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DETERMINATION OF THE INFLUENCE OF CERTAIN FACTORS ON THE QUALITY OF MARSALA WINES

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Abstract. The study of the physicochemical composition indicators of Marsala blended wine samples aged at 45–50°C has shown that during the aging period under O_2-containing conditions, especially in initially heat-treated samples, a decrease in the amount of sugars, an increase in the amount of saturated esters, and a decrease in the amount of phenolic compounds were observed. A similar situation was also observed in the amount of saturated esters. This is related to the acceleration of melanoidin and ester formation reactions during heat treatment under oxygenated conditions. In samples aged for one year under oxygenated conditions with heat treatment, a dark amber color, a soft balanced taste, and a vanilla-noted aroma were formed, whereas under oxygen-free conditions this was expressed quite weakly. During the heat treatment of blended samples by traditional and electrothermal methods, it was found that, compared to the control, the amounts of hydroxymethylfurfural (HMF), furfural, and furan-2 acid were significantly lower in the experimental samples. If in the control variant treated by the traditional method HMF was 87 mg/dm^3, furfural 0.8 mg/dm^3, and furan-2 acid 2.1 mg/dm^3, then in the first electrothermally treated experimental sample these indicators were 64 mg/dm^3, 0.6 mg/dm^3, and 1.7 mg/dm^3, respectively. It was found that by adding concentrated juice to the blend, the amounts of glucose, sucrose, and fructose in the samples increased significantly, which in turn affected the previous indicators, namely the amounts of HMF, furfural, and furan-2 acid, creating a basis for their significant increase. If in samples boiled at 40°C the HMF amount was 2.8 g/kg, furfural 0.05 g/kg, and furan-2 acid 0.21 g/kg, then at 80°C these indicators were 3.3, 0.08, and 0.82 g/kg, respectively, being higher. Since the intensity of formation of these compounds increases at high temperature, it is important to take this into account in the production of concentrated juice.

Keywords: marc, oxygen, nitrogen, Marsala, blend, heat treatment, alcohol, electrothermal

Introduction. Formulation of the problem

Marsala-type wines, as is evident from their name, were first produced in the Marsala region of Italy. Marsala-type wines belong to a special group of oxidized wines. Their production requires specific technological methods, without which the characteristic features of these wines cannot be formed. Marsala-type wines are primarily characterized as high-extract wines. Therefore, in order to obtain such wines, wine materials and blending components must possess sufficiently high extract content. If this condition is met and the subsequent essential technological methods are also applied, it becomes possible to develop the characteristic properties of these wines.

In the production process of Marsala-type wines, the products of sugar–amine reactions play a major role, ensuring the specific color, bouquet, and taste of the wine. The accumulation of melanoidin products has a significant impact on the quality of Marsala wines. At the same time, higher amounts of reaction products such as aldehydes, particularly formaldehyde, furfural, hydroxymethylfurfural, acetic, isovaleric aldehyde, and others, are observed. As can be seen, the products of carbonyl–amine reactions play a fundamental role in the production of Marsala-type wines, ensuring the specific color, bouquet, and taste of the wine.

All these factors are strongly influenced by the composition of raw materials and intermediate products (initial wine material) as well as the preparation scheme. In order to ensure these indicators, thermal treatment, i.e., heat processing, is one of the key factors. During heat treatment, the characteristic taste and aroma of these wines are formed, and color development also takes place. However, the correct selection of the heat treatment method is one of the factors affecting quality.

Although certain studies have been conducted on Marsala-type wines and the heat treatment of blending materials used in their production, the effect of oxygen depending on temperature during this process has not been sufficiently investigated. At the same time, the role of other heat treatment methods in this process cannot be considered adequately studied. Therefore, determining the most optimal variant of the complex influence of the initial material composition, temperature, and oxygen under different storage periods is of particular relevance.

Analysis of recent research and publications

Microoxygenation (MOX) is a technology that ensures a slow and continuous oxidation reaction throughout the entire wine production process in order to improve wine quality. Compared with passive MOX, active MOX provides more precise control over oxygen. Innovation in MOX equipment based on active MOX techniques will generate inspiring interest in enological research. The integration and development of precise MOX will enable targeted control of wine quality and the creation of distinct wine style characteristics [1].

Understanding wine storage duration is complex and involves factors such as aroma preservation, taste development, and market acceptance. Balancing the effects of oxygen, temperature, and light is essential. Effective management, including the strategic use of preservatives and additives, is crucial for maintaining quality and extending shelf life. This review emphasizes the delicate balance required to preserve wine quality from production to consumption [2].

We compared static and dynamic methods of must clarification in terms of oxygen regime, phenolic acid content, and physicochemical parameters of wine. The highest amount of dissolved oxygen in must was observed in control variants using static clarification. After fermentation of the must, a statistically significant decrease in oxygen content to the level of 0.40–0.92 mg/L was observed in all studied variants [3].

This review presents the key characteristics of the world's major fortified wines—Madeira, Port, Sherry, Muscat, and Vermouth—in three sections. Given the global popularity and economic importance of fortified wines, there is a need for more extensive research to better understand the reactions and mechanisms occurring during different stages of production, particularly oxidative and heat aging processes [4].

