

# Research on the Production of Rose Wines from Madrasa and Hindogna Grape Varieties

E.E.Heydarov, Kh.K.Fataliev, A.M.Alekberov, N.S.Qadimova, K.F.Imanova, Sh. Kh. Fataliyeva\*

<sup>1</sup>Head of the Department of «Food Engineering and Expertise of the Azerbaijan State Agrarian University, Doctor of Technical Sciences, professor. Address: AZ 2006, Vezirov Street108, Ganja City, Republic of Azerbaijan

<sup>2</sup>Imanova Konul Fikrat, doctoral student, AZ1033Distric Montin. Dash Karkhana Str. 543 Baku Business and Corporation College

<sup>3</sup>Fataliyeva Shabnam Khasil, assistant AZ 2006 District Gulustan, block 36, apartment 5 Azerbaijan State Agrarian University Ganja, Republic of Azerbaijan

**\*Corresponding Author:** Sh. Kh. Fataliyeva, assistant AZ 2006 District Gulustan, block 36, apartment 5 Azerbaijan State Agrarian University Ganja, Republic of Azerbaijan.

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## Abstract

Rose wines were an important group of natural wines, characterized by unique quality indicators. It is notable not only for the organization of the transition between red and white wines, but also for the presence of a well-formed consumer layer. Therefore, the study of this type of wine is on the agenda. Since this group of wines has anthocyanins and apomas, which differ in quantity and quality from white and red, their research becomes relevant. In the course of research, rose wines were developed using the Madrasa and Hindogna grape varieties. For this purpose, the squeeze was without exposure (control) and the squeeze with exposure at different times – 3, 6, 12, 18 and 24 hours. Before maceration, a sulfurous treatment of the squeeze is carried out. The squeeze was mixed with the compressed and obtained juice fraction. The resulting juices were sulfitized at the rate of 50 mg/l and put on fermentation at room temperature. The resulting wine material is separated from the sediment by decanting and left alone.

General indicators of the composition and organoleptic evaluation of rose wines were carried out in accordance with the methods available in enochemistry. While the Madrasa grape variety allows for more refined wine samples, the samples obtained from the Hindogna variety featured a higher alcohol and extract content. If the ratio of leucoanthocyanins to anthocyanins in the samples of wine made by years from the madrasa variety, according to the average indicators for 3 years, ranged from 1.81-2.50 and the color intensity-from 0.33-0.47, then in the Hindogna varieties - from 1.87-2.16 and 0.40-0.52, respectively.

In the process of maceration from 3 to 24 hours in the squeeze, an increase in the amount of anthocyanins was observed. When considering the samples, the anthocyanin content in the sample obtained with 3-hour maceration was minimal, and with 6-and 12-hour maceration-optimal, and, finally, with 18 - and 24-hour maceration-maximum, that is, 86.02-91.35 mg/dm<sup>3</sup>. As for the quality, 14 representatives of anthocyanins were identified in the wine samples.

In the wine samples, 8 representatives of aromatic volatile phenols, 7 representatives of lactones and 5 representatives of 6-carbon compounds were found. Organoleptic analysis of rose wine samples obtained from the Madrasa grape variety with aging for 3, 6, 12, 18 and 24 hours was carried out. Samples of rose wine prepared by maceration for 3 and 6 hours were rated 0.6-0.8 points higher than their other counterparts.

**Keywords:** grapes, juice, rose wine, crushing, maceration, anthocyanins, lactones

## Introduction

Pink table wines are an independent group and have their own distinct quality characteristics. The proportion of anthocyanins in the total content of

phenolic compounds in wines of this type is 3-7 p.c. In pink wines, the group of phenolic compounds is mainly represented by monomeric flavonoids, the main part of which consists of leucoanthocyanins [1-

## Radiology Research and Diagnostic Imaging

3]. The ratio of leucoanthocyanins to anthocyanins is one of the important indicators that characterize rose wines. The growing demand for rose wines is determined by their attractive appearance, pleasant fresh taste, well-defined aroma and the fact that they better retain the fullness inherent in fresh grapes, compared to red wines [4].

