Vacuum distillation in the technology of non-alcoholic wines from Isabella grapes

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Abstract

Introduction. The aim of the study was to develop the technology of non-alcoholic wine from Isabella grapes by vacuum dealcoholization with maximum preservation of varietal sensory properties of the initial wine material and harmonization of taste by adding grape must cryoconcentrate.

Materials and methods. The wine material was unprocessed dry red from the Isabella grape variety (Ukraine) cryoconcentrate of grape must. Dealcoholization was carried out using vacuum distillation. Physico-chemical indicators of dealcoholized wine were determined using chromatography. Sensory properties were evaluated using special methods.

Results and discussion. In the process of dealcoholization, the content of ethyl alcohol decreased from 9.8% to 0.4% by volume, which was accompanied by a decrease in the concentration of volatile acids, aldehydes, and esters while preserving a significant part of phenolic compounds (catechins, proanthocyanins), important for the antioxidant activity of the drink. Studies have shown that vacuum distillation reduces not only the content of ethanol, but also methanol by up to 30%, which increases the level of safety of non-alcoholic wine consumption.

Dealcoholization by vacuuming leads to an increase in the concentration of organic acids, tartaric and malic, which negatively affects the taste characteristics. To correct the taste properties, cryoconcentrate of grape must was added to the dealcoholized wine in different dosages. Sensory evaluation, carried out according to the methodology of the International Organization of Grapes and Wine, made it possible to note that this approach contributed to obtaining the taste harmony of the drink. The sweetness of the drink reduced the feeling of excessive acidity, which helped to balance the taste of non-alcoholic wine.

Conclusions. The results confirmed that the application of vacuum distillation in combination with the use of grape must cryoconcentrate is an effective way of obtaining non-alcoholic wine with pleasant sensory characteristics, ensuring its appeal to consumers and preservation of useful properties.

Introduction

Non-alcoholic wine Dealcoholization Sensory indicators Vacuum distillation Cryoconcentrate

The global non-alcoholic wine market is over USD 10 billion and is estimated to still grow at a significant CAGR (compound annual growth rate) of over 7% between 2019 and 2027, reaching a revenue share of over USD 30 billion (Sarkissian and Liganenko, 2020).

The first references to the production of alcohol-free wines date back to 1869, when the American Thomas Bramwell Welch, being a strong supporter of the movement for the moderation of alcohol consumption, produced this product by the pasteurization method (Sawler et al., 2013). Later, in 1908, the German scientist Carl Jung patented the technology of alcohol-free wine by applying a vacuum to lower the distillation temperature to 35 °C (Gornostay, 2011).

Dealcoholization of wine can be achieved by several methods. These methods can be classified into three groups based on the principle or mechanism of ethanol reduction and removal at various stages of wine production, including reduction of fermentable sugars (pre-fermentation stage), reduction or limitation of ethanol production (fermentation stage), and ethanol removal by membrane separation or heat treatment (post-fermentation stage) (Uspalenko et al., 2024).

In literary sources, there are studies of various ways of reducing the alcohol content in wines. The simplest of them is diluting the wort with water or adding juice from unripe grapes to the wort, which is called "green wort". In countries such as South Africa, New Zealand, Australia, and the USA (except California), water is allowed only as an aid in the preparation of stabilizing materials for the processing of wine materials, and the use of "green must" leads to negative changes in the sensory quality of wines (Kroiruddin et al., 2018).

To reduce the fermentable sugars in the wort, juice filtration is used using nanofiltration, ultrafiltration or reverse osmosis membranes, which have very small pore sizes and can retain sugar. At the same time, these methods affect the reduction of the content of polyphenols, anthocyanins and color intensity, which negatively affects the sensory properties and biological value of wine (Blackman et al., 2017).

Another way to reduce alcohol in wine is the use of glucose oxidase, which reduces glucose in grape juice before fermentation (Ebert et al., 2016). The producer of the enzyme is the fungus *Aspergillus niger.* Glucose oxidase converts β-D-glucose to D-gluconolactone in the first step of the reaction, releasing hydrogen peroxide, and catalyzes the conversion of D-gluconolactone to gluconic acid in the second step of the reaction. These reactions cause the oxidation of glucose in the must, which, accordingly, leads to a decrease in the alcohol level in the wine. The increased content of gluconic acid gives the wine excessive acidity and has a negative effect on the manifestation of fruit aroma and the reduction of varietal characteristics of the aroma (Di Renzo et al., 2014).