Post-harvest dehydration of Marselan wines increased the levels of isobutanol, isoamyl alcohol, phenylethyl alcohol, ethyl acetate, isoamyl acetate, and ethyl butyrate, and also enhanced floral, fruity, and sweet sensory attributes. At a high dehydration level of 25%, Marselan wine showed the lowest acceptability, which was associated with a significant increase in tannin content. In summary, post-harvest dehydration was beneficial for improving the quality of Marselan wine [5].

Sorghum wine lees are a by-product of sorghum wine production and can be used as a valuable biomass resource through pyrolysis. However, there are very few studies on the pyrolysis of sorghum wine lees. During pyrolysis, as the conversion degree increased, the activation energy first increased to 210–220 kJ mol^{-1}, then stabilized at 190–200 kJ mol^{-1}, and finally decreased rapidly to approximately 100 kJ mol^{-1}. This study provides a theoretical basis for the utilization of sorghum wine lees via pyrolysis [6].

This study aimed to investigate the feasibility of using Phase Change Materials (PCM) to control fermentation temperature of grape must and provides insights into potential energy-saving strategies in winemaking processes. While the control sample reached approximately 30 °C after about 50 hours, fermentation with PCM reached this level more than 30 hours later and peaked at an average of 29.5 °C. This led to an average fermentation temperature of 24 °C with PCM compared to 27 °C in the control, demonstrating that PCM effectively absorbs fermentation heat and maintains temperature below the 30 °C target. Potential applications for improved temperature control in winemaking are proposed [7].

High-power ultrasound, high hydrostatic pressure, pulsed electric fields, ultra-high pressure homogenization, and more recently cold plasma are among the most studied emerging technologies. This review summarizes recent scientific studies on these five non-thermal technologies and their current status in enology. Their potential applications include improving extraction, shortening maceration time, inactivating microorganisms and oxidative enzymes, reducing the use of chemical additives, accelerating aging, improving wine quality, and more [8].

Today, world-famous wines such as Marsala, Madeira, Port, and Sherry are classified as dessert wines due to their high alcohol content, sweet taste, and intense aromatic profile, and are sometimes served as aperitifs. This review provides an overview of traditional vinification processes, including microbiota and autochthonous yeast species, as well as regulatory aspects of major fortified wines in Italy, Portugal, and Spain. The winemaking process plays a key role in the formation of volatile organic compounds (VOCs) that define each fortified wine's aroma, giving them a distinctive organoleptic "fingerprint" and "terroir" character. Volatile and odor-active compounds found in fortified wines during oxidative aging are also discussed [9].

This study investigated the effects of fermentation temperature (22 °C, 25 °C, 28 °C) and grape must Brix concentration (26°, 29°, 32°) on the physicochemical and aromatic profiles of Musalais wine, a traditional fermented alcoholic beverage from the Xinjiang region of China. Results showed that higher fermentation temperature (28 °C) increased total acidity (TA) and residual sugar content (RSC), while lower temperature (22 °C) resulted in higher pH, phenolic content, and anthocyanin levels. Ethanol content reached its maximum at 25 °C, particularly in wines produced from must with 29 Brix concentration [10].

The concentration of dissolved oxygen in wine is a critical parameter that must be controlled during production to ensure quality preservation after bottling. However, oxygen ingress occurs at multiple stages including production, stabilization, and packaging. This review provides a comprehensive analysis of how changes in these parameters affect kLa. In addition, the influence of hydrodynamics, design parameters, and operating conditions on kLa is also examined [11].

This review compiles information on emerging wine processing technologies such as high-pressure processing, pulsed electric fields, ultrasound, microwave, and irradiation. Overall, non-thermal processing technologies have been shown to improve wine color characteristics (phenolic and anthocyanin content), stability, and sensory properties, reduce the need for SO_2, shorten maceration time, reduce microbial load, and thereby enhance overall wine quality, safety, and shelf life [12].

It has been determined that the melanoidin fraction significantly contributes to wine color, accounting for 65–75% of the browning index, 70–77% of total color intensity (CI), and 76–88% of the To parameter. In vitro antioxidant assays (DPPH) showed that melanoidins account for 77% to 90% of total radical scavenging activity. These results indicate that melanoidins play an important role in the sensory and functional properties of Madeira wines. They provide preliminary evidence of the functional potential of melanoidins as natural antioxidants in Madeira wine. Future studies should include a broader range of wines and further investigate their chemical structure and bioavailability [13].

Although the Maillard reaction has mainly been studied in the context of thermally processed foods, Maillard-derived products such as thiazoles, furans, and pyrazines have also been identified in aged sparkling wines, where they are associated with bready, roasted, and caramel-like aromas. The review discusses physicochemical factors affecting the Maillard reaction, particularly pH-dependent reactions and Maillard activity under low temperature and/or low pH conditions in sparkling wine. Attention is given to the origin and composition of precursors (amino acids and sugars), as well as the role of metal ions in accelerating the reaction. Understanding the contribution of individual physicochemical factors helps clarify reaction mechanisms and sensory outcomes [14].