23 aromatic substances were found in samples of pink juice from the madrasa grape variety grown at different heights above sea level. Of these, 6 were acids, 4 were grain alcohols, 4 were hexacarbon compounds, 3 were carboxylic compounds, 5 were volatile phenols, and 1 was a lactone compound. Cold processing of juices and wine materials affects the composition parameters, including the amount of aromatic compounds. In the experimental samples of wine, the representatives of grain alcohols with a higher content were isobutyl alcohol, isoamyl alcohol and 2-phenyl ethanol. It is known that 2-phenyl ethanol has a flower aroma and plays a special role as a flavoring agent in wine [5,6].

### Materials and methods of research

As an object of research, technological methods and means are used to take the grape variety, the extract obtained from it, the juice and the wine material. The local variety of Madrasa and Hindogna grapes grown in its different regions was used.

Grape varieties are harvested in a state of technical ripeness. The grape comb is separated, the berries are crushed, and suspended particles are separated from the resulting squeeze. Using these parts, experiments are performed taking into account different durations, temperatures, and other factors.

The experiments were carried out without exposure (control) and with exposure at different times - 3, 6, 12, 18 and 24 hours. Before maceration, a sulfurous treatment of the squeeze is carried out. The squeeze is mixed with the squeezed and obtained juice extract. The resulting juices were sulfitized at the rate of 50 mg/l and put on fermentation at room temperature. The resulting wine material is separated from the sediment by decanting and left alone.

General indicators of the composition and organoleptic evaluation of rose wines were carried out in accordance with the methods available in enochemistry [7,8]. At the same time, modern methods of analysis were used. To determine the anthocyanins, 6 ml of the juice or wine to be analyzed was passed through a SuperClean LC-18 cartridge and thus the anthocyanins were tarred. Then 18 ml of H<sub>2</sub>O-HCC (99.9/0.1; h/h) was passed from the cartridge and the sugars contained in the medium

were removed. At the same time, 12 ml of McOH-HCl (99.9/0.1; h/h) anthocyanins are added to this solvent, the tarring of which is carried out through the cartridge. After mixing the anthocyanins with the solvent, the resulting mixture is thickened in an evaporator until it dries at 250C. Then the anthocyanins stuck in the wall of the evaporator cylinder were dissolved in 1 ml of methyl alcohol/water/formic acid solvent (40/55/5:h/h/h) and injected into the HPLC, determining the amount and profile of the anthocyanins. While pre-configured skin extractors were injected directly, anthocyanin profiles were thus detected. HPLC-MS is used in the recognition of anthocyanin compounds. The standards of delphinidin, cyanidin, petunidin, pyonidin, and malvidin-3-glycoside were used to determine the amount of anthocyanins. For each standard, five different layers of solution were developed by introducing them into HPLC, calibration curves were established, and based on the obtained curves, the number of compounds was calculated and determined.

For the detection of anthocyanins, an Agilent-1100 HPLC with a double pump, a double long-wave and a diode array detector was used.

The modified method of Rio-Anmatella and his collaborators was used to determine the perfume combinations. Therefore, 5 ml of Na Cl is added and mixed within 30 seconds using the Vortex Type Mixer brand 1 CA MS3 basic. After 40 minutes of fiber exposure (65 µmPDMS / DVB (Supelco, Bellefonte, PA, USA)) At 400C, it was introduced into the GC-MS device (Shimadzu GCMS-QP2010). Before each introduction, the fiber was conditioned for 10 minutes at 2000C. Restec RTX-5 (30 m x 0.25 mm x 0.25 microns) was used as the capillary Kolumn, and helium was used as the carrier phase. The temperature of the capillary column is programmed in such a way that after waiting 5 minutes at 400C,

### Discussion of the results obtained

The indicators of the total composition of samples of rose wines made in different years from the grape varieties of Hindogna and Madrasa were studied. It turned out that the alcohol level, which plays an important role in the stability of wines, varied in the range of 11.5-12.5 h percent in the samples of madrasa wines and 12.5-13.1 h percent in Hindogna wines, respectively. As you know, recently the preference is given to the production of such wines with a higher viscosity. From this point of view, the viscosity of Hindogna wine samples can be considered more optimal (Tables 1 and 2).

