Досліджено вплив технологій отримання сусла на кількісний вміст та якісний склад поверхнево-активних речовин у виноматеріалах із винограду сортів Сухолиманський білий і Піно Нуар. Розроблено регресійні моделі залежності хімічного складу поверхнево-активних речовин від схем відбору виноградного сусла. Встановлено, що пінисті властивості виноматеріалів визначаються компонентним складом біополімерних комплексів

Ключові слова: технологія сусла, виноматеріали для ігристих вин, поверхнево-активні речовини, пінисті властивості

Исследовано влияние технологий получения сусла на количественное содержание и качественный состав поверхностно-активных веществ в виноматериалах из винограда сортов Сухолиманский белый и Пино Нуар. Разработаны регрессионные модели зависимости химического состава поверхностно-активных веществ от схем отбора виноградного сусла. Установлено, что пенистые свойства виноматериалов определяются компонентным составом биополимерных комплексов

Ключевые слова: технология сусла, виноматериалы для игристых вин, поверхностно-активные вещества, пенистые свойства

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RESEARCH INTO TECHNOLOGIES OF OBTAINING GRAPE MUST IN THE PRODUCTION OF SPARKLING BASE WINES

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1. Introduction

The basic quality criteria of sparkling wines are their typical properties: sparkling and foamy. High indicators of aromatic and taste characteristics [1, 2] distinguish sparkling wines with improved sparkling and foaming properties.

Typical properties of sparkling wines mainly depend on the qualitative composition and quantitative content of surface active substances (SAS) in the wine medium [3–5]. In this case, stability of the wine-gas system is provided by SAS, which are in the kinetically stable forms.

Qualitative composition of SAS and their content in sparkling wines depend not only on the variety of grape and terroir but to a large degree on the technologies of wine and base wine production [6–9]. As a result of applying carefully targeted technological techniques, it becomes possible to regulate the composition of most SAS in base wines and sparkling wines [1, 10, 11].

A task of selecting rational technological operations in the production of base wine with the optimal content of SAS, high indicators of sparkling and foaming wine properties has for many years attracted specialists from the wine industry. There are known studies that examined the effect of grape maturity on the formation of low molecular SAS (alcohols, organic acids) in the base wine and on its capacity to form foam [1]. French and Spanish oenologists [6, 12] focus their scientific and practical activities on exploring foaming properties of base wine depending on the aggregately stabilized SAS of protein nature. For this purpose, at the stages of must clarification and stabilisation of base wine, oenological products are applied. This, as a result, contributes to prolonged formation of stable foam at the surface of wine medium [13, 14]. Researchers in Ukraine, Moldova and Russia work in the direction of examining dependence of the processes of selecting must, its clarification, the impact of applied technological equipment in the grape processing on the content of biopolymeric SAS and foaming properties in the obtained base wine.

Modern scientific research into the area of sparkling wine production, however, does not take into account how the applied technological operations may affect in general the SAS formation and manifestation of base wine typicality. In addition, proper attention is often not paid to innovative technologies in the production of sparkling base wines, which may greatly improve the qualitative and quantitative composition of SAS and, as a result, typical properties of the sparkling wine. In this regard, it is a relevant direction of research to examine a comprehensive impact of technological processes of obtaining grape must on the dynamics of the content and composition of SAS and typical properties of obtained base wines.

2. Literature review and problem statement

The formation of typical properties of high quality sparkling wines is achieved by a lengthy release of ball-like low dispersed carbon dioxide bubbles that occur in the thickness of the liquid. The CO_2 bubbles slowly ascend to the wine surface. As a result, stable and dense foam forms at the surface of the wine-gas interphase. In the foam, the bubbles are maximally close, deformed, separated by thin films, which prevent their merging (coalescence) to enlarged sizes [1, 2, 19].

Mechanical strength, elasticity and plasticity of the film of the bubbles, which are born in the wine medium, as well as those of foam bubbles, are due to its adsorption of the molecules of SAS [3].

Wine SAS are divided into two groups by the molecular mechanism of action. The first group of SAS is the true water-soluble compounds that do not form colloidal structures in the volume of liquid. SAS of the second group are the compounds, which belong to colloids or such that form a gel-like structure in the adsorption layer of the bubbles.

SAS of the first group are characterized by low-molecular weight and diphillic nature. They are mainly represented by alcohols, organic acids, amino acids and their amides, esters, aldehydes, melanoids [5]. Their stabilizing action is to form liquid and movable adsorption layers at the outer and inner surface of the bubble film. This, in turn, provides for the elongation of adsorption layers of such two-side films and, as a result, prevents liquid from leaking. However, the main role of such SAS is to decrease the rate of mass exchange processes (evaporation, condensation), to hinder processes of their CO_2 absorption and gas release, and the growth of gas bubbles in wine [6, 19]. Therefore, low molecular SAS positively influence sparkling properties of wine; they are, however, weak foam forming agents.

SAS of the second group include biopolymers: proteins and peptides, polysaccharides, polyphenolic compounds, protein-polyphenolic and protein-polysaccharide complexes [3]. Such compounds, when adsorbed at the interphase of contact liquid-gas, contribute to the reduction in surface tension and to the increase in the viscosity of adsorption layers of bubble film [6]. Even at weak adsorption of carbon dioxide, SAS form strong structured protective shells with hydrophilic outer surfaces, which are distinguished by high elastic-plastic and mechanical characteristics. Therefore, these are the natural macromolecules that contribute to the improvement in the foaming properties of wine: they reduce the rate of gas bubbles release, decrease their size, improve foam formation and stability of foam, and reduce speed of the syneresis process (leakage of liquid from the film).

Of the biopolymeric SAS of base wine and wine, the most thoroughly studied are protein substances. Proteins and peptides whose molecular weight is within 24–60 kDa even in low concentrations provide for the foam formation in base wine [10, 20, 21]: they form adsorption layers with high mechanical strength and, as a result, increase stability of the sparkling wine foam.

