, which makes them easier to grow in regions with cold winters. According to recent findings, the chemical and macromolecular composition of wines made from hybrid red grape varieties can match those made from *Vitis vinifera* [19]. This observation holds promise for the future cultivation of these varieties in regions that experience challenging weather conditions and heightened risk of diseases. Despite the fact that selected varieties may not have the same global impact as more famous grape varieties such as Cabernet Sauvignon or Chardonnay, they are still important in their respective regions.

There are several international grape varieties that are comparable to Rondo, Regent, Cabernet Cortis, Acolon, and Zweigelt in terms of their suitability for cool climate regions. Some examples include:

(i) Pinot Noir—this grape variety is well suited to cool climate regions and is popular around the globe. It produces light- to medium-bodied red wines with a fruity aroma, low tannin content, and good aging potential.

(ii) Riesling—this white grape variety is well suited to cool climate regions and is popular in Germany and other countries. It produces wines with high acidity, floral and citrus aromas, and a wide range of sweetness levels.

(iii) Sauvignon Blanc—this white grape variety is popular around the globe and is well-suited to cool climate regions. It produces wines with a grassy and herbaceous aroma, high acidity, and a range of fruit flavors.

(iv) Chardonnay—this white grape variety is popular around the globe and can be grown in a range of climates. It produces wines with a range of flavors and aromas, depending on the region and winemaking techniques used.

Although widely known *Vitis vinifera* varieties produce highly regarded wines, they generally exhibit lower resistance to cold weather, diseases, and pests compared to specially bred varieties such as Rondo, Regent, Cabernet Cortis, Acolon, and Zweigelt that are specifically adapted to such conditions. Additionally, the ability of these varieties to resist fungal diseases presents a significant opportunity for reducing pesticide usage, which is worth considering in terms of their international meaning [16].

Total acidity

Total acidity determination was carried out using OIV-MA-AS313-01: R2009 methods described in the Compendium of International Methods of Analysis of Wines and Musts (Organisation Internationale de la Vigne et du Vin).

Free sulfur oxide (IV)

Free sulfur oxide (IV) determination was carried out using OIV-MA-AS323-04B: R2009 methods described in the Compendium of International Methods of Analysis of Wines and Musts (Organisation Internationale de la Vigne et du Vin).

Determination of organic acids

Before analysis, the wine samples were diluted 40 times with Mili Q water. Before measurement, all samples were filtered using 13 mm diameter nylon filters with a pore size of 0.22 µm (Labex Ltd. Filter-Bio). Organic acid concentrations were measured by using a high-performance liquid chromatography system (Prominence, Shimadzu, Japan) consisting of a Shimadzu LC-20AD liquid chromatography SIL-20A autosampler and SPD-M20A-diode array detector. Organic acids were separated using a Synergi 4u Hydro-RP 80A C18 column (250 × 4.6 mm ID 4 µm). The column temperature was set at 35 ºC. The measurement was performed under isocratic conditions. The mobile phase was the solution composed of 1 mL of ethylamine, 1.4 mL of orthophosphoric acid, and 20 mL of acetonitrile in 1 L of Milli-Q Water, and its flow rate was 1 mL/min. The volume of the dosed sample was 75 µL.

Analysis of pH

The pH was measured with an Elmetron CP-505 (Elmetron, Poland) pH meter.

Determination of total phenolics

Before analysis, red wines were diluted depending on the intensity—two, three, or six times with Milli-Q Water. The total content of phenolic compounds (TPC) was determined using the Folin–Ciocaletau reagent method according to the procedure previously described by Dobrowolska-Iwanek et al. [20] In summary, 60 µL of samples, 60 µL 7% Na_2CO_3, and 30 µL Follin–Ciocalteu solution were added to 540 µL of deionized water. The mixture of the reagents and water was used as a blank. Absorbance measurements were made using the Synergy 2 Multimode Microplate reader spectrophotometer by BioTek Instruments at 725 nm. The total concentration of polyphenols was determined based on a calibration curve derived from the measured absorbances for gallic acid standards (0.05–0.30 g/L gallic acid). The obtained results were expressed as gallic acid equivalents—g GAE/L (GAE—gallic acid equivalent).