**Table 1:** Total composition of wines by year (Hindogna grape variety)

Connections	Years		
	2018	2019	2020
Spirit, h p.c.	13.1	12.5	12.7
Titrateable acids, g/dm <sup>3</sup>	7.6	7.3	7.8
pH	3.16	3.11	3.22
Volatile acids, g/dm <sup>3</sup>	0.26	0.22	0.24
Index HCl	37.9	36.4	35.7
Gelatin index	53.4	56.2	53.5
Sugar, g/dm <sup>3</sup>	0.15	0.18	0.12
General SO <sub>2</sub> , mg/dm <sup>3</sup>	75.6	88.9	82.7
Free SO <sub>2</sub> , mg/dm <sup>3</sup>	15.4	15.1	16.2
The above extract, g/dm <sup>3</sup>	23.1	22.6	23.5
Ash, g/dm <sup>3</sup>	2.3	2.1	2.0
Color intensity (i)	0,41	0,52	0,40
Anthocyanins, mg/dm <sup>3</sup>	51	59	47
Leucoanthocyanins, mg/dm <sup>3</sup>	106	128	88
Leucoanthocyanins/anthocyanins	2,07	2,16	1,87

**Table 2:** Total composition of wines by year (Madrasa grape variety)

Connections	Years		
	2018	2019	2020
Spirit, h p.c.	12.1	11.5	12.5
Titrateable acids, g/dm <sup>3</sup>	6.8	6.5	7.0
pH	3.15	3.30	3.25
Volatile acids, g/dm <sup>3</sup>	0.31	0.28	0.25
Index HCl	27.3	28.4	25.6
Gelatin index	48.31	50.30	46.25
Sugar, g/dm <sup>3</sup>	0.11	0.15	0.17
General SO <sub>2</sub> , mg/dm <sup>3</sup>	91.4	88.9	90.0
Free SO <sub>2</sub> , mg/dm <sup>3</sup>	18.7	19.2	20.1
The above extract, g/dm <sup>3</sup>	20.3	19.8	21.7
Ash, g/dm <sup>3</sup>	1.7	1.5	1.8
Color intensity (i)	0,33	0,41	0,37
Anthocyanins, mg/dm <sup>3</sup>	48	63	54
Leucoanthocyanins, mg/dm <sup>3</sup>	122	119	98
Leucoanthocyanins/anthocyanins	2,54	1,89	1,81

It is known that the HCl index indicates the degree of polymerization. Usually, the HCl index ranges between 5-40 and approaches the maximum in wines with a high tannin content. As can be seen, in the sample of wine from the Hindogna grape variety, this indicator ranged from 35.7-37.9 and in the Madrasa varieties-25.6-28.4, respectively. And this indicates that the fermentation properties of Hindogna wine samples are higher than those of Madrasas.

The gelatin index, the price of which varies between 25-80, reflects the ability of tannin to react with protein. According to Ribero-Gaillon and his collaborators, a gelatin index of less than 40 indicates

a weak ability of tannin to react with proteins, and more than 60 indicates a high ability to react. The gelatin index in the Madrasa wine samples ranged from 46.25 to 50.30, and in the Hindogna wine samples - from 53.4 to 56.2. Apparently, the ability of tannin to react with proteins in the Hindogna wine materials was higher.

The amount of titrated acids varied in the range of 7.3-7.8 g/dm<sup>3</sup> depending on the year of harvest and slightly less than them in the samples of Madrasa, that is, 6.5-7.0 g/dm<sup>3</sup>. It is known that titrated acids give the wine freshness and aroma and play an important role in stability. The amount of acids varying in this interval was characterized by its positive effect and gave the wine a unique quality.

The above extract is one of the important indicators that reflect the completeness and typicality of the wine. According to this indicator, the samples of Madrasa and Hindogna wine also distinguished themselves. It turned out that the amount of the brought extract was 19.8-21.7 g/dm<sup>3</sup> in the samples of Madrasa wine and 22.6-23.5 g/dm<sup>3</sup> in Hindogni, respectively. And if you look at the years, a higher amount of extract for both varieties was recorded in 2020.

If the ratio of leucoanthocyanins to anthocyanins in the samples of wine made by years from the Madrasa variety, according to the average indicators for 3 years, ranged from 1.81-2.50 and the color intensity-from 0.33-0.47, then in the Hindogna varieties-from 1.87-2.16 and 0.40-0.52, respectively. The amount of anthocyanins changed depending on the technology used (Table 3).