Unlike protein substances, polysaccharides do not affect capability of base wine to foaming, but they act as foam stabilizers [5]. Polysaccharide SAS are represented mainly by branched hetero- and homopolysaccharides (pectin compounds, arabinans, arabino-galactans, manans, glucans). They are characterized by the distribution of molecular weight in a wide range 7 to 200 kDa [22, 23]. The products of interaction between polysaccharides and proteins are also distinguished by high surface activity [24, 25].

Polymeric forms of phenolic compounds (tannins), according to authors of papers [22, 26], contribute to the stabilization of foam in base wine and improve indicators of sparkling properties in sparkling wines. However, Spanish scientists point to the inverse correlation regarding the content of proanthocyanidins in wine and its foam forming capacity [5]. The reason for this, obviously, is the interaction between tannins and protein substances. Protein-tannin products precipitate due to the growth in their molecular weight and loss of aggregate stability. In this case, scientists do not rule out the possibility of absorption of such compounds at the interphase of gas-liquid. As a result, mechanically strong film may form around the bubbles that would contribute to the formation of stable and dense foam.

In the process of base wine production, composition of the biopolymeric SAS changes, as well as their characteristics and properties (molecular weight, structure, solubility, reactivity) [1]. At the stage of obtaining the must (destemmed grapes, grape berry crushing, pressing marc, clarification of must) and its further fermentation, biopolymers undergo the largest changes. Protein substances are mainly concentrated in the central part of the berry. That is why they pass to a large degree to the Cuvee first press fraction. Enrichment of must with polysaccharides and phenolic compounds from the skins and seeds of grape berry occurs during intensive mechanical impacts on the raw material, in particular in the pressing process. The total amount of biopolymeric compounds is reduced at the stage of must clarification (treatment with ionites leads to a decrease in the mass fraction of protein, treatment with pectolitic enzyme preparations – in the mass fraction of polysaccharides) [7]. Hydrolytic processes of polysaccharide SAS of grape origin actively proceed also during alcohol fermentation of must. However, in the process of base wine aging on lees, there occurs the hydrolysis of outer layers of cell membranes of yeast and the partial transition of soluble fractions of yeast polysaccharides into wines [21]. During technological operations, as a result of oxidation of natural compounds, there appear preconditions to their polymerization and, as a result, the growth in molecular weight and loss of aggregate stability. This leads to irreversible colloid opacity in wines [23]. The transformation of SAS that occurs as a result of applied technological operations in the production of sparkling wines should provide for the aggregate-stable balance of natural macromolecules-colloids and quality indicators of typical properties of wines.

At present, technologies of sparkling base wines include different approaches to the selection of grape must. In Ukraine, there is a regulated separation of must in the amount not exceeding 65 dal/t [16]. In France, Italy and Spain they obtain Cuvee first press fraction must and subsequent press fractions must 1st Taille, which differ in type and quantitative content of biopolymeric SAS [27]. In order to stabilize SAS, grape must and is clarified and fermented to base wine. In Ukraine, the must is clarified, mainly, by the sedimentation method. In the European countries, various oenological preparations are used for this purpose. Base wines obtained from the Cuvee and 1st Taille must are combined for secondary fermentation provided they are positively evaluated by their organoleptic or physico-chemical parameters. However, at these variants of selecting the must,

the poor quality of the first 5 dal/t Cuvee is not taken into account. Lipids and varieties of wild microorganisms enter such must from the surface of grape berries.

Authors of paper [11] proposed appropriate mathematical models to predict the foaming properties of wine depending on its chemical composition. Scientists stress, however, that experimental data obtained by this method are rather controversial [1].

Thus, typical properties of base wine for the production of sparkling wines are predetermined, first of all, by SAS biopolymers. Information in the literature on the characteristics of natural macromolecules – colloids (free and in the composition of complexes), their role in the formation of sparkling and foaming properties of wine differs often. Commonly applied technologies of sparkling wines in the world wine producing countries are not aimed at obtaining base wine with the specified content and composition of SAS of colloidal nature. Therefore, it is important to examine a possibility of applying innovative approaches to fraction separation of must, its clarification by the method of using oenological preparations for the purpose of targeted regulation of aggregate-stable SAS, in particular of biopolymeric origin, and foaming properties of base wine.

3. The aim and tasks of the study

The aim of present work was to examine quantitative content and composition of SAS in base wines for the production of Ukrainian sparkling wines depending on the techniques of must fractionation and its clarification, as well as determining the foaming properties of base wine.

To achieve this aim, the following tasks were to be solved:

– to explore the effect of traditional and innovative fractionation techniques and must clarification on the dynamics of content of SAS of protein, carbohydrate and phenolic origin in base wine;

– to devise a mathematical model for predicting the chemical composition of SAS in base wine depending on the must fractionation method;

– to determine the quantitative content of biopolymers in base wine and to define their characteristics;

– to establish dependence of indicators of foaming properties in base wine on the technology of obtaining must and the composition of SAS in base wines.

4. Materials and methods for examining the effect of must fractionation and clarification techniques on the quantitative content and composition of SAS and foaming properties of sparkling base wines

4. 1. Examined materials and equipment used in the experiment

The objects of research are the sparkling base wines, vintage 2014. They were made of technical varieties of grape Sukholimansky white and Pinot Noir, cultivated under climatic conditions of Odessa Region. Ukraine. The grape was harvested at the accumulated concentration of sugars in the berry at 160–200 g/dm^3, titrated acids – 8.0–11.0 g/dm^3, pH index did not exceed 3.1.