The use of yeast of the genus *Metschnikowia* or modified yeast strains with a low ability to produce alcohol contributes to the reduction of alcohol content by synthesizing a higher content of secondary fermentation products. This method only partially reduces the level of ethyl alcohol in wine (Suo et al., 2019).

Hydrophobic absorbents such as zeolites can also absorb ethanol from wine by absorption and filtration. This method can be used to produce alcohol-free wines with an ethanol content of up to 0.5% by volume (Akyerenko et al., 2021). But extraction methods of alcohol reduction are expensive and rarely used in the production of low-alcohol and alcohol-free wines.

Modern methods of dealcoholization of wines include vacuum and osmotic distillation, nanofiltration, and reverse osmosis (Cachon et al., 2006; Schelezki et al., 2020). These techniques minimize the loss of important volatile aromatic compounds.

The formation of harmonious sensory characteristics in non-alcoholic wines during dealcoholization or the use of other methods that contribute to reducing the alcohol content and harmonizing the balance is extremely important.

The purpose of the research was to develop the technology of non-alcoholic wine from Isabella grapes by vacuum dealcoholization with maximum preservation of varietal sensory characteristics of the initial wine material and harmonization of taste by adding cryoconcentrate of grape must.

Materials and methods

Materials

Unprocessed dry red wine made from grapes of the Isabella variety, cultivated in the Kyiv region, with an alcohol content of 9.8% vol. and cryoconcentrate of grapes must with a sugar content of 50-55%.

Experimental setup and dealcoholization conditions

Dealcoholization was carried out by vacuum distillation. Usually, in this method, the wine is placed in a strong vacuum. The wine was gently heated to boil off the alcohol at temperatures between 20 °C and 38 °C, maintaining a pressure of less than 0.1 bar, since vacuum is crucial for effective dealcoholization while preserving the initial sensory properties of the wine material (Figure 1) (Uspalenko et al., 2024).

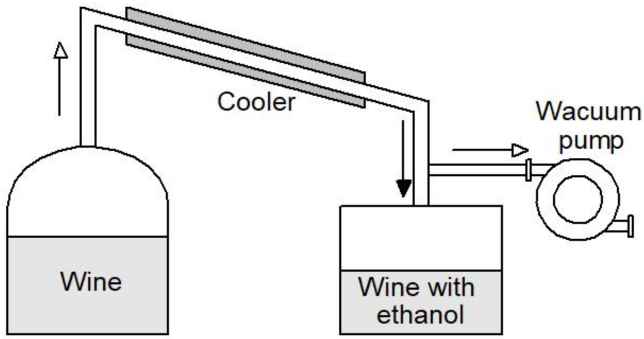

Figure 1. Scheme of the vacuum distillation method

But,since the Isabella grape variety is known for its high methanol content, dealcoholization was carried out in a vacuum apparatus, maintaining a pressure of 6 kPa and heating the wine to a temperature of 65 °C, under such conditions, the vapors passed into the gas phase and entered another tank, where they condensed and collected. Dealcoholization lasted 15 minutes, where 4.5 l of distillate and 13.5 l of dealcoholized wine were obtained from 18.00 l of wine. After that, the alcohol-free wine was filtered through laboratory filter paper.

To reduce acidity and to harmonize the taste of dealcoholized wine, a cryoconcentrate was added at the rate of 5-25 g/l and 30-80 g/l to bring non-alcoholic wine to the level of semi-dry and semi-sweet, respectively.

Setting up the experiment

During the experiment, the physico-chemical and sensory quality indicators of non-alcoholic wines obtained by the vacuum distillation method were investigated in comparison with the control sample, and a sensory analysis of the tested wine sweetened with cryoconcentrate to different levels of sweetness was also carried out.