As you can see, the amount of anthocyanins changed depending on the timing of maceration of the squeeze. Thus, during maceration from 3 to 24 hours, the amount of anthocyanins ranged from 57.22-91.35 mg/dm<sup>3</sup>. At the same time, it is noticeable that there is a pattern that with increasing maceration time, the number of anthocyanins also increases. The lowest amount of anthocyanins was in the control variant, 43.86 mg/dm<sup>3</sup>, followed by 61.22 mg/dm<sup>3</sup> when observed for 3 hours of maceration. When considering the samples, the anthocyanin content in the sample obtained with 3-hour maceration was minimal, with 6- and 12-hour maceration optimal, and, finally, with 18 - and 24-hour maceration-maximum, that is, 86.02-91.35 mg/dm<sup>3</sup>. A similar situation is observed in the samples of wines made from the Hindogna grape variety (Table 8). But unlike the previous one, there is a slight increase in the total number of anthocyanins. Thus, the total anthocyanin

## Radiology Research and Diagnostic Imaging

content in these wine samples ranged between 48.62-99.00 mg/dm<sup>3</sup>, while in the wine sample prepared without extract, the anthocyanins showed a minimum of 48.62 mg/dm<sup>3</sup>. With an increase in the aging period of the squeeze from 3 hours to 24 hours, an increase in the amount of anthocyanins was observed by about 100 p.c.

Naturally, an increase in the amount of anthocyanins provides a tendency to red color in wine samples, which is not desirable for high-quality rose wines.

Volatile phenolic compounds are known to play an important role in flavor. Apparently, the wine samples contained 8 names of volatile phenolic compounds (Table 5).

Of these, 2-methoxy-4-vinifenol, 4-vinifenol are relatively high; propiovaniline, acetovaniline, and homovaniline are medium; vanillin, zinferon, and 2,6-dimethoxyphenol are found in low amounts. But, despite the small amount, their role in flavoring is undeniable.

Among the aromatic compounds, lactones also occupy a special place. During the analyses, 7 representatives of lactones were found in the samples (table 6).

Among the lactones, the most noticeable in terms of quantity was  $\gamma$ -butyrolactone. Its amount varied between 621-686  $\mu\text{g}/\text{dm}^3$  on the samples. Approximately 80 p.c. of the total amount of lactones is accounted for only by this lactone. representatives of smaller amounts were  $\gamma$ -octalactone 6.5  $\mu\text{g}/\text{dm}^3$ , pantolactone -9.4  $\mu\text{g}/\text{dm}^3$ ,  $\delta$ -decalactone 3.7  $\mu\text{g}/\text{dm}^3$ ,  $\delta$ -octalactone  $\gamma$ -hexolactone 15.6  $\mu\text{g}/\text{dm}^3$ . 4-ethoxycarbonyl- $\gamma$ -butanolactone 27.6  $\mu\text{g}/\text{dm}^3$  and 4-(hydroxyl-ethyl)  $\gamma$ -butanolactone-76.3  $\mu\text{g}/\text{dm}^3$  they had an average position.

The analysis revealed 5 representatives of aromatic carbonyl compounds (table 7).

Of the carbonyl compounds, 3-hydroxy-2-butanone (acetoin) is present in an amount of 61.8-7.8  $\mu\text{g}/\text{dm}^3$ , which is about 55% of the total amount. Of the

carbonyl compounds, the smallest amount was 3-hydroxy-4-phenol-2-butanone, which was found in the range of 5.9-9.2  $\mu\text{g}/\text{dm}^3$ . The total amount of carbonyl compounds in the wine samples varied in the range of 111.8-138  $\mu\text{g}/\text{dm}^3$ .

The quantity and qualitative composition of colorless phenolic compounds in different regions were studied, depending on whether the madrasa grape variety was macerated in grinding or not. In the wine samples, 6 different compounds of the flavanol group were found, including catechin, epicatechin, procyanidins B1, B2, B3, B4. Catechin, epicatechin, and procyanidin B1 were distinguished by their amount. The amount of phenolic acids prevailed in the samples of wine from the Madrasa grape variety grown in both regions. But in terms of quantity, the advantage was mainly in the two acids. These are trans-succinic and trans-coumaric acids. The amount of these two compounds was about 90 p.c. of the total amount of colorless phenolic compounds. Organoleptic analysis of samples of rose wine taken from the grape variety "madrasa" with an aging of 3, 6, 12, 18 and 24 hours in crushing was carried out. The tasting was conducted according to 10-point system with the participation of 9 tasters. It turned out that a sample of rose wine prepared by aging for 3 hours became more noticeable due to its transparency and color. At the same time, this sample was valued higher for its taste, typicality, aroma and bouquet. The total score that this sample received was 9.6. In a sample of rose wine prepared by aging for 12 hours in crushing, there was a slight decrease in the intensity of color and taste.