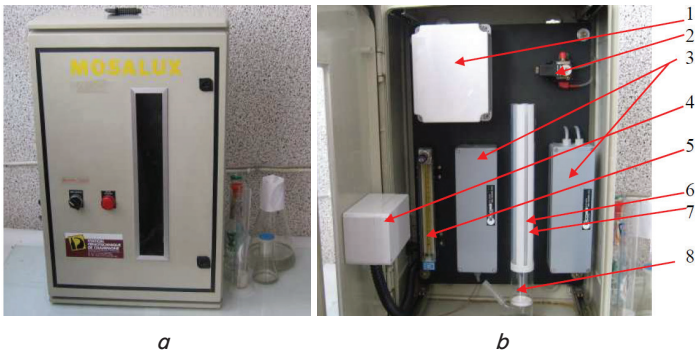
Grape must and base wines were obtained under the micro winemaking conditions at NNC "IViV named after V. Tairov" (Odessa Region, Ukraine). To press the grape and marc, we used the pneumatic press SF9 (made by Puleo, Italy). Separation from stems and crushing of berries were carried out at the destemmer-crusher Pratika 30 (made by Puleo, Italy). The must was clarified using commercially available oenological preparations of fining and stabilizing action (made by Martin Vialatte Oenologie, France): halotanin Tanigal (HT), enzyme preparation Dipectil Clarification (EP), bentonite Electra (B), complex preparation Polygreen (CP – a mixture of plant protein, bentonite and polyvinylpolypyrrolidone). Fermentation of the must was carried out in the presence of preparation of dry yeast DV-10 (made by Martin Vialatte Oenologie, France). The grape, marc and base wine were sulfated using the aqueous solution of potassium disulphate Baktol P (made by Martin Vialatte Oenologie, France).

To conduct gel chromatography of base wine, we used Sephadex S-100 (Pharmacia Company, Ukraine). The column for gel chromatography was calibrated using markers-dextrans with known molecular weights: dextran *Leuconostoc mesenteroides* 9000–11000 Da (No. 31416 Sigma, Germany); dextran *Leuconostoc mesenteroides* 15000 Da (No. 51227 Sigma Aldrich, Germany); dextran *Leuconostoc mesenteroides* 25000 Da (No. 31419 Fluka, Germany); dextran *Leuconostoc mesenteroides* 35000 Da (No. D1662 Sigma, Germany); dextran *Leuconostoc spp.* 70000 Da (No. 31390 Sigma Aldrich, Germany); dextran *Leuconostoc spp.* 100000 Da (No. 09184 Sigma Aldrich, Germany). In the eluates and starting base wines, mass fraction of protein substances, carbohydrates and phenolic compounds was determined at the photometer KFK-3 (Zagorsky Optical-Mechanical Plant, Russia), by the magnitude of absorbance.

Foaming properties of sparkling base wines were examined using the specialized Mosalux apparatus (made by Station Oenotechnique de Champagne, France) (Fig. 1).

Before determining foaming properties, the base wine was preliminary filtered using the device for vacuum filtering PVF-47/NB2 (ZAT Laboratory Equipment and Instruments, Russia).

Mathematical processing of the obtained experimental data was performed in the Matlab 14 programming environment.



a *b*

Fig. 1. Device Mosalux for determining foaming properties of base wines: *a* – general view; *b* – inside view (1 – interface box; 2 – electric valve; 3 – transmitter/receiver of IR-radiation; 4 – breaker; 5 – flow meter; 6 – foam; 7 – wine; 8 – cylinder

4. 2. Method for obtaining the sparkling base wines and determining their physical-chemical parameters (experimental procedures)

The must and base wine were obtained from the grape of Sukholimansky white and Pinot Noir varieties using two techniques by the "white" method.

According to the first traditional technique of white base wine production destined for the production of a sparkling wines closed tank method, the grape was processed in line with the requirements of TI U 00011050-15.93.11-2:2009, Ukraine. For this purpose, grape was processed at the destemmer-crusher. The aqueous solution of potassium disulfite calculated not to exceed 40 mg/dm^3 of the total amount of sulphite acid in the must was added to the resulting marc. The separation of must in the amount not larger than 65 dal/t was conducted by a pneumatic press of the closed type of periodic action. Next, the must was sent to clarification by the sedimentation method (process of self-clarification) at temperature 12–14 °C for 24 hours until thick sediment formed. Upon completing the must clarification process, its decantation was carried out from the thick sediments. Preparations of active dry yeast were introduced to the clarified grape must. The process of fermentation was carried out at temperature 16–18 °C until attaining such content of residual sugar that should not exceed 2.0 g/dm^3. Next, the resulting base wine was decanted from the yeast sediment and the solution of potassium disulfite was introduced calculated not to exceed 20 mg/dm^3.

According to the second, innovative, processing technique, the sulfated grape in whole clusters was pressed in the pneumatic press of the closed type of periodic action. At the same time, the must was fractioned: we selected the first 5 dal/t and the next 45 dal/t Cuvee. Slowly and gradually, pressure in the pressing chamber of the equipment was increased from 3×10^{-6} to 8×10^{-6} Pa, fractions of must were separated in the amount of 15 dal/t. Next, these factions were combined with the first fractions Cuvee – it is the 1 st Taille must by total volume of 20 dal/t. At the next stage, we carried out the clarification of two received fractions of must (Cuvee and 1 st Taille) with the use of auxiliary oenological preparations (method of fining) separately and in combination. The resulting concentrations of oenological preparations in the must are listed in Table 1. Control samples of the must were not introduced with the auxiliary preparations. That must was clarified by the sedimentation method. The solutions of oenological preparations were prepared according to the instructions on their application. The process of must clarification was carried out at temperature 12–14 °C for 12–24 hours until thick sediment formed. On completion the clarification process, the must was decanted from thick sediments, after which the technological process proceeded similar to the traditional technique of grape processing.

In the obtained samples of base wine we determined spectrophotometrically the total content of protein substances (PS) by the Bradford method absorbance at 605 nm [28], phenolic compounds (PC) by applying the Folin-Ciocalteu reagent absorbance at 760 nm [29], carbohydrates (C) – by the anthrone method absorbance at 670 nm [30]. Fractionation of these compounds by their molecular weights was carried out on the column with Sephadex S-100 (380×22 mm) [31]. The column for gel chromatography was calibrated using markers-dextrans with known molecular weights. Experimental samples of base wines in the amount of 5 cm^3 were placed in the column. Elution was achieved with distilled water. In the eluates of 4 cm^3 we quantitatively determined protein [28], phenolic [29] and carbohydrate component [30]. Obtained results were used to construct curves in the gel chromatography.