Determination of FRAP

Before the analysis, the wine samples were diluted ten times with Milli-Q Water. Measurement of antioxidant activity using the FRAP method (ferric reducing antioxidant power) in the studied wines was carried out using the method of Benzie and Strain [21] with some modifications. The FRAP method is based on the principle of reduction of Fe^{3+} ions to Fe^{2+} by compounds with the antioxidant potential present in the sample, which are then complexed with 2,4,6-tris(2-pyridyl)-s-triazine. Blue-colored ferrous tripyridyltriazine (Fe(II)-TPTZ) is monitored by measuring absorption at 593 nm (Synergy 2 multimode microplate reader spectrophotometer, BioTek Instruments). The absorbance was determined after 30 min and was proportional to the antioxidant capacity of the antioxidants in the wines. The final results were given as mmol Fe^{2+}/L.

Sensory analysis

WSET Diploma in Wines and Spirits certified assessor performed a sensory evaluation in compliance with the WSET SAT (Systematic Approach to Tasting) standards.

Statistical approach

The differences between repeated measurements were tested using Student's *t* test. Differences with $p < 0.05$ were considered to be statistically significant. The partial least square model (PLS) was constructed to explain the relationship between changes in the original examined parameters (these changes were considered as predictive parameters) and the change in the wine rating made by the sommelier (response parameter). It was assumed that the parameters with large loadings (i.e. with absolute values > 0.3) on the first two latent components determine the axes (latent components) of the new coordinate system (in PLS model) to the greatest extent. To express the strength of bivariate associations between such parameters, the cosines of the corresponding angles (i.e., correlation coefficients) were calculated. The "corresponding angle" means the angle determined by the two lines connecting the origin with coordinates of both parameters on the PLS loadings plot. Statistical analysis was conducted using package SIMCA-P v.9 (Umetrics, Umeå, Sweden). Package STATISTICA v.13 (TIBCO Software Inc., Palo Alto, CA, USA) was used for graphic representation of results. Program provided by MP System Co. (Kraków, Poland) was used to calculate correlation coefficients for parameters showing correlations in the PLS model.

Results

In this study, parameters such as pH and total acidity, free sulfur oxide, the total concentration of polyphenols, FRAP, and organic acids were determined in five types of red wine (Rondo, Regent, Cabernet Cortis, Acolon, and Zweigelt) 1 year after bottling the wines and also after 3 years of aging. In addition, sensory analysis of wines was performed in the years in which their chemical analysis was performed. The results of the analysis are presented in Tables 2–3.

Sensory analysis

Table 2 shows how the wines sensory parameters tested changed over 3 years. Details on the results of the sensory analysis of wines in individual years are described in the supplement attached to the article (SI).

When comparing the average sommelier rating of the wines (Table 2, Fig. 1), the wines tasted in 2015 received a score that was significantly lower than that determined during the tasting of the 2018 wines.

Chemical analysis

The average pH and total acidity expressed as tartaric acid of the wines analyzed in 2015 did not differ significantly from the results for the same wines in 2018 (Table 3). Free sulfur oxide concentration in all tested wines decreased significantly in the course of aging the wine ($p < 0.05$). The average FRAP determined in red wines 1 year after their preparation turned out to be significantly lower than that determined after 3 years of aging ($p < 0.05$). The total concentration of polyphenols in red wines increased during aging, but this change was statistically insignificant ($p > 0.05$). Average concentrations of tartaric, malic, and citric acid were lower in wines analyzed in 2018 compared to wines tested 1 year after bottling (2015). However, statistically, significant differences were observed only in the case of tartaric and citric acid ($p < 0.05$). In the case of acetic acid, its concentration increased significantly during the 3 years of aging ($p > 0.05$). The mean concentrations of the remaining acids did not change significantly during storage.