- **control sample** (Control) – wine with an alcohol content of 9.8% vol.
- **sample 1** – dealcoholized wine by vacuum distillation with an alcohol content of 0.4% vol.
- **sample 2** – dealcoholized wine by vacuum distillation with an alcohol content of 0.4% vol. sweetened with cryoconcentrate up to 20 g/l
- **sample 3** – dealcoholized wine by the vacuum distillation method with an alcohol content of 0.4 % vol. sweetened with cryoconcentrate up to 75 g/l

At the first stage of the experiment, dealcoholized wine was produced by vacuum distillation. At the second stage, a physical and chemical analysis was carried out using the chromatography method. To harmonize the taste of dealcoholized wine, grape must cryoconcentrate was added to the level of semi-dry and semi-sweet wine.

The final stage was sensory analysis by a specialized tasting commission.

Determination of sensory properties

Tastings took place in a special room equipped with individual booths and air conditioning at 20°C. The testing was conducted by two independent tasting commissions.

For wine tasting, a standard tasting glass made of thin, clean, transparent glass with a capacity of 210-220 ml was used, which makes it possible to operate 60-70 ml of wine for a comprehensive sensory assessment of all elements of quality.

Wine with alcohol content and de-alcoholized wine were evaluated according to the Standards of the International Organization of Vine and Wine (Resolution OIV/Competition ECO 332A/200). The maximum tasting rating of the experimental samples was 100 points and was determined as the sum of points for each indicator: appearance (transparency, color) – 14; aroma (authenticity, intensity, quality of aroma) - 30; taste (authenticity, intensity, harmonic stability, taste quality) - 44; harmony (general impression) – 11.

To create the aromatic profiles of the experimental samples, a descriptive method was used according to a 10-point rating scale based on the following descriptors: citrus, fruity, nutmeg, floral, sweetness, acidity, bitterness, body/fullness, intensity, astringency.

Graphical representations of experimental data were performed using Microsoft Excel 2010.

Comparing the profiles of the experimental samples made it possible to determine their differences and draw conclusions about the change in wine quality during dealcoholization.

Determination of physical and chemical indicators

Determination of physical and chemical parameters was carried out in accordance with the methods and regulations of the International Organization of Grapes and Wine (OIV). Standard protocols and analytical methods recommended by the OIV were used to conduct the research, ensuring the accuracy and reproducibility of the results obtained.

Processing research results

Determination of physico-chemical indicators of non-alcoholic wine samples was carried out in two repetitions. Results are shown as mean ± standard deviation.

Statistical data processing was performed, including determination of mean content and standard deviation (±SD), with four replicates (four collections) for the main compounds identified in all four collections analyzed.

Results and discussion

Changes in the physical and chemical parameters of wine during dealcoholization

During dealcoholization of wine, complex physicochemical processes take place, in which, in addition to the removal of ethyl alcohol, other components of wine are also changed - such as volatile ones - aldehydes, acids, higher alcohols, ethers, acetates, organic acids, and amino acids (Diban et al., 2008). During the experiment, the alcohol content was reduced from 9.8% vol. (control) up to 0.4% vol. It should be noted that due to the removal of alcohol from wine, the concentration of wine components occurs, because of which the concentration of non-volatile compounds increases (Table 1).

Table 1

Physico-chemical parameters of dealcoholized wine from Isabella grapes

Indicator	Control	Sample 1
Ethanol content, % vol.	9.80 ± 0.40	0.40 ± 0.02
Mass concentration of sugars, g/l:	3.00 ± 0.15	4.20 ± 0.20
Organic acids, mg/l:		
titrated acids	6.10 ± 0.30	7.80 ± 0.30
volatile acids	0.20 ± 0.01	0.15 ± 0.01
tartaric acid	4.90 ± 0.20	4.20 ± 0.20
citric acid	0.015 ± 0.007	0.010 ± 0.005
malic acid	1.00 ± 0.05	2.90 ± 0.15
Alcohols, mg/l:		
methanol	84.00 ± 4.20	25.20 ± 1.26
glycerol	3020.0 ± 151.00	3020.0 ± 151.00
acetaldehyde	19.00 ± 0.95	16.00 ± 0.80
ethyl acetate	42.00 ± 0.21	40.00 ± 0.20
higher alcohols (isoamyl and isobutyl)	309.70 ± 15.28	301.30 ± 15.06
furfural	2.80 ± 0.15	2.00 ± 0.10
5-hydroxymethylfurfural	13.20 ± 0.66	12.90 ± 0.65
Hydroxymethylfurfural (HMF), mg/l	2.50 ± 0.12	2.10 ± 0.10