Table 1

Techniques of must clarification by fining and stabilizing oenological preparations

Technique of must clarification	Dosage of oenological preparations			
	HT, g/dm^3	EP, cm^3/dm^3	B, g/dm^3	CP, g/dm^3
Cuvee (45 dal/t)				
HT+EP+B	0,05	0,01	0,2	–
HT+EP	0,05	0,01	–	–
EP+B	–	0,01	0,2	–
B	–	–	0,2	–
EP+CP	–	0,01	–	0,4
HT+ EP+CP	0,05	0,01	–	0,4
1 st Taille (20 dal/t)				
HT+EP+B	0,05	0,02	0,4	–
HT+EP	0,05	0,02	–	–
EP+B	–	0,02	0,4	–
B	–	–	0,4	–
CP	–	–	–	0,8
EP+CP	–	0,02	–	0,8
EP+CP+B	–	0,02	0,4	0,8
HT+EP+CP	0,05	0,02	–	0,8

Mathematical processing of experimental data regarding chemical composition of base wine depending on technique for the must fractionation was implemented in the Matlab 14 programming environment. The root mean square error (RMSE) served as a key informative index.

Determining foaming properties in the base wines by the device Mosalux is based on the interruption of infrared beams of IR-rays by the foam of the examined base wines, which is created as a result of injection of gaseous carbon dioxide under standard conditions through a filter with calibrated pores. When studying the foaming properties, base wine should be transparent. That is why their preparation for the study was conducted by preliminary filtering. After warming up the device Mosalux, clarified base wines in the amount of 100 cm^3 was placed into the working cylinder, then, under pressure 100 kPa, carbon dioxide was supplied calculated as 7 dm^3/h. As a result of conducted research, we determined the indicators of foaming properties of base wines: foam maximum height (MH) and foam stability time (TS). Calculation of appropriate indicators was carried out by using the Mosalux device's software [32].

5. Results of examining the effect of must fractionation and clarification techniques on the quantitative content and composition of SAS and foaming properties of sparkling base wine

It is known that over the past decades the expansion and improvement of the domestic raw material base has been achived in Ukraine by increasing the acreage of grape planting with high-quality varieties of the champagne type – Pinot Noir and Chardonnay. The former grape variety is traditionally used for the production of rose and red wines,

the latter – white wines. However, given the basic physical and chemical characteristics provided by the viticulturists and winemakers from the southern regions of Ukraine, sparkling base wines made from grape of Sukholimansky white variety is practically on par with the reference samples of classical Chardonnay variety [9]. In addition, the grape of Sukholimansky white variety is the property of domestic viticulture – it was cultivated at NNC "IViV named after V. Tairov" by a method of crossing Chardonnay and Plavay varieties.

Technology for obtaining the must from grape of Sukholimansky white variety and Pinot Noir variety by the "white" method, in particular, must fractionation and its clarification, was implemented under conditions of micro winemaking at the NNC "IViV named after V. Tairov" by two techniques (chapter 4.2).

Experimental samples of base wine from the grape of Sukholimansky white variety and Pinot Noir variety, received by innovative proposed technique, were preliminarty examined by the methods of sensory analysis [33]. As a result of research, we established the most efficient techniques for clarification the must to form a balance of floral-fruit flavour of base wine. For the fining of Cuvee, it is expedient to use the following combinations of oenological preparations: HT+EP+B, EP+B, HT+EP+CP. 1 st Taille should be clarified by such combinations of preparations as: EP+CP, HT+EP+CP.

At the next stage of research, we determined quantitative content of protein substances, phenolic compounds, carbohydrates (chemical composition of SAS) in the base wine, obtained depending on the techniques of must fractionation, and defined effective ways of mash clarification by oenological preparations. Results of experimental studies are represented in Tables 2, 3.

Table 2

Chemical composition of SAS in the base wine from grape of the Sukholimansky white variety depending on the must fractionation and clarification techniques

Must clarification technique	Mass concentration, mg/dm^3		
	Protein substances	Phenolic compounds	Carbohydrates
Cuvee (45 dal/t)			
Self-clarification	68,5	173,0	1875,0
HT+EP+B	34,6	170,9	1700,0
EP+B	22,5	144,2	1500,0
HT+EP+CP	19,7	115,4	1300,0
1 st Taille (20 dal/t)			
Self-clarification	20,1	192,3	2550,0
EP +CP	19,4	156,5	2360,0
HT+EP+CP	18,4	144,2	2250,0
Must obtained by traditional technology (65 dal/t)			
Self-clarification	60,3	180,1	2450,0

Table 3

Chemical composition of SAS in the base wine from grape of the Pinot Noir variety depending on the must fractionation and clarification techniques

Must clarification technique	Mass concentration, mg/dm^3		
	Protein substances	Phenolic compounds	Carbohydrates
Cuvee (45 dal/t)			
Self-clarification	35,8	210,2	2310,0
HT+EP+B	22,1	200,5	2020,0
EP+B	15,8	174,8	1950,0
HT+EP+CP	10,2	155,2	1650,0
1 st Taille (20 dal/t)			
Self-clarification	21,8	266,6	2850,0
EP +CP	20,5	248,9	2750,0
HT+EP+CP	17,0	214,4	2240,0
Must obtained by traditional technology (65 dal/t)			
Self-clarification	29,8	234,2	2750,0

Data in Tables 2, 3 on the chemical composition of base wine from the grape of both varieties, taking into account techniques only for separating the grape must and its self-clarification, indicate that the dominant component is the carbohydrates (1875.0–2850.0 mg/dm^3); protein substances are contained in the lowest amount (27–129 times lower than carbohydrates) while an intermediate position is taken by phenolic compounds. Varietal base wine received from the by the innovative technique are distinguished by the highest content of protein substances: in the base wine from Sukholimansky white grape variety, their mass fraction exceeds that of the base wine of Pinot Noir grape variety by 47.7 %. The concentration of phenolic compounds and carbohydrates increases in the process of pressing the grape in whole clusters, meaning that their content is the largest in the base wine obtained from the 1 st Taille. The base wines prepared in accordance with the traditional technology, by the content of protein substances, are slightly close to those received from the Cuvee, whereas by carbohydrates – to those received from the 1 st Taille. By the total content of protein, carbohydrate, phenolic SAS, the indicated base wine are similar to those obtained from the 1 st Taille.