Table 2 Sensory changes in wines over 3 years

Type of wine	Sensory changes in wine
Cabernet cortis 2013	Wine changed significantly since last tasting in 2015. First, the color changed from purple to ruby with orange shades. Second, the aromas showed more secondary and tertiary character in 2018 than in 2015. After 3 years of aging, the quality of aromas and flavors also changed significantly; in 2018, the wine seemed to be more elegant and pleasant, although the balsamic vinegar fraction (acetaldehyde) made the overall impression closer to the earth. Thirdly, the structure of the wine had a better balance in 2018, without an impression of unripe wine what was a problem in 2015 tasting. The acidity, tannin, and alcohol balance were also on good level, and flavors character met acidity level well. The wine gained 88/100 points
Zweigelt Rosé 2013	Sensory analysis showed better quality of the sample in 2018 than in 2015. The wine has changed its color from pale pink to pink-orange on the core and orange shades on the rim. The main difference was in the quality of the aroma and flavors, as well as in the balance of the structure. The significant advantages of this wine were: varietal aroma and flavor characteristic, acidity and alcohol integration, acidity and flavors finesse and medium length of the aftertaste. The overall wine assessment was surprisingly good, especially compared to 2015 tasting results, 85/100
Acolon 2013	There were clearly detectable differences between the samples tasted in 2015 and 2018. Firstly, the color of this wine has not changed significantly (deep ruby color on the core and purple shades on the rim). Aroma and flavors development with proper varietal character, secondly, structure balance built by acidity, tannins and alcohol, thirdly, the width of the aromatic spectrum. Definitely, the most significant difference between the quality of samples tasted in 2015 and 2018 were in the tannins elegance. The overall wine assessment was: 85/100
Regent 2013	Clearly detectable differences between the samples tasted in 2015 and 2018. First and most significant: cloudiness transformed into sediment. That feature allowed for higher score in the overall quality assessment. The color of the wine (deep ruby core) changed mainly on the rim, whose shades took on an even more intense dark ruby color. The structure of the wine in 2015 was described as full bodied, in 2018 as medium bodied, the difference is caused by tannins transformation, its silkiness, however, in 2018 accounted for higher scores. Thirdly, the quality of aroma and flavors finesse and elegance were among major advantages. The overall wine assessment was: 88/100
Rondo 2013	The detectable differences between the samples tasted in 2015 and 2018 were in the aroma and flavor area. The color of this wine has not changed significantly (deep ruby color on the core and intensely purple on the rim). The spectrum of aromas was definitely wider in 2018 than it was assessed in 2015; moreover, the quality of aromas was also improved, which significantly accounted for the higher overall rating. Same as in 2015 assessment, the largest disadvantage of this wine occurred to be low alcohol level resulting in lack of vigor. The overall wine assessment was: 89/100

Table 3 Composition (mean ± SD) and sensory evaluation of wines (white fields—results of a chemical and sensory analysis carried out in 2015, dark fields—results of a chemical and sensory analysis carried out in 2018)