The aim of this study was to determine the optimal temperature and heating time required to obtain Madeira wine considered typical by expert panel evaluation. Descriptive analyses of typical Madeira wines were performed, and seven descriptors were selected: “dried fruit,” “nutty,” “moldy,” “cooked,” “oak,” “mushroom,” and “brown sugar.” GC-olfactometry identified up to 10 aroma-active zones corresponding to these descriptors. It was observed that typicity indices were positively correlated with sotolon concentration, sugar content, and heating time, and negatively correlated with fermentation time [15].

The brown color formed during thermal processing of foods is mainly due to the formation of melanoidins. Despite their widespread presence and importance in foods, melanoidins remain among the most mysterious food macromolecules because their chemical structure is still not fully understood and is highly dependent on food composition (simple sugars/polysaccharides, proteins/peptides/amino acids, phenolic compounds, etc.) [16].

This study investigates the effect of barrel surface-to-volume ratio on extraction processes during the early stages of Sherry Brandy production. Distillate aged at 60% alcohol was studied in barrels of two different volumes (500 L and 250 L) and three oak types—Quercus alba (American oak), Quercus robur (French Limousin oak), and Quercus petraea (Spanish oak), either pre-treated with Sherry wine or not. Total phenolic index (TPI), total phenolic extraction, and color change (CIEDE2000) compared to the initial distillate were evaluated in all aged samples [17].

This study aimed to clarify the formation of furfural compounds in apple products treated by pasteurization and high-pressure processing (HPP). During initial thermal processing of apple puree, 5-HMF, F, FMC, and MF increased significantly. Based on changes in fructose, glucose, and sucrose after thermal treatment, it was determined that sugars and heat treatment strongly influence furfural formation. Commercial juice and puree samples processed by different methods were also analyzed. Five furfural compounds were more frequently detected in pasteurized and ultra-high temperature (UHT) processed juices compared to HPP-treated samples. 5-HMF and FMC were detected in all pasteurized puree samples, followed by F, MF, and HDMF with detection rates of 79.31%, 72.41%, and 51.72%, respectively. These results may be useful for risk assessment and nutritional recommendations, especially for infants and young children. In addition, medium-level HPP processing (300 MPa for 15 min) is considered a promising alternative to reduce furfural formation in juice and puree production [18].

This study investigates the potential of wine residues as substrates for HMF production and examines the use of Saccharomyces cerevisiae as a whole-cell biocatalyst for converting HMF into high-value compounds, offering an alternative to chemical synthesis. Different strains of S. cerevisiae were compared for their ability to convert HMF, showing different oxidation and reduction capacities. For the first time, industrially relevant HMF derivatives were obtained from HMF-rich hydrolysates derived from winery waste such as grape pomace and excess must, demonstrating the potential for sustainable processing [19].

In recent years, attention has increased regarding the potential carcinogenic effects of 5-hydroxymethylfurfural on human health. This compound is used as a marker of honey adulteration and as an indicator of heat treatment in sugar-containing foods. In this study, levels of 5-hydroxymethylfurfural, furfural, and 2-furoic acid were evaluated in sapa syrup, Marsala wines, and bakery products containing sapa. The average 5-HMF content in sapa syrup was 2.3 ± 0.8 g/kg, while in bakery products it was 167 ± 133 mg/kg. In addition, Marsala wine contained 175 ± 150 mg/L of 5-HMF. Furfural and furoic acid levels in bakery products were 7.0 ± 4.5 and 48 ± 46 mg/kg, respectively, while in Marsala wines they were 3.7 ± 1.7 and 27 ± 18 mg/L, respectively. This study showed that 5-HMF can be used as a marker to detect the use of sapa in food preparation [20].

The aim of the study is to determine the influence of certain factors on the quality of Marsala wines.

To achieve this aim, the following objectives are set:

- To investigate the effect of enzyme preparations on the physicochemical composition parameters of Marsala wine materials;
- To study the effect of extracts obtained from the solid parts of grape clusters on the composition of Marsala wine materials;
- To examine the effect of the production method on the content of aromatic compounds in Marsala wine materials.

Materials and methods.

The research object includes grapes, must, wine materials, etc. during the research, white grape varieties are used. The grape variety used for wine production should accumulate a large amount of sugar and dry matter, while having a low content of titratable acids; it should ensure the maximum extraction of individual chemical components (nitrogenous compounds, phenolic compounds, and other extractive substances) from the solid parts of the berry; it should enrich the wine material with products of sugar-amine reactions and create optimal conditions for the formation of specific characteristics typical for the taste and bouquet of Marsala.

For this purpose, blends of wine materials obtained from locally cultivated Rkatsiteli (at least 50%) and other white grape varieties (Bayan Shira, Chardonnay, Aligote, Feteasca, etc.) are used. The blend composition includes acidified fortified wine material; fortified must and fortified; vacuum must (bekmez).

Wine materials are prepared in different conditions according to residual sugar content. During fortification, rectified ethyl alcohol (96 vol.%) and raw grape spirit (25–90 vol.%) are used to produce wine materials with different alcohol strengths.