The total content of organic acids undergoes significant changes during dealcoholization, this is due to the change in concentration caused by the removal of ethanol and water. Organic acids are an important group of compounds in wines because they affect the physicochemical and microbiological stability of wines, as well as their sensory properties (Coelho et al., 2018).

Content of titrated acids increased by 1.28 times. At the same time, the concentration of representatives of organic acids varied in different ways. The content of volatile acids decreased by 1.33 times, which is characteristic of dealcoholization. The mass concentration of tartaric acid decreased by 1.17 times and citric acid by 1.5 times, while the content of malic acid increased by 2.9 times. Such transformations lead to a change in the general taste profile of non-alcoholic wine, which is expressed in an imbalance of taste and disharmony in the sensory perception of wine. Thus, some researchers noticed that dealcoholized wines have higher acidity and less bitterness compared to the original (control) sample (Corona et al., 2019).

However, it should be noted that organic acids have the ability to interact with alcohols, forming complex esters that add a rich aroma and taste to wines. They create harmony of taste, which is extremely important for consumers' perception of wine quality.

A slight decrease, compared to other acids, was observed in tartaric acid, which helps maintain acidity, lower pH, inhibit bacterial growth and preserve freshness in wine for a long time. In addition, tartaric acid is necessary to improve the structure and flavor profile of wine (Yang, 2021).

As shown in Table 1, malic acid increased 2.9-fold during dealcoholization. This can lead to the formation of lactic acid, which negatively affects the stability of the wine and can introduce an unpleasant bitter taste. Therefore, it is important to control the level of malic acid during dealcoholization.

Concentrations of volatile substances also changed after dealcoholization of wines. For example, when applying vacuum distillation, the content of acetaldehyde slightly decreased by 16%. It should be noted that aldehydes play an important role in the formation of sensory indicators of wine (aroma, taste). One of the main aldehydes formed during fermentation is acetaldehyde. Increasing the content of this aldehyde gives the wine a sharp smell with hints of toasted bread, but the decrease resulted in less expressive fruit aromas and flavors, making it less saturated and complex in sensory perception (Sam et al., 2021).

The dealcoholization process also affects other volatile compounds that affect the aroma and taste of the wine. Wine contains more than 1000 volatile compounds of various chemical classifications (alcohols, esters, fatty acids, aldehydes, terpenes, ketones, sulfur compounds), where about 400 volatile compounds are formed during wine fermentation (Esteras-Saz et al., 2021). Higher alcohol in wine are formed in the process of alcoholic fermentation, which is carried out by yeast. They are by-products of yeast metabolic processes. Higher alcohol can be formed from amino acids by the so-called Ehrlich pathway. During this process, amino acids are deaminated to their corresponding aldehydes, which are then reduced to higher alcohol.

A representative of monoatomic alcohols is methyl alcohol, which is synthesized before and during alcoholic fermentation due to the hydrolysis of pectins by methyl-pectinesterase, which is contained in grapes. Pectinase catalyzes the cleavage of ester bonds in pectin, which leads to the formation of methyl alcohol and pectic acid (Kucherenko and Bilko, 2020). It is known that methanol is a toxic substance that can accumulate in the body and have a harmful effect on various organs and systems. In red wines, the methanol content is up to 250 mg/l, the results of the study showed that the methanol content in the control sample was 84.00 mg/l, which is 33.6% less than the maximum permissible norm, and the methanol content in the non-alcoholic sample is even lower - 25.2 mg/l. This indicates that methanol is removed along with ethyl alcohol during the wine dealcoholization process.

Other indicators of volatile compounds, such as ethyl acetate and higher alcohols, remain almost unchanged, demonstrating the stability of the wine's aroma after treatment.