Parameter	Wine					*P**
	Regent	Rondo	Cabernet Cortis	Acolon	Zweigelt	
pH	3.7	3.4	3.5	3.6	3.2	
	3.8	3.5	3.6	3.6	3.2	
Total acidity as tartaric acid [g/L]	5.3 ± 0.6	5.1 ± 0.3	6.3 ± 0.3	5.0 ± 0.2	7.4 ± 0.4	0.170
	5.1 ± 0.7	5.1 ± 0.4	6.2 ± 0.2	4.9 ± 0.6	6.6 ± 0.2	
Free sulfur oxide (IV) [mg/L]	29.9 ± 0.6	76.8 ± 0.6	44.8 ± 0.5	57.6 ± 0.8	21.1 ± 0.5	0.040
	19.2 ± 0.3	29.0 ± 0.2	35.8 ± 0.9	28.16 ± 0.2	7.5 ± 0.1	
FRAP [mmol Fe^{2+}/L]	47.3 ± 2	33.7 ± 1	69.4 ± 2	26.8 ± 2	0.79 ± 0.01	0.035
	31.0 ± 2	22.9 ± 1	53.7 ± 1	18.7 ± 1	1.97 ± 0.27	
Total phenolic content [g GAE /L]	2.13 ± 0,15	1.50 ± 0.01	3.64 ± 0.01	1.42 ± 0.01	0.20 ± 0.01	0.054
	4.13 ± 0.26	2.86 ± 001	7.4 ± 0.04	2.42 ± 0.05	0.32 ± 0.04	
Tartaric acid[mg/L]	4969 ± 248	7323 ± 366	5553 ± 278	5996 ± 300	3187 ± 32	0.015
	1396 ± 25	1848 ± 44	1809 ± 9	1770 ± 23	2838 ± 14	
Malic acid [mg/L]	2418 ± 4	3380 ± 200	15,358 ± 212	6374 ± 30	6322 ± 32	0.071
	1700 ± 4	2755 ± 28	14,314 ± 114	6075 ± 115	3464 ± 33	
Lactic acid [mg/L]	3038 ± 152	2470 ± 124	1138 ± 57	3223 ± 161	607 ± 5	0.354
	2747 ± 1	2425 ± 47	1346 ± 76	3141 ± 27	350 ± 6	
Acetic acid [mg/L]	392 ± 5	319 ± 16	354±18	334 ± 17	114 ± 1	0.041
	1152 ± 8	480 ± 9	899 ± 199	1368 ± 187	226 ± 34	
Succinic acid [mg/L]	844 ± 42	727 ± 36	1276 ± 64	740 ± 37	567 ± 10	0.165
	1056 ± 19	768 ± 15	1215 ± 70	794 ± 43	855 ± 126	
Citric acid [mg/L]	383 ± 12	419 ± 12	601 ± 16	326 ± 12	759 ± 15	0.041
	242 ± 8	166 ± 8	424 ± 21	250 ± 17	254 ± 27	
Sensory assessment of wine	87/100	85/100	82/100	80/100	78/100	0.011
	88/100	89/100	88/100	85/100	85/100	

*Significance level of difference was set to 0.05 (5%)

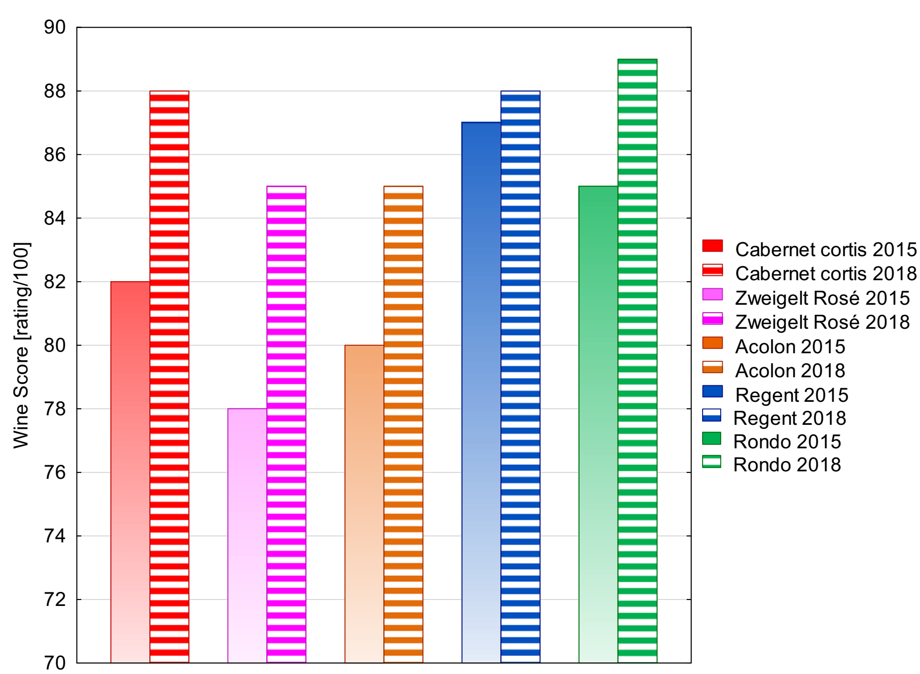

Fig. 1 Win scores in the years 2015 and 2018

Chemometric approach

The PLS model fulfilling cross-validation criteria was constructed. The predictive parameters were the changes in the following parameters: pH, FRAP, concentrations of acetic acid, citric acid, malic acid, lactic acid, succinic acid and tartaric acid, total acidity, total phenolic content, while the change in the wine rating made by the sommelier was the response parameter. One remaining parameter (free sulfur oxide (IV)) was not included in the model as it was considered non-informative, i.e., it had relatively small loadings on both latent components.