In the process of must clarification by the sedimentation method and fining with the defined combinations of oenological preparations, the desired lowest values of total mass fraction of protein, phenolic, carbohydrate components in SAS were demonstrated by the samples of base wine whose must were clarified using HT+EP+CP. For the base wine from the Sukholimansky white grape variety (Table 2), made from the Cuvee and 1 st Taille, a decrease in this indicator amounted to 19.0 and 32.2 %, respectively, while for Pinot Noir is, accordingly, 29.0 and 21.3 % (Table 3).

Results of mathematical processing of experimental data regarding the dependence of chemical composition of SAS of varietal base wines, made from different fractions of must by the traditional and innovative techniques of grape processing, are shown in Fig. 2.

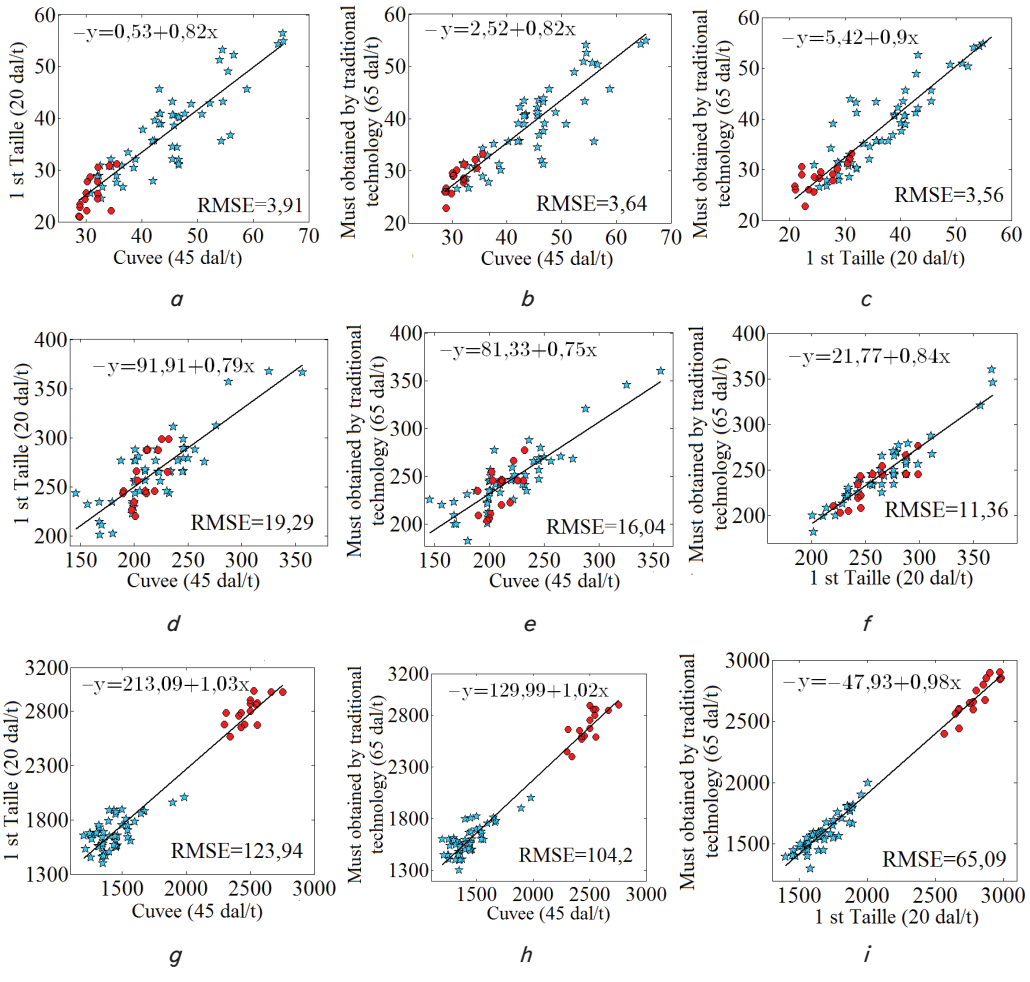


Fig. 2. Dependence of chemical composition of SAS in the base wine on the must fractionation techniques: ☆ – grape of Sukholimansky white variety; ● – grape of Pinot Noir variety; *a–c* – protein substances; *d–f* – phenolic compounds; *g–i* – carbohydrates

Based on the data of statistical processing of experimental studies on establishing the relationship between SAS composition (protein, phenolic, carbohydrate components) and techniques for the must fractionation, we obtained regression equations. We also calculated indicators of the root mean square error (RMSE) for all possible variants of comparing techniques of the must fractionation by the domestic traditional and innovative methods of base wines production. The lower the RMSE indicator, the stronger the dependence of chemical composition of the examined base wine on the effect of grape processing technique at the stage of must fractionation is. Minimal magnitudes of the RMSE indicator are characteristic of protein components in the base wine, maximal – of carbohydrates.

Fractional composition of protein, carbohydrate, phenolic SAS in the base wines of both grape varieties by their molecular weights was examined by the gel chromatography method. Fig. 3 shows results of gel chromatography on the example of base wine, received from the Cuvee–from the grape of Sukholimansky white variety and clarified by the sedimentation method.

According to data presented in Fig. 3, the sample of base wine contains low- and highmolecular SAS, among which the former prevail. At the initial curves of gel chromatography in the zone of high molecular mass (50–60 kDa) there is a coincidence in the peaks of carbohydrate, protein and phenolic components. This may point to the existence of the mentioned compounds in the examined base wine in the form of the complex: protein substances – phenolic compounds – polysaccharides (biopolymeric SAS). Similar results were obtained when examining all other experimental samples of base wine from both grape varieties.

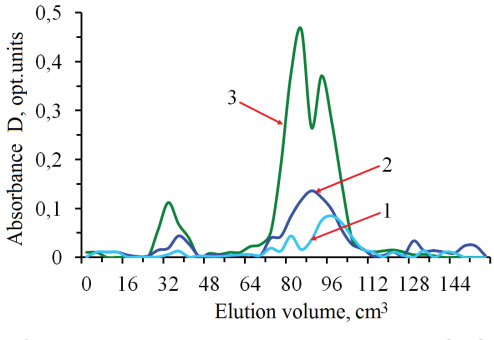


Fig. 3. Initial curve of gel chromatography for SAS of base wine from the grape of Sukholimansky white variety by Sephadex S-100: 1 – protein substances; 2 – phenolic compounds; 3 – carbohydrates

Tables 4, 5 show component composition of biopolymeric complexes and their quantitative content, based on the results of gel chromatography of varietal base wine.