The concentration of hydroxymethylfurfural (HMF) decreased by 1.19 times 2.1 mg/l versus 2.5 mg/l, and the mass concentration of higher alcohols decreased by 1.03 times 301.3 mg/l in non-alcoholic wine versus 309.7 mg/l. The content of furfural decreased by 1.4 times to 2.0 mg/l versus 2.8 mg/l, and 5-hydroxymethylfurfural decreased by 1.02 times to 12.9 mg/l in the non-alcoholic sample versus 13.2 mg/l. Hydroxymethylfurfural and furfural, which are markers of heat treatment, have slightly reduced concentrations in the dealcoholized sample, which indicates the preservation of the naturalness of the product.

Therefore, when switching to a non-alcoholic version of wine, there are both significant increases in individual indicators and decreases in other parameters, which can significantly affect the sensory properties of the product.

Changes in sensory parameters of wine during dealcoholization

During the study, slight changes in the color of the wine were recorded compared to the initial (control) sample. Sample 1, obtained by the method of vacuum distillation, retained its deep red color with a purple tint and transparency, but also added a light brick shade, which was formed during dealcoholization (Figure 2).

Control **Sample 1**

Figure 2. Comparison of the color of the control sample with the dealcoholized sample

However, the greatest impact of dealcoholization was found on the aromatic and taste characteristics of the wine, which emphasizes the importance of choosing processing methods and improving the balance to preserve the sensory properties of the product. The results of the comparative sensory analysis of the studied wine made it possible to establish their sensory characteristics (Figure 3).

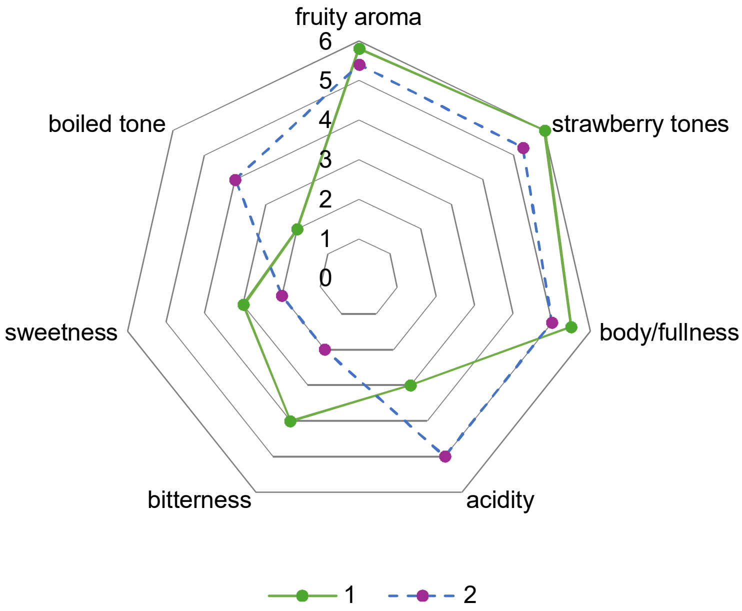

Figure 3. Profilogram of red wine from the Isabella grape variety:
1 — alcoholic wine (control), 2 — dealcoholized wine (sample 1)

A careful analysis of the changes in the main descriptors of the experimental samples made it possible to establish the following. Dealcoholization of wine is usually associated with the loss of volatile aromatic components (Kumar et al. 2024). On the other hand, decreasing the ethanol level improves the perception of aromatic components, which can compensate for the loss of aroma (Goldner et al. 2009). In this case, both effects probably resulted in the perception of the strawberry flavor not changing. The processed wine showed a similar expression of fruit tones. Dealcoholized samples are usually described as having a slightly reduced but slightly altered intensity of this descriptor.

The investigated dominant aromatic properties, such as strawberry tones, were evaluated almost equally for the dealcoholized wine and did not differ significantly from the original untreated wine. Perception of sweet was clearly altered because of dealcoholization. The de-alcoholized wines had significantly less sweetness compared to the original wine, but the dealcoholized wine sweetened with cryoconcentrate had a higher perceived sweetness (Figure 4).