The model had two significant components with eigenvalues 2.84 and 1.08, respectively, and explained 78.4% of the variance in the predictive parameters and 99.1% of response parameter variance. The loadings for the first two latent components are shown in Fig. 2. The first latent component in this model had positive weights predominantly for changes in sommelier rate, and changes in values of FRAP and tartaric acid, and negative rates for changes in values of pH, malic acid, and citric acid (the last two being strongly positively correlated). The second latent component was loaded positively mainly by changes in values of lactic acid and negatively by succinic acid, and, to a lesser extent, by changes in pH and sommelier rate (also going in different directions). Therefore, changes in lactic and succinic acids were strongly negatively correlated (Fig. 2, Table 4). The response parameter correlated positively mainly with changes in lactic acid and FRAP, and negatively with changes in succinic acid.

Table 4 Correlation coefficients for the pairs of parameters based on first two latent components of PLS model (only first 10 correlation coefficients for predictive parameters with highest absolute values were shown and only first 5 correlation coefficients with highest absolute values for response parameter; the letter "d" means that the model takes into account changes in parameter values, not the values themselves)

Pairs of correlated parameters		Correlation coefficients
Predictor parameters		
d-malic acid	d-citric acid	0.996
d-FRAP	d-tartaric acid	0.901
d-pH	d-malic acid	0.852
d-pH	d-citric acid	0.802
d-pH	d-succinic acid	0.792
d-FRAP	d-malic acid	− 0.780
d-FRAP	d-citric acid	− 0.833
d-lactic	d-succinic acid	− 0.907
d-tartaric acid	d-malic acid	− 0.974
d-tartaric acid	d-citric acid	− 0.990
Response parameter and predictive parameters		
d-sommelier rate	d-lactic acid	0.922
d-sommelier rate	d-FRAP	0.911
d-sommelier rate	d-tartaric acid	0.642
d-sommelier rate	d-citric acid	− 0.530
d-sommelier rate	d-succinic acid	− 0.672

Discussion

One of the parameters that changed significantly during the aging of the wine tested was the concentration of free

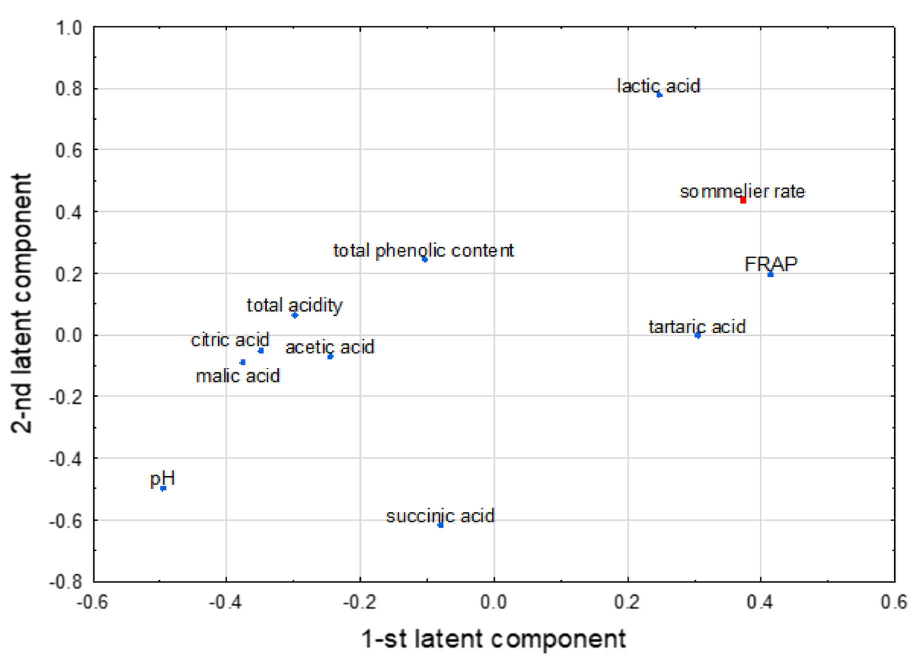

Fig. 2 The parameter loadings on the first and second latent components in the PLS model (for simplicity, the letter "d" has been omitted, which further means that the model takes into account changes in the values of the parameters, and not the values of these parameters themselves)