Marsala blend components were stored and aged for 12 months under different conditions according to the following variants, and samples were taken every three months to study physicochemical parameters:

1. Blend wine material + heat (45–50°C) + oxygen;
2. Blend wine material + room conditions (18–20°C) + oxygen;
3. Blend wine material + heat (45–50°C) + oxygen-free;
4. Blend wine material + room temperature conditions (18–20°C) + oxygen-free

In these variants, wine materials enriched with nitrogen, fermented on the mash, and obtained by thermal treatment of the mash were also processed under oxygenated and oxygen-free conditions, both at normal and elevated temperatures.

Grapes are harvested when they accumulate at least 20 g/100 ml of sugars and 6–8 g/dm³ of titratable acidity. During harvesting, grapes are sorted by removing clusters and berries infected with diseases and pests.

The determination of physicochemical parameters of must, wine materials, and wines was carried out using generally accepted, new, and modified analytical methods [22,23].

Determination of organic acids and mass concentration of glucose and fructose. This method is carried out using a chromatograph of the Shimadzu company. For analysis, a Phenomenex Luna C18 chromatography column is used. Its dimensions are 2.1 × 150 mm, filled with sorbent.

Determination of mass concentration of amino nitrogen. The principle of the method is based on the interaction of the amino group with formaldehyde, resulting in the formation of methylene derivatives. These are stronger acids and can be titrated with alkali. The compliance norm is 23 mg/dm³.

With the help of modern analytical methods, the amino acid composition, aromatic substances, volatile components, anthocyanins, polyphenols, etc. are studied. Determination of the mass concentration of volatile substances. The main components of the volatile complex were determined by direct chromatography using a plasma ionization detector on an Agilent Technologies 6890 chromatograph. The column is a quartz capillary DV-5, 60 meters long. The carrier gas is nitrogen, with a flow rate of 3 cm³/min. The column diameter is 0.33 mm. The temperature of the evaporator and detector is 230°C. The thermostat temperature is programmed from 70 to 190°C at a rate of 4°C/min. The sample volume is 1 µl. Concentration calculations are carried out using the absolute calibration method.

Mathematical processing of the obtained results is performed in accordance with existing methods [24,25].

Results of the research and their discussion

The effect of oxygen and initial heat treatment on the physicochemical composition indicators of heat-aged Marsala blend samples.

Studies were conducted to determine the influence of oxygen on the development of wine formation processes and on wine composition. The physicochemical composition indicators of Marsala blend wine samples aged for three months at 45–50°C were investigated under oxygen-containing and oxygen-free conditions, as well as depending on whether the initial wine material had been subjected to heat treatment or not. First, the pH values and the amount of acids determined in the samples according to different variants are presented (Table 1).

As seen from Table 1, no significant change occurred in the pH values of the samples during the heat treatment period. When examining the amount of titratable acids, it becomes evident that the initial wine material subjected to heat treatment under oxygen-free (O_2-free) conditions underwent less change compared to heat treatment under oxygen-containing (O_2-rich) conditions. During the 3-month aging at 45–50°C, regardless of whether the initial wine material was heat-treated or not under O_2-free and O_2-rich conditions, a slight decrease in titratable acid content was observed. However, this decrease was more pronounced in samples heat-treated under oxygen-containing conditions.

During the 3-month storage period, although a decrease in titratable acids was observed, an increase in volatile acids was recorded. This increase was more significant in samples heat-treated under oxygen-containing conditions.

The content of sugars, phenolic compounds, and saturated esters in wine samples treated for 3 months at 45–50°C under O_2-rich and O_2-free conditions was investigated. During storage and aging, analyses were carried out on the samples every month. The results obtained regarding the changes in the amounts of sugars, phenolic compounds, and saturated esters are presented below (Table 2).

Table 1 – pH indicator and acid content in Marsala blend samples, n = 6, $p < 0.05$

Names and compositions of the studied experimental variants	pH			Titratable acidity, mg/dm^3			Volatile acidity, mg/dm^3		
	1 month	2 month	3 month	1 month	2 month	3 month	1 month	2 month	3 month
O_2-free initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	3,4	3,4	3,4	8,6	8,5	8,4	0,33	0,34	0,35
O_2-containing initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	3,4	3,4	3,4	8,6	8,5	8,3	0,40	0,43	0,45
O_2-free initial wine material, without heat treatment; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	3,4	3,4	3,4	8,3	8,2	8,2	0,36	0,37	0,37
O_2-containing initial wine material, without heat treatment; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	3,4	3,4	3,4	7,9	8,1	8,0	0,50	0,54	0,57
O_2-containing initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	3,4	3,4	3,4	7,9	8,1	8,0	0,47	0,49	0,53

Table 2 – Changes in the content of sugars, phenolic compounds, and saturated esters in different wine samples subjected to heat treatment over a 3-month period, n = 6, $p < 0.05$