Compared to the control, the tasting received a score of 0.2 points less. As a result, this sample received a score of 0.2 points higher than the control, but it turned out that it received a score of 0.6 points lower than the 3-hour storage option and 0.4 points lower than the 6-hour storage option. A sample of rose wine obtained by 24-hour aging in crushing was rated 0.8 points lower than the best sample.

**Table 3:** The amount of anthocyanins in samples of rose wine from the madrasa variety (mg/dm<sup>3</sup>)

Connections	Control (without squeeze time)	Squeeze holding time, hour				
		3	6	12	18	24
Delphinidin-3-glycoside	0,36	1.60	3.16	5.01	5.95	6.46
Cyanidin-3-glycoside	1,38	2.02	2.46	2.95	3.16	3.22
Petunidin-3-glycoside	0,47	1.07	2.21	3.03	8.67	7.15
Peonidin-3-glycoside	8,51	10.32	10.15	11.56	11.96	12.07
Malvidin-3-glycoside	31,98	38.22	42.86	48.95	53.46	57.09
Delphinidin-3-glycosyl acetates	0,00	0.45	0.67	0.71	0.73	0.74
Cyanidin-3-glycosyl acetates	0,00	0.38	0.35	0.41	0.41	0.43



Petunidine-3-glycosyl acetates	0,00	0.32	0.45	0.49	0.47	0.51
Peonidin-3-glycosyl acetates	0,15	0.17	0.19	0.16	0.14	0.18
Malvidin-3-glycosyl acetates	0,79	0.94	1.27	1.35	1.41	1.53
Delphinidin-3-glycoside-p-coumarate	0,00	0.22	0.23	0.21	0.21	0.21
Petunidin-3-glycoside-p-coumarate	0,00	0.27	0.29	0.31	0.32	0.35
Peonidine-3-glycoside-p-coumarate	0,00	0.16	0.14	0.13	0.13	0.15
Malvidin-3-glycoside-p-coumarate	0,22	1.08	0.39	0.77	1.16	1.26
The amount	43,86	57.22	64.82	76.04	86.02	91.35

**Table 4:** The amount of anthocyanins in samples of rose wine from the Hindogna variety (mg/dm<sup>3</sup>)

Connections	Control (without squeeze time)	Squeeze holding time, hour				
		3	6	12	18	24
Delphinidin-3-glycoside	0,39	1.36	3.25	3.96	4.06	4.12
Cyanidin-3-glycoside	1,40	1.49	1.55	1.76	1.88	1.86
Petunidin-3-glycoside	0,89	1.08	2.41	2.96	3.01	3.07
Peonidin-3-glycoside	8,30	10.25	10.35	11.05	11.55	11.62
Malvidin-3-glycoside	35,67	38.30	40.12	48.40	61.39	62.78
Delphinidin-3-glycosyl acetates	0,00	0.65	0.77	0.96	0.83	1.03
Cyanidin-3-glycosyl acetates	0,00	0.15	0.19	0.18	0.25	0.31
Petunidine-3-glycosyl acetates	0,00	0.84	0.91	0.97	1.17	1.36
Peonidin-3-glycosyl acetates	0,38	0.52	0.63	0.75	0.96	1.07
Malvidin-3-glycosyl acetates	1,25	2.95	2.36	3.99	4.55	5.81
Delphinidin-3-glycoside-p-coumarate	0,00	0.27	0.28	0.28	0.31	0.32
Petunidin-3-glycoside-p-coumarate	0,00	0.22	0.22	0.24	0.25	0.27
Peonidine-3-glycoside-p-coumarate	0,00	0.94	0.97	1.02	1.08	1.13
Malvidin-3-glycoside-p-coumarate	0,34	1.16	2.52	3.01	3.94	4.25
The amount	48,62	59.63	66.53	79.53	86.23	99,00

**Table 5:** Effect of maceration on the amount of volatile phenols in rose wine samples (µg/dm<sup>3</sup>) Madrasa variety