Table 4

Characteristics of biopolymeric complexes of the base wine from grape of the Sukholimansky white variety for the production of sparkling wines

Base wine sample	Complex of biopolymers in base wine								
	total content (from ΣPS+PC+C in base wine)		component composition						Mass ratio PS:PC:PoS
			PS		PC		PoS		
	mg/dm³	%	mg/dm³	%	mg/dm³	%	mg/dm³	%	
Cuvee (45 dal/t)									
Must self- clarification	51,0	2,4	11,0	21,6	8,0	15,7	32,0	62,7	1:0,7:2,9
Must +HT+EP+B	38,7	2,0	8,3	21,5	6,4	16,5	24,0	62,0	1:0,8:2,9
Must +EP+B	32,0	1,9	6,4	20,0	4,8	15,0	20,8	65,0	1:0,8:3,3
Must +HT+EP+CP	43,2	3,0	16,8	38,9	1,6	3,7	24,8	57,4	1:0,1:1,5
1 st Taille (20 dal/t)									
Must self- clarification	68,2	2,5	7,4	10,9	4,8	7,0	56,0	82,1	1:0,6:7,6
Must +EP+CP	62,0	2,4	10,4	16,8	3,0	4,8	48,6	78,4	1:0,3:4,7
Must +HT+EP+CP	65,6	2,7	14,4	22,0	3,2	4,9	48,0	73,2	1:0,2:3,3
Must obtained by traditional technology (65 dal/t)									
Must self- clarification	62,3	2,5	8,9	13,4	6,6	10,0	46,8	76,6	1:0,7:5,7

Note: PS – protein substances, PC – phenolic compounds, PoS – polysaccharides, C – carbohydrates

Table 5

Characteristics of biopolymeric complexes of base wine from the grape of Pinot Noir variety for the production of sparkling wines

Base wine sample	Complex of biopolymers in base wine								
	total content (from ΣPS+PC+C in base wine)		component composition						Mass ratio PS:PC:PoS
			PS		PC		PoS		
	mg/dm³	%	mg/dm³	%	mg/dm³	%	mg/dm³	%	
Cuvee (45 dal/t)									
Must self- clarification	123,5	4,8	6,4	5,2	8,6	7,0	108,5	87,9	1:1,3:16,9
Must +HT+EP+B	95,7	4,3	4,8	5,0	8,0	8,4	82,9	86,6	1:1,7:17,3
Must +EP+B	95,2	4,3	3,9	4,1	7,2	7,6	84,1	88,3	1:1,8:21,6
Must +HT+EP+CP	104,1	5,7	9,2	8,8	5,3	5,1	89,6	86,1	1:0,8:9,7
1 st Taille (20 dal/t)									
Must self- clarification	196,2	6,3	3,5	1,8	12,4	6,3	180,3	91,9	1:3,5:51,5
Must +EP+CP	157,1	5,2	4,4	2,7	9,1	4,3	143,6	91,4	1:2,1:32,6
Must +HT+EP+CP	164,5	6,7	4,8	2,9	8,8	5,3	150,9	91,7	1:1,8:31,4
Must obtained by traditional technology (65 dal/t)									
Must self- clarification	181,3	6,0	6,9	3,8	8,8	4,9	165,6	91,3	1:1,3:24,0

Note: PS – protein substances, PC – phenolic compounds, PoS – polysaccharides, C – carbohydrates

Results of the experiments conducted indicate that in the base wine from the grape of Sukholimansky white variety the mass fraction of high molecular complexes (biopolymeric SAS) does not exceed 3.0 %, in the base wine from the grape of Pinot Noir variety – reaches 6.7 %. In this case, in the base wine from white grape, clarified by the sedimentation method, quantitative content of high molecular complexes is almost the same. However, these complexes differ by the share of polysaccharide component. In the base wine from red grape, the concentration of complex increases by 4.8 % to 6.3 % as a result of mechanical impacts. An introduction into the must clarification procedure of oenological agents in combination HT+EP+CP contributes to an increase in the mass share of biopolymeric SAS in base wine (by 6.4–25.0 %) due to the increased content of protein component (by 1.6–2.0 times). In this case, the base wine whose musts were clarified by other fining techniques are characterized by a reduced indicator in the mass share of complexes.

Indicators of foaming properties in base wine (maximal foam height, foam stability time) obtained by the traditional and innovative technological techniques of grape processing are shown in Fig. 4.

It was discovered that the foaming properties of base wine depend on the variety of grape and technology of obtaining the must. Among the examined in present work techniques of must separation, the highest values of foaming indicator characterize the base wine, made of the Cuvee (MH of base wine from the grape of Sukholimansky white variety – 113 mm, Pinot Noir – 98 mm). In contrast to the base wine of white grape, the highest foam stability indicators are inherent to the base wine made from red grape: the most stable one turned out to be the foam of base wine from the Cuvee (foam stabilization time TS is 269 s). Application of oenological preparations for the must clarification compared with the deposition method leads to decreasing the MH indicators of wine. In this case, the magnitudes of foam

stabilization TS in the examined samples of base wine within a particular variety of grape change irregularly relative to the magnitudes of HM indicator. This is probably due to the presence in the composition of base wine of SAS, different by mass share and component composition.