Names and compositions of the studied experimental variants	Sugar, mg/dm^3			Phenol compound, mg/dm^3			Saturated esters, mg/dm^3		
	1 month	2 month	3 month	1 month	2 month	3 month	1 month	2 month	3 month
O_2-free initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	10,8	10,8	10,7	480	475	470	261,0	264,8	267,6
O_2-containing initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	10,8	10,7	10,6	540	490	470	176,0	216,8	282,4
O_2-free initial wine material, without heat treatment; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	10,8	10,8	10,8	960	960	950	450,0	452,8	458,0
O_2-containing initial wine material, without heat treatment; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	10,7	10,6	10,5	780	760	730	492,8	504,8	563,0
O_2-containing initial wine material, heat-treated; Rk 83% (18% alcohol) + 7% mistelle + 10% concentrated juice	10,8	10,7	10,6	610	580	556	422,4	441,6	480,8

It is evident from Table 2 that during the aging period, a decrease in sugar content, an increase in the amount of saturated esters, and a decrease in phenolic compounds were observed, particularly in samples subjected to heat treatment under oxygen-containing conditions. In the samples aged for 3 months at 45–50°C under both oxygen-containing and oxygen-free conditions, the content of nitrogen–aldehyde compounds was investigated (Table 3).

As can be seen (Table 3), in samples subjected to heat treatment under both oxygen-containing (O_2-rich) and oxygen-free (O_2-free) conditions, a decrease in the content of total nitrogen and amino nitrogen, as well as an increase in the content of aldehydes, was observed. These changes were more pronounced during heat treatment under oxygen-rich conditions. This may be attributed to the more active participation of oxygen in oxidation–reduction reactions during heat treatment and the transformations that occur [26].

As can be seen, the content of total nitrogenous compounds in the initial samples varied between 263.2–486.4 mg/dm^3. When heat-treated under oxygen-containing conditions, this amount gradually decreased over the months, reaching 231.2–453.6 mg/dm^3 after 3 months. The content of amino nitrogen in the initial samples ranged between 50.4–74.8 mg/dm^3, and under oxygen-containing heat treatment, this value steadily decreased over time, reaching 46.6–54.6 mg/dm^3 after 3 months. An increase in aldehyde content was observed, and this increase was more intensive under oxygen-containing conditions, while it was relatively weaker under oxygen-free conditions. A similar situation was also observed in the content of saturated esters.

The effect of heat aging of Marsala blend samples with different additives on physicochemical composition indicators

The physicochemical composition of Marsala blend components obtained by different technological methods, as well as samples aged up to 12 months at 45–50°C with and without nitrogen and oxygen addition, was investigated, and the resulting changes were studied. The results regarding the variation of nitrogenous compounds, especially total nitrogen and amino nitrogen, in different wine materials under O_2-rich and O_2-free conditions over a 12-month period are presented (Table 4 and Figure 1).

Table 3 – Changes in the content of nitrogen–aldehyde compounds in wine samples subjected to heat treatment over a 3-month period, n = 6, $p < 0.05$

Names and compositions of the studied experimental variants	Total nitrogen, mg/dm^3			Amino nitrogen, mg/dm^3			Aldehydes, mg/dm^3		
	1 month	2 month	3 month	1 month	2 month	3 month	1 month	2 month	3 month
O_2-free initial wine material, heat-treated; Rk 83% (18% vol alcohol) + 7% mistelle + 10% concentrated juice	263,2	261,2	253,7	50,4	49,8	48,4	111,0	113,0	117,0
O_2-containing initial wine material, heat-treated; Rk 83% (18% vol alcohol) + 7% mistelle + 10% concentrated juice	291,2	249,0	231,2	74,8	52,4	46,6	142,0	163,0	166,0
O_2-free initial wine material, without heat treatment; Rk 83% (18% vol alcohol) + 7% mistelle + 10% concentrated juice	425,6	424,8	425,1	63,6	64,8	62,7	150,0	152,0	153,4
O_2-containing initial wine material, without heat treatment; Rk 83% (18% vol alcohol) + 7% mistelle + 10% concentrated juice	486,4	472,0	453,6	63,6	55,3	54,6	161,0	170,0	179,0

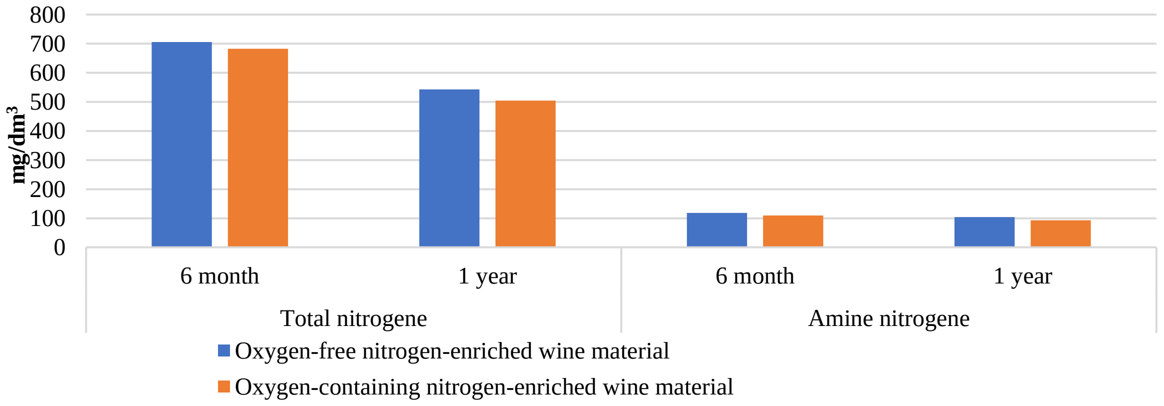

Figure 1. Changes in the content of nitrogenous compounds in blend samples during aging at different time intervals, n = 6, $p < 0.05$