Volatile phenols	Control (without squeezing time)	Squeeze holding time, hour				
		3	6	12	18	24
2-methoxy-4-viny phenol	191,7	198,6	202,9	202,9	203,5	204,9
2,6-dimethoxy phenol	13,0	13,7	14,0	14,1	14,2	14,2
4-viny phenol	114,6	116,3	116,9	117,2	117,8	117,9
Vanillin	9,7	11,4	12,2	12,7	12,9	13,1
Acetovaniline	44,9	55,3	60,5	61,0	61,7	61,9
Zinjeron	8,6	15,3	19,2	20,0	20,5	21,2
Propiovdnillon	56,0	77,4	86,4	90,2	92,1	94,6
Homovaniline alcohol	31,4	34,2	43,5	43,8	44,0	44,2
The amount	469,9	522,2	554,7	561,9	572,7	572,0

**Table 6:** Effect of maceration on the amount of lactones in rose wine samples (µg/dm<sup>3</sup>) Madrasa variety

Lactones	Control (without squeezing time)	Squeeze holding time, hour				
		3	6	12	18	24
γ- butyrolactone	621	665	674	676	680	686
Pantolactone	9,4	11,3	14,2	14,9	15,3	15,7
γ- octalactone	6,5	7,9	9,3	10,1	10,6	11,2
8- octo -γ- hexolactone (soleron)	15,6	16,2	17,1	17,8	18,2	18,9
4-ethoxy carbonyl-gama-butanolactone	27,6	34,3	39,5	49,1	40,7	40,7
4-(hydroxyl-ethyl) Gama-butyrolactone	76,3	781	79,7	79,9	80,4	81,7
δ-decalactone	3,7	4,2	5,3	5,5	5,8	6,0
The amount	760,1	817,0	839,1	844,3	851,0	860,2

**Table 7:** Effect of maceration on the amount of carbonyl compounds in rose wine samples ( $\mu\text{g}/\text{dm}^3$ ) Madrasa variety

Carbonyl compounds	Control (without squeezing time)	Squeeze holding time, hour				
		3	6	12	18	24
3-hydroxy-2-butanone (acetoin)	61,8	64,1	68,2	70,3	71,1	71,8
4-hydroxy-4 methyl-2 pentanone	17,5	18,2	21,3	22,1	22,9	23,1
3-hydroxy-4-phenol-2-butanone	5,9	7,1	8,3	8,7	9,0	9,2
Benzophenone	12,7	13,0	13,5	13,6	13,6	13,7
3-okzo-alpha-ionol	13,9	17,2	19,4	19,8	20,0	20,2
The amount	111,8	119,6	130,1	134,5	136,6	138

### Conclusion

1. Samples of rose wines with and without maceration were made from local varieties of red grapes of Madrasa and Hindogna in aging for various periods of time (from 3 to 24 hours). The prepared samples were analyzed by year (2018-2020) and in accordance with the manufacturing technology.

2. While the Madrasa grape variety allows for more refined wine samples, the samples obtained from the Hindogna variety featured a higher alcohol and extract content. If the ratio of leucoanthocyanins to anthocyanins in the samples of wine made by year from the Madrasa variety, according to the average indicators for 3 years, ranged from 1.81-2.50 and the color intensity-from 0.33-0.47, then in the Hindogna varieties-from 1.87-2.16 and 0.40-0.52, respectively.

3. It turned out that the amount of the above extract was 19.8-21.7 g/dm<sup>3</sup> in the samples of Madrasa wine and 22.6-23.5 g/dm<sup>3</sup> in Hindogna, respectively. And if you look at the years, you can see that the higher amount of extract for both varieties falls in 2020.

4. In the squeeze, an increase in the amount of anthocyanins was observed during the maceration period from 3 to 24 hours. When considering the samples, the anthocyanin content in the sample obtained with 3-hour maceration was minimal, and with 6-and 12-hour maceration-optimal, and, finally, with 18 - and 24-hour maceration-maximum, that is, 86.02-91.35 mg/dm<sup>3</sup>. As for the quality, 14 representatives of anthocyanins were identified in the wine samples.

5.8 representatives of aromatic volatile phenols, 7 representatives of lactones and 5 hexacarbonyl compounds were found in wine samples. Organoleptic analysis of rose wine samples taken from the Madrasa grape variety with a squeeze extract of 3, 6, 12, 18 and 24 hours was performed. Samples of rose wine prepared by macerating the squeeze for 3 and 6 hours were rated 0.6-0.8 points higher than their counterparts.

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