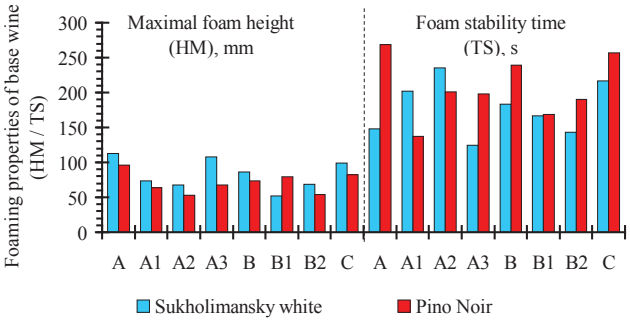


Fig. 4. Foaming properties of base wine depending on the must fractionation and clarification techniques: A, A1, A2, A3 — Cuvee-(45 dal/t); B, B1, B2 — 1 st Taille (20 dal/t); C — must obtained by traditional technology (65 dal/t); A, B, C — must self- clarification; A1 — clarification of must by preparations PT+EP+B; A2 — clarification of must by preparations EP+B; A3, B2 — clarification of must by preparations PT+EP+CP; B1 — clarification of must by preparations EP+CP

6. Discussion of results of the effect of must fractionation and clarification techniques on the quantitative content and composition of SAS and foaming properties of sparkling base wines

The first stage of the research was to study chemical composition of SAS in the base wine from the grape of Sukholimansky white and Pino Noir varieties (Tables 2, 3), received in accordance with the generally accepted technology of base wine in Ukraine. It was established that due to a prolonged contact between the must and solid elements of berries at the stages of crushing, pumping with marc pressing, as well as an active course of processes of oxidation, there occurs an accumulation of extractive substances of carbohydrate and phenolic nature at these stages as opposed to protein compounds. Base wine from red grape, compared to the one from white grape, are characterized by an enhanced content of phenolic compounds due to anthocyanin pigments [1]. High content of carbohydrate and phenolic components in base wine is consequently the cause of their colloidal instability. Due to the occurring processes of oxidation of these biocompounds and their additional polymerization, formation of complexes with other substances, they become prone to the aggregation and, consequently, are derived from base wines.

According to the innovative technique of grape processing (grape pressing is done in whole clusters), in the base wine, produced from the, in comparison with those obtained by the traditional technology, one observes an increase in the mass share of protein substances by 13.6–20.1 %, reduction in the concentration of phenolic compounds by 5.6–10.3 %, carbohydrates – by 16.0–23.5 % (Tables 2, 3). In the base wine, produced from the fractions of 1 st Taille, there is an inverse pattern regarding the formation of SAS composition. This is obviously due to the saturation of must with skins components and seeds of grape berries, and the stems of bunch of grapes as a result of increasing pressure in the device's pressing chamber. This aspect demonstrates production of base wines, similar by the content of phenolic compounds and carbohydrates to the base wine obtained by the traditional technology.

To evaluate SAS chemical composition in the base wine, we devised mathematical regression models that take into account the effect of grape must separation techniques (Fig. 2). Small magnitudes of the root mean square error (RMSE) indicate the quality of description of experimental data by mathematical models. The lowest magnitude of error was observed in terms of dependence of protein substances content in base wine on the existing variants of obtaining the must, the largest – regarding carbohydrate content at the same techniques of must separation. It becomes apparent from the derived regression equations that by the SAS content of protein, carbohydrate, phenolic nature, the closest are the samples of base wine, obtained from the 1 st Taille must and by the traditional domestic technology of grape processing.

Given the aforementioned, in order to obtain high quality base wines with the desired content of SAS, it is necessary in the technology of must to minimize contact between the grape berry juice and its solid parts, as well as contact between must and oxygen. In this connection, grape processing technology by the "white" method is compulsory, which takes into account pressing the raw material in whole clusters and the fractionation of must.

An important technological operation in the production of high quality sparkling base wines is the clarification of must by the fining method. This method in the production of wine is the most widely used due to rapid clarification of must, maximal removal of oxidative enzymes and insoluble products of polymerization of biocompounds, which negatively affect the foaming properties. It was established that the implementation of all techniques for must clarification by fining provides for a reduction in the total mass fraction of protein substances, phenolic compounds and carbohydrates. The most effective in terms of binding such biocompounds are the oenological preparations in combination HT+EP+CP.

At the next stage of present work, as a result of gel chromatographic studies, we established a presence in the base wine of SAS with low molecular weight and high molecular SAS in the form of complex: protein substances – phenolic compounds – polysaccharides. The mean molecular weight of the complexes reaches 50–60 kDa (Fig. 3). According to data represented in Tables 4, 5, mass fraction of biopolymeric SAS in base wine depends on the variety of grape and technological procedure of obtaining must. In the base wine from grape of Sukholimansky white variety, the composition of biopolymeric complexes is dominated by the protein-carbohydrate component, in the base wine from grape of the Pinot Noir variety – phenolic-carbohydrate component. In addition, in the base wine from grape of the Sukholimansky white variety from the 1 st Taille, compared with those of the Cuvee, we observed an increase in the mass concentration of polysaccharide share in the complex, and reduced amount of protein and phenolic compounds. In the respective base wine from grape of Pinot Noir variety, we observed increased indicators of the mass fraction of polysaccharides and phenolic compounds in contrast to protein substances.

Results of the study on the effect of combinations of oenological preparations on the dynamics of biopolymeric SAS in base wines (Tables 4, 5), obtained by the innovative

technology, demonstrate an increase in the mass share of complexes in the base wine whose must was clarified by the technique HT+EP+CP. Compared with a control sample (without oenological preparations), we observed an increase in the share of protein substances, reduction in the content of phenolic compounds and polysaccharide component in the biopolymeric complexes. It is probably caused by the penetration of molecules of CP proteins into the composition of complexes. In this case, EP hydrolyzes polysaccharides to the compounds with a much lower molecular weight, thus reducing somewhat their content in the complex. In contrast to the aforementioned technique of clarification, other variations of must processing with oenological preparations lead to a decrease in the mass share of biopolymeric complexes in all samples of base wines.