Table 4 – Changes in the content of nitrogenous compounds in aged blend samples, $n = 6$, $p < 0.05$

Names of the studied experimental variants	Total nitrogen, mg/dm^3		Amino nitrogen, mg/dm^3	
	6 month	1 year	6 month	1 year
O_2-containing Rk 83% + 7% mistelle + 10% concentrated juice	476,0	381,0	116,0	102,0
O_2-free wine material at 18.1% vol alcohol, 8.6 sugar condition	408,0	404,8	105,0	101,3
O_2-containing wine material at 18.1% vol alcohol, 8.6 sugar condition	325,0	290,0	106,0	94,6
O_2-free nitrogen-enriched wine material	705,6	703,2	118,0	114,0
O_2-containing nitrogen-enriched wine material	683,0	504,0	109,0	93,0
O_2-free pomace-fermented wine material	722,0	699,8	104,8	102,4
O_2-containing pomace-fermented wine material	588,0	468,8	102,9	86,5
O_2-free pomace-fermented wine material	509,6	502,5	102,9	101,8
O_2-containing wine material obtained by heat treatment of pomace fermentation	459,8	385,0	101,5	93,2
O_2-free nitrogen-enriched wine material 60% + 30% mistelle + 10% concentrated juice	677,6	669,2	160,4	156,3
O_2-containing nitrogen-enriched wine material 60% + 30% mistelle + 10% concentrated juice	606,4	485,2	146,2	111,3
O_2-free heat-treated wine material 60% + 30% mistelle + 10% concentrated juice	627,2	564,5	151,7	148,0
O_2-containing heat-treated wine material 60% + 30% mistelle + 10% concentrated juice	660,8	485,6	148,0	111,5

It was found that (Table 4 and Figure 1), during a 1-year storage period, the content of total nitrogen and amino nitrogen decreased. These changes were observed to be more intensive under oxygen-containing conditions.

The research results related to the changes in the content of saturated esters and aldehydes in different samples aged for 12 months are presented below (Table 5, Figure 2).

It was found that (Table 5 and Figure 2), during a 1-year storage period, changes occurred in the content of saturated esters and aldehydes, and their levels increased as the storage period increased. These changes were observed to be more intensive under oxygen-containing conditions. A similar situation was also observed in samples enriched with nitrogen and aged for 1 year under both oxygen-containing and oxygen-free conditions.

Investigation of the effect of certain factors on quality safety in Marsala wines. In recent years, attention has increased regarding the potential carcinogenic effects of 5-hydroxymethylfurfural on human health. This compound is used as an indicator of honey adulteration and as a marker of heat treatment of sugar-containing foods. 5-hydroxymethylfurfural (HMF) is a water-soluble heterocyclic aldehyde and is usually present in small amounts in fresh foods containing sugars. HMF is formed during thermal processing of foods as a result of acid-catalyzed dehydration of carbohydrates, the Maillard reaction, or thermal caramelization. The formation of HMF is promoted by fructose, sucrose, and to a lesser extent glucose, from which mainly furfural is also formed.

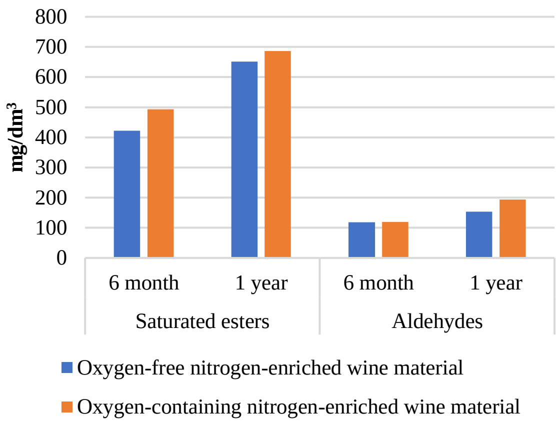

Figure 2. Changes in the content of saturated esters and aldehydes in blend samples aged under oxygen-containing and oxygen-free conditions, $n = 6$, $p < 0.05$

Considering the carcinogenic potential of hydroxymethylfurfural (HMF), it is important in Marsala wine production to investigate sugar transformations during heat treatment and the resulting formation of compounds such as HMF, furfural, and furan-2-carboxylic acid. Marsala wine samples prepared under the following variants were studied:

I – Control:
1.1. Traditional method;
1.2. Traditional method + concentrated juice;
II – Experimental:
2.1. Electrothermal treatment;
2.2. Electrothermal treatment + concentrated juice.

The results of the analyses are presented below (Table 6, Figure 3).