SO_2. Free sulfur oxide is defined as the sum of SO_2 and HSO_3^- [22]. As expected, the average concentration of free sulfur oxide decreased significantly over 3 years. Gambuti et al. (2020) analyzed the change in the concentration of sulfur compounds and phenolic compounds in wines rich in tannins during the 5-year aging process. During the analysis of three types of wine: Aglianico, Casavecchia, and Pallagrello found a significant decrease in sulfur oxide concentration in all wines after 5 years of wine aging. Many studies have shown that this compound is involved in regulating all reactions that occur during red wine, with long-term effects after bottling [23]. Thus, it has been found that the HSO_3^- ion (the dominant form of free sulfur oxide at wine pH 3.0–3.7) in equilibrium with SO_2 is used in reactions with wine components, among them also oxidation products such as quinones and acetaldehydes. Furthermore, with the long aging of red wine, SO_2 reactions with flavanols dominate, leading to the synthesis of catechin and proanthocyanidin sulfonates [23–25].

On the other hand, no significant changes were observed during the several years of aging in terms of the change in pH and acidity expressed as the tartaric acid concentration.

During the 3 years of bottle aging (time between the first and second chemical sensory analyzes), it was observed that the total content of polyphenols in all wines slightly increased. These observations were not consistent with the results of the study on wine produced from *Vitis vinifera* L. Tempranillo grapes by the group of Guadalupe and Ayestarán [26]. They found a 10% drop in the concentration of polyphenol family over 2 years of aging. Balga et al. in turn, investigated the effect of 1-year aging of wines from five different grape varieties on changes in the concentration of polyphenolic compounds, the process taking place in 25 l glass balloons [27]. In the course of the analysis, the same trend was not observed in the change in the total content of polyphenols during the aging year. In some wines, the concentration values increased during this period, and in others, they decreased [27]. Ivanova et al. [28] investigated the change in polyphenol concentrations in wines from the *Vranec Vitis vinifera* L. grape variety in four phases: after maceration and after 2, 6, and 16 months of storage Three different maceration times were used for production wines: 3, 6, and 10 days, a different amount of SO_2 and two various types of yeast. The results obtained from the analysis of the wines indicated, among others, a slight decrease in the total polyphenol content in wines that had been macerated for 3 days (irrespective of the SO_2 concentration), but similar changes were not observed in the case of wines produced with a longer maceration time [28]. Yıldırım et al., in turn, analyzed the profile of phenolic acids in organic wines over 5 years of aging and found that after this period the concentration of phenolic compounds in all wines decreased [29]. Similar observations were made by Peri et al. examining wines after 3 years of aging and then after 7 years [30]. Wine, as a mixture of different substances, undergoes a continuous transformation over time, and, particularly, phenolic compounds can undergo condensation reactions turning into chemical compounds with slightly different chemical properties and therefore having different reactivity with salts of phospholiphatic and phosphomolybdic acids (components of the Folin–Ciocalteu reagent) [31]. Other authors point out that these compounds can gradually be degraded by oxidation over time when the wine is matured and stored in bottles [3, 32]. Changes found in TPC can also be caused by the enzymatic activity of residual microorganisms present in wine [31]. The determination of the total content of polyphenols using the Folin–Ciocalteu reagent is a method widely used in the wine industry because of the ease of experimentation and the use of relatively inexpensive equipment (a spectrophotometer is sufficient). However, it should be remembered that the Folin–Ciocalteu reagent is not specific for phenolic compounds, and many compounds that do not belong to the phenols group may also reduce it [33]. In the case of antioxidant activity (determined as FRAP), its average value decreased significantly over the 3 years of wine age. The antioxidant potential of these beverages is influenced not only by the phenols present in the wines but also by other components such as organic acids or minerals.