Important indicators in the technology of sparkling wines are the foaming properties of base wine. Using the Mosalux device, we determined indicators of foam maximum height (MH) and foam stability time (TS) (Fig. 4). It was discovered that base wine from grape of the Sukholimansky white variety, obtained by the traditional technology and from the fractionated self-clarified must, was characterised by somewhat larger MH indicators compared with those from grape of the Pinot Noir variety. Among the examined samples, the base wine, produced from the, demonstrated the highest indicators of foaming. Obviously, a large mass fraction of protein substances in base wine predetermines their high foaming ability and vice versa. Enhanced content of carbohydrates and phenolic compounds in the wine contributes to the stability of foam TS. However, there is no any relationship between the foaming properties of base wine from the Cuvee and the 1 st Taille, which were lightened with the use of oenological preparations, and the concentration of SAS of protein, carbohydrate and phenolic origin in them. No close connection was established between the foaming properties and the general content of biopolymeric complexes in the examined samples of base wines. It was shown, however, that stability of the disperse system wine-CO_2 is due mainly to the component composition of such SAS-biopolymers. Certain components of the biopolymeric complexes, which are characterized by the largest stabilizing action, improve corresponding indicators of the foaming properties of base wines for the production of sparkling wines. A protein component of biopolymeric SAS of all the base wines that were lightened by the sedimentation method, directly proportionally contributes to the foaming ability. As we know from data in Tables 4, 5, the mass fraction of protein substances in the composition of complexes of base wine from grape of the Sukholimansky white variety is 3.5–6.1 times larger than in the composition of base wine complexes from grape of the Pinot Noir variety. That is why the foam height MH of the first line of the examined samples is higher than that of the second line of the corresponding samples. The magnitudes of foam stabilization time indicator TS of the base wine from red grape are larger than those for the base wine, obtained from white grape. This demonstrates a dependence of the given indicator of foaming properties on the content of polysaccharides in the composition of biopolymeric complexes. Phenolic component in the base wine complexes correlates insignificantly by its content with the magnitudes of MH. When treating the fractionated base wine with combinations of oenological preparations, the mass fraction of complexes of biopolymeric SAS in the examined base wines is reduced within the variety, except when applying the variant of must clarification HT+EP+CP. In these samples, we observe a similar tendency in the magnitudes of indicator of mass fraction of protein substances and maximal foam height of the base wine. However, in spite of the fact that treating the must with HT+EP+CP contributes to the increase in protein substances in the complexes (foam forming factor) compared with the control samples (self-clarification of must) and does not provide for a significant growth in the magnitudes of HM indicator. It is established that as a result of must fining, foam stability in the base wine of both varieties changes directly proportionally depending on the share of polysaccharides in the composition of biopolymeric complexes of base wines. Phenolic component in the complexes of base wine obtained from the 1 st Taille, compared with those of the Cuvee, contributes to improving the indicators of foam stability time TS in base wine.

Results for the indicators of foaming in the examined samples of base wines, received using the Mosalux device, in most cases allow us to establish relationships between these indicators and chemical composition of SAS and the technologies for obtaing must. However, in some cases, the data received are contradictory and do not yield objective information. This is probably explained by the fact that the methodology of studying sparkling base wines by the Mosalux device requires preliminary filtering of wine. Under these conditions, a part of SAS, in particular biopolymers are adsorbed at the fмltering element of equipment [1]. In addition, at the inner surface of the cylinder of Mosalux device there is a possibility of foam buildup, formed as a result of injecting carbon dioxide through the calibrated pores of the filter [34].

It should be noted that the results on the foaming properties of base wine, determined using the Mosalux device in the presence of exogenous carbon dioxide, are preliminary and are used only for the prediction of these properties in the wines. Therefore, it is expedient to determine sparkling (a key indicator of typical properties of sparkling wines, which defines the foaming properties) and foaming properties of the finished products from the appropriate base wine under real conditions (in a glass), given the presence of endogenous CO_2 in them, formed during secondary fermentation of base wines. In addition, it was not established how the content and composition of SAS would change, as well as the typical properties of wines, obtained as a result of combining (egalisation) base wine from the and the 1 st Taille.

In future, the results of research obtained will lay the foundation for developing an algorithm for regulating the content and composition of SAS of base wines, typical properties in the technologies of high quality sparkling wines.

7. Conclusions

1. Base wines from the grape varieties of Sukholimansky white and Pinot Noir, independent on the technology of obtaining must are characterized by a high content of carbohydrates (1300.0–2850.0 mg/dm^3), phenolic compounds are present in less amount (115.4–266.6 mg/dm^3), and the smallest mass fraction is that of protein substances (10.2–68.5 mg/dm^3). Base wine from white grape, in comparison with that from red grape, is characterized by a larger concentration of protein substances, with less content of phenolic compounds and carbohydrates. Base wines, made according to the traditional technology, by the content

of protein substances are close to those from the Cuvee, whereas by carbohydrates – to those from the 1 st Taille. As a result of clarification the fractionated must with oenological preparations, there occurs a decrease in the content of protein, carbohydrate and phenolic SAS.

2. As a result of mathematical processing of experimental data, we developed regression models for the dependence of chemical composition of SAS on the techniques of grape must separation. We calculated optimality criteria – indicators of the root mean square error RMSE. By the smallest magnitudes of RMSE, a close relationship was established between the content of protein substances in base wine and methods for obtaining the must, by the largest magnitudes of RMSE – weak dependence by the mass fraction of carbohydrates.

3. Results of gel chromatography revealed that the base wine contain low– and highmolecular SAS. The latter are in the examined samples in the form of the biopolymeric complexes: protein substances – phenolic compounds – polysaccharides with molecular weight 50–60 kDa. In the base wine from grape of the Sukholimansky white variety, mass fraction of highmolecular complexes (dominated by the protein polysaccharide component) does not exceed 3.0 %, in the base wine from grape of the Pinot Noir variety – reaches 6.7 % (dominated by phenolic polysaccharide component). Variations in the separation of must and its clarification affect the transformation of biopolymeric SAS in base wine by their mass fraction and component composition. An aggregate stabilization of biopolymeric complexes in the base wine is provided by the processes of must fractionation and its clarification using a combination of commercially available oenological agents halotanin in combination with enzyme and complex preparations.

4. Foaming properties of base wine, which were determined using the Mosalux device, depend on the grape variety and technology for obtaining the must. The magnitudes of indicators of maximum height and time of foam stabilization are not affected by the overall content of SAS. They depend mainly on the component composition of biopolymeric complexes. We established the following trend: the protein component of the biopolymeric complexes defines mainly the ability to foaming; carbohydrate and, partially, phenolic components in most cases contribute to the stabilization of foam.

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