Table 5 – Content of saturated esters and aldehydes in different blend samples, n=6, $p<0.05$

Names of the studied experimental variants	Saturated esters, mg/dm^3		Aldehydes, mg/dm^3	
	6 month	1 year	6 month	1 year
O_2 with Rk 83% + 7% mistelle + 10% concentrated juice	402,0	426,4	114,4	137,8
O_2-free 18.1% vol, 8.6 sugar condition	299,2	315,2	70,4	77,0
O_2 with 18.1% vol, 8.6 sugar condition	352,0	457,6	99,4	115,0
O_2-free nitrogen-enriched wine material	422,4	498,2	118,8	144,0
O_2 with nitrogen-enriched wine material	492,8	586,4	119,7	153,6
O_2-free pomace-fermented wine material	352,8	333,6	66,9	69,9
O_2 with pomace-fermented wine material	316,8	406,0	76,6	110,0
O_2-free pomace-fermented wine material	369,6	314,0	77,4	91,8
O_2 with wine material obtained by heat treatment of pomace	316,8	484,0	103	129,8
O_2-free nitrogen-rich wine material 60% + 30% mistelle + 10% concentrated juice	369,6	348,0	96,8	124,6
O_2 with nitrogen-rich wine material 60% + 30% mistelle + 10% concentrated juice	404,8	524,0	99,4	149,6
O_2-free heat-treated wine material 60% + 30% mistelle + 10% concentrated juice	334,4	376,0	77,4	83,0
O_2 with heat-treated wine material 60% + 30% mistelle + 10% concentrated juice	316,8	438,0	83,6	122,0

Table 6 – Levels of furan aldehydes and sugars in Marsala wine samples

Marsala wine samples	Hydroxymethylfurfural (HMF), mg/dm^3	Furfural (F), mg/dm^3	Furan-2 carboxylic acid (FT), mg/dm^3	Glucose, g/dm^3	Sucrose, g/dm^3	Fructose, g/dm^3
Control Traditional method	87	0,8	2,1	21	1,6	23
Traditional method + concentrated juice	177	3,9	46	33	2,5	39
Experimental Electrothermal treatment	64	0,6	1,7	21	1,7	23
ETT + concentrated juice	97	1,8	8,5	34	2,7	39,2

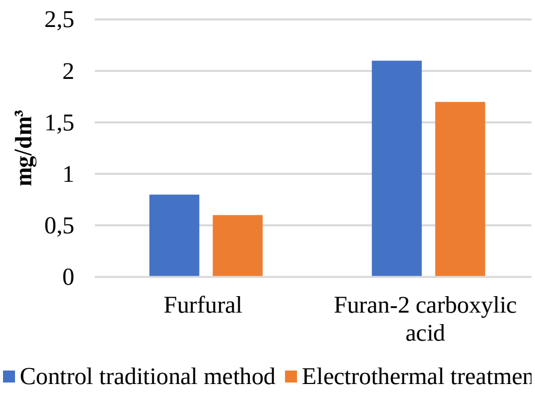

Figure 3. Changes in the content of furfural aldehyde and furan-2-carboxylic acid in Marsala wines produced by different meth

As can be seen from the table and figure, the control samples were analyzed in two groups. In both cases, the samples were prepared by the traditional method, i.e., conventional heat treatment under production conditions. In the first group, concentrated juice was not added, while in the second group, concentrated juice was added during blending.

In the experimental wine samples, unlike the control, heat treatment was carried out by electrothermal processing. It was found that, compared to the control samples, the amounts of hydroxymethylfurfural (HMF), furfural, and furan-2-carboxylic acid were significantly lower in the experimental samples. If in the traditional control variant the HMF was 87 mg/dm^3, furfural 0.8 mg/dm^3, and furan-2-carboxylic acid 2.1 mg/dm^3, in the first experimental sample produced by electrothermal treatment these values were 64 mg/dm^3, 0.6 mg/dm^3, and 1.7 mg/dm^3, respectively.

No significant difference was observed in sugar content between both variants. Thus, despite similar amounts of glucose, fructose, and nearly similar sucrose levels, the differences in the formation of HMF, furfural, and furan-2-carboxylic acid are related to the method of heat treatment.

A similar situation was also observed in Marsala wines prepared with concentrated juice added to the blend. In this case, in the control variant, the HMF content was 170 mg/dm³, furfural 3.9 mg/dm³, and furan-2-carboxylic acid 46 mg/dm³, whereas under electrothermal treatment these values were 97 mg/dm³, 1.8 mg/dm³, and 8.5 mg/dm³, respectively.

No significant difference in sugar content was observed in both variants. It was found that the addition of concentrated juice to the blend significantly increased the levels of glucose, fructose, and sucrose, which in turn influenced and promoted a considerable increase in HMF, furfural, and 2-furan carboxylic acid.

To clarify the effect of total sugar content on the formation of HMF and other indicators in Marsala wines, these parameters were studied in samples prepared under four variants differing in sugar content (Table 7).

As seen from the table, the samples, which are almost identical in terms of alcohol content by volume, differ from each other only in sugar content. In the samples with a sugar content of 4%, the amount of HMF was 65 mg/dm³, furfural 1.8 mg/dm³, and furan-2-carboxylic acid 2.4 mg/dm³, whereas in the 4th variant with a sugar content of 10%, these values were 190, 4.7, and 3.9 mg/dm³, respectively. In other words, an increase in sugar content led to a significant rise in the levels of the mentioned compounds.