Based on this, it becomes clear why non-alcoholic wines are usually produced with an increased level of sugar, that is, semi-dry or semi-sweet. To compensate for this change due to dealcoholization, a cryoconcentrate was added to sweeten the wine to a semi-dry 25 g/l, thereby rebalancing the balance. Acidity perception was rated significantly higher for still wines without sweetening. Therefore, when choosing wines for dealcoholization, this factor should be taken into account and wines with lower-than-normal acidity values should be chosen to obtain a balanced product or sweeteners such as cryoconcentrate should be added.

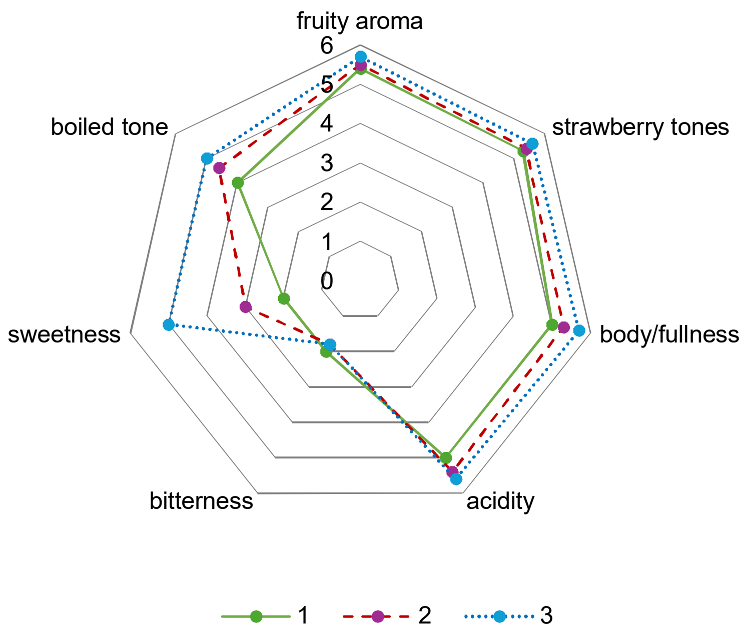

Figure 4. Profilogram of non-alcoholic red wines from grapes of the Isabella variety with the addition of cryoconcentrate:
1 — dry (control without the addition of cryoconcentrate);
2 – semi-dry,
3 – semi-sweet

The bitterness of the original wine was significantly higher in the control wine compared to the dealcoholized variants. The direct effect of ethanol in wine is to increase bitterness.

The perception of body and fullness of wine is clearly correlated with alcohol content (Grainger, 2009). The higher the alcohol content, the higher this perception. The results of this study show a significant difference between the processed variant and the original wine. Since body and fullness are clearly considered positive sensory characteristics of wine, oenological strategies such as the addition of cryoconcentrate, mannoproteins or tannins can partially compensate for the reduction of these sensory parameters due to dealcoholization.

The study shows that dealcoholization of wine significantly affects its sensory characteristics. The sensory characteristics of the aromatic parameters were changed less than the attributes affecting the perception of the wine on the palate. The main effects of dealcoholization are consistent with the complex sensory characteristics of ethanol in wine.

Conclusions

1. The process of vacuum distillation allows you to effectively reduce the content of ethyl alcohol in wine to the level of 0.4% vol. A decrease in the concentration of volatile acids, aldehydes and esters is observed within limits that do not critically affect the overall quality of the product, ensuring its chemical stability.

2. As a result of the dealcoholization process, there is an increase in the concentration of organic acids, such as tartaric and malic, which creates a certain imbalance in the sensory properties of the drink.
3. Adding grape juice cryoconcentrate allows you to adjust the sensory properties of the drink, creating a balance between acidity and sweetness. This contributes to the formation of a pleasant taste profile, softening of bitterness and improvement of the aromatic component, which is confirmed by sensory evaluation.
4. The combination of vacuum distillation and the use of cryoconcentrate is a promising technology to produce high-quality non-alcoholic wine. The obtained results demonstrate the possibility of creating a drink that meets modern consumer demands, preserves the benefits of natural components and provides an attractive taste profile.

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