Among the methods of similar purpose (Folin-Ciocalteu, FRAP and DPPH$^\bullet$ assays), the FRAP method is considered to be the most reliable. It gives a better indication of the concentration of potentially oxidizable polyphenols than the other two as it is more selective than Folin–Ciocalteu assay and more resistant to solvent impurities and changes in hydrogen ion concentration than DPPH$^\bullet$ assay [34]. However, free SO_2 present in wine samples may enhance the antioxidant effect and overestimate the results obtained by FRAP method, because this compound reduces quinones (which are products of polyphenols oxidized by Fe(III) during the analytical reaction) back to catechols, reversing the oxidation process. The limitation of this work is the lack of consideration for this phenomenon, the mechanism of which is still not fully explained, as other antioxidants present in wine, may also synergize the effect caused by SO_2 [34, 35]. The removal of SO_2 from the tested wine samples or the estimation of its impact on the FRAP results, based on a separate measurement of SO_2 concentration, is a task for the future studies.

For the organic acids determined, a significant decrease in the concentration of tartaric and citric acid and an increase in acetic acid was observed as the bottled wines aged. The latter observation can be explained by the action of acetic acid bacteria leading gradually from the oxidation of ethanol through acetaldehyde to acetic acid. These bacteria belong to the natural bacterial flora of grapes and wine and are well adapted to the aerobic conditions of grape must and wine.

Bartowsky and Henschke reviewed the acetic acid bacteria associated with wine, including the changes in wine aroma and flavor caused by their metabolism [36]. The concentrations of the other acids did not change significantly. Reduced tartaric and citric acid concentrations can be caused by the formation of ethyl esters during wine aging by chemical or enzymatic esterification reactions [37]. In the case of tartaric acid, salts of this compound (such as potassium bitartrate) can form even after bottling. It is a relatively stable acid from a microbiological point of view [11].

The constructed PLS model revealed that the increase in the sommelier's rating was directly correlated with the decrease in the FRAP value and the decrease in tartaric acid concentration (with changes in both explanatory parameters being statistically significant). At the same time, the increase in the sommelier's rating was inversely correlated with the decrease in citric acid concentration (statistically significant change in this parameter) and succinic acid concentration (change in itself insignificant). In turn, the change in the concentrations over the years of tartaric, malic, and citric acids had a significant effect on the determined change in FRAP; however, the decrease in the FRAP value and the decrease in tartaric acid concentration were directly correlated, while the decrease in the FRAP value and the decrease in malic and citric acids concentrations were inversely correlated. Therefore, as sequestrants, these acids had a varied effect on the antioxidant activity of wines, in contrast to polyphenols, for which no such relationship was demonstrated. Other observed correlations between changes in the values of individual parameters require further clarification.

The wine samples tested after 3 years showed differences in aroma and taste. Differences were also noticeable in the appearance of the wines, according to well-known principles of color changes during aging. Considering the relatively short aging period of only 3 years, the changes were visible. The wines were rated very positively, and their development showed that Polish wines behave during maturation the same way as wines from other regions of the world. It should be emphasized that there were no factors that could undermine the credibility of the research, i.e. that professional wine storage conditions were maintained between the testing phases. It is worth mentioning that the test results may be also related to the type of bottle closures used for the samples. In the case of closures with different parameters or construction, the results may differ; however, this is an issue for a separate investigation.

Conclusions

It should be stressed that such studies like ours were carried out for the first time using wines made in Poland, which undoubtedly contributes to a greater understanding of the chemical composition and sensory quality of Polish wines. The results of the analyses also provide a premise for conducting research on a larger scale, the results of which could help to provide a more in-depth description of the qualitative and quantitative changes that occur during the aging of wine. The results of the PLS analysis suggest that the sommelier's assessment associated with the change in FRAP depends to a greater extent on the change in the concentrations of various acids in wines than on the concentration of polyphenols.

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Data availability We declare transparency of the data for publication. The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors declare no conflict of interest.

Ethics approval Ethics approval was not required for this research. This study does not involve any human or animal testing.

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