The indicated parameters were also analyzed in juice samples concentrated at different temperatures. Concentration was carried out for the same duration but at different temperatures. According to the variants, the juice samples were concentrated by boiling at 40°C, 60°C, and 80°C for 3 hours (Table 8).

As seen from the table, during boiling at lower temperature, the amount of sugars was lower than in other variants, while with increasing temperature the sugar content increased. At the same time, at lower temperature the levels of HMF, furfural, and furan-2-carboxylic acid were relatively low, whereas at higher temperature their amounts also increased. If at 40°C the boiled samples contained 2.8 g/kg HMF, 0.05 g/kg furfural, and 0.21 g/kg furan-2-carboxylic acid, then at 80°C these values increased to 3.3 g/kg, 0.08 g/kg, and 0.82 g/kg, respectively. From this, it can be concluded that both sugar content and temperature play a fundamental role in the formation of HMF, furfural, and furan-2-carboxylic acid. Compared to lower temperatures, the formation intensity of these compounds increases at higher temperatures. Therefore, we consider it very important to control the preparation of concentrated juice used in Marsala wine production and to ensure quality management.

Conclusion

The physicochemical composition indicators of Marsala blend wine samples aged for three months at 45–50°C were investigated under oxygen-containing and oxygen-free conditions, as well as depending on whether the initial wine material was heat-treated or not. During the aging period, a decrease in sugar content, an increase in saturated esters, and a decrease in phenolic compounds were observed, particularly in samples subjected to initial heat treatment under oxygen-containing conditions. The content of total nitrogenous compounds in the initial samples varied between 263.2–486.4 mg/dm³, and under oxygen-containing heat treatment this value gradually decreased over the months, reaching 231.2–453.6 mg/dm³ after 3 months. The content of amino nitrogen in the initial samples ranged between 50.4–74.8 mg/dm³, and under oxygen-containing heat treatment this value also steadily decreased to 46.6–54.6 mg/dm³ after 3 months. An increase in aldehyde content was observed, which was more intensive under oxygen-rich conditions, while it was weaker under oxygen-free conditions. A similar situation was also observed in the content of saturated esters.

Table 7 – Effect of sugar content on the formation of furan aldehydes in Marsala wine

Names of samples	Sugar, %	Alcohol, % vol	HMF, mg/dm^3	Furfural, mg/dm³	Furan-2 carboxylic acid (FT), mg/dm³
Rkatsiteli-1	4	18,1	65,8	1,8	2,4
Rkatsiteli-2	6	18,0	96	2,0	1,8
Rkatsiteli-3	8	18,4	140	3,1	2,8
Rkatsiteli-4	10	18,1	190	4,7	3,9

Table 8 – Levels of furan aldehydes and sugars in concentrated juice samples

Variants	Content, g/kg					
	HMF	F	Furan -2	Glucose	Sucrose	Fructose
40°C for 3 hours	2,8	0,05	0,21	163	14	216
60°C for 3 hours	3,0	0,07	0,23	220	15	238
80°C for 3 hours	3,3	0,08	0,82	260	21	316

The physicochemical composition of Marsala blend components obtained by different technological methods, as well as samples aged up to 12 months at 45–50°C with and without nitrogen and oxygen addition, was investigated. The results showed that during this period the content of total and amino nitrogen decreased, while volatile acids, esters, and aldehydes increased in different wine materials under O_2-containing and O_2-free conditions. As a result, a quality close to classical Marsala-type wine is formed. Under such conditions, within 12 months a dark amber color, a soft balanced taste, and a vanilla-like aroma develop. During oxygen-free aging, a moderate darkening of color, weak oxidation tones in aroma, and slight burnt notes in taste are formed, indicating weaker oxidation processes.

Blend samples were subjected to heat treatment using both traditional and electrothermal methods. It was found that compared to the control samples, the experimental samples contained significantly lower amounts of hydroxymethylfurfural (HMF), furfural, and furan-2-carboxylic acid. In the traditional control variant, HMF was 87 mg/dm³, furfural 0.8 mg/dm³, and furan-2-carboxylic acid 2.1 mg/dm³, whereas in the first experimental sample prepared by electrothermal treatment these values were 64 mg/dm³, 0.6 mg/dm³, and 1.7 mg/dm³, respectively. The addition of concentrated juice to the blend significantly increased the levels of glucose, sucrose, and fructose, which in turn affected the previously mentioned indicators, leading to a considerable increase in HMF, furfural, and furan-2-carboxylic acid. In samples with 4% sugar, HMF was 65 mg/dm³, furfural 1.8 mg/dm³, and furan-2-carboxylic acid 2.4 mg/dm³, whereas in the 4th variant with 10% sugar these values were 190, 4.7, and 3.9 mg/dm³, respectively. Thus, an increase in sugar content led to a significant increase in these compounds. A similar trend was also observed with increasing temperature. Therefore, we consider it essential to strictly control the preparation of concentrated juice used in Marsala wine production and to ensure quality management.

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