The object of the study is the intensification of the wine clarification process. Although a number of studies have been conducted on the clarification of wines, the effect of enzymes, ultrasound, membrane filter, ultrasound and maceration factor on the clarification intensity has not been sufficiently studied.

It has been found that the β-glucanase enzyme, compared to other enzymes, creates a basis for a greater decrease in the amount of polysaccharides, especially glucomannan, in pink wines and that the sample remains stable for 10 months. For white table wines, treatment with pectofoetidin П 10x and Г 10x enzymes and subsequent pasting, cold processing and filtration gave better results. It has been established that ultrasonic processing of wine materials increases the aromatic substances in the composition, color, acidity, and improves the stability of wines. The productivity of membrane filters increases, the filtration and clarification processes of wine materials are intensified. Ultrasonic frequency, acoustic oscillations applied to the liquid increase the permeability of the filters and allow them to operate in a self-cleaning mode. Cold maceration of Madrasa and Merlot grape varieties at 7–8°C for 4 days before fermentation allows for the production of juice with high color density, rich in total anthocyanins and phenolic compounds.

The production of stable wines using enzymes and cold maceration, as well as the intensification of clarification using ultrasound and membrane filter ultrasound, solves the problem and makes these studies important for production. The results obtained can be used in factories and wineries

Keywords: enzyme preparations, clarification, wine material, ultrasound, membrane filtration, cold maceration, monomeric anthocyanins, phenolic compounds

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IDENTIFYING THE FACTORS ON THE INTENSIFICATION AFFECTING OF THE WINE CLARIFICATION PROCESS

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1. Introduction

In modern times, among the processes involved in the production and processing of wine and wine materials, special attention is required for intensifying the clarification process, improving stability, and enhancing the taste quality of the final product. One of the most time-consuming key operations in winemaking is the sedimentation of complex suspended substances present in the wine. The most widely used clarification method is to allow the wine to settle for 1–2 weeks.

To accelerate clarification, various chemical adsorbents (such as montmorillonite, palygorskite, etc.) and different flocculants (such as PAA, KF-4, polyethylene oxide, etc.) are used. The simplest method is to let the wine settle naturally so that the suspended particles precipitate on their own. However, this method is characterized by its long duration and periodic nature. For this reason, the reserves for intensifying the process have been studied.

Numerous studies have examined the role of the wine's colloidal system in turbidity and the influence of proteins, phenolic substances, and metals on these processes. As a result, the winemaking industry now possesses a number of technological methods for stabilizing wines.

It should be noted that, to date, in the study of wine polymers related to stabilization problems, the main attention has been given to proteins. As for neutral polysaccharides, their role in the formation of colloidal hazes has not been sufficiently studied. Nevertheless, the results of our long-term research on certain grape varieties have shown that the polymer fractions contain proteins, high-molecular-weight

polyphenols, and polysaccharides. Proteins make up 11–35% of these fractions. Among the total amount of high-molecular-weight substances, polysaccharides (such as arabinogalactan, arabinoglucan, and glucomannan) account for the largest share.

Polysaccharides based on galacturonic acid (pectin substances) and those rich in pectin are very labile and tend to degrade under the influence of juice and yeast enzyme systems. As a result, the amount of polysaccharides based on hexoses increases in wines. The study of sediment in cloudy wines after bottling shows that 15–20% of its composition consists of neutral polysaccharides with low molecular weight, such as glucan and galactoglucomannan. The most probable type of linkage in the main chain of these polymers is through C_1 and C_2 atoms.

In such wine materials, the content of high-molecular-weight substances is significantly higher than in wines made from self-filtered fractions. For example, in wines made from the press fractions of Riesling, Bayan Shira, and Rkatsiteli grape varieties, the amount of polymers was 27–47% higher than in wines made from self-filtered juice. Developing effective methods to reduce the amount of these polymers in wines to a minimum would help solve this problem, which is particularly relevant for hard-to-clarify wine materials obtained from the press fractions of grape juice.

2. Literature review and problem statement

This study evaluated the influence of two pre-fermentation clarification methods (static settling and flotation) on the concentration of volatile compounds in Albariño and Treixadura wines. Tasters generally gave higher scores to wines obtained by the flotation method, which, combined with a shorter processing time, makes this method suitable for clarifying the most of these two white grape varieties [1]. As can be seen, this study focused on wine clarification using static settling and flotation. However, the hydrolysis of polymeric compounds, particularly polysaccharides, was not considered. The referenced article discusses the complex interactions among wine components-such as proteins, polysaccharides, and phenolic substances-as well as the physicochemical aspects and current knowledge in the field of wine clarification. In addition, it highlights potential directions for fundamental research on the mechanisms and interactions leading to successful clarification, which are essential for improving the process and can enhance both process efficiency and wine quality [2]. These studies examined the complex interactions between wine components, including proteins, polysaccharides, and phenolic compounds, and modern approaches to wine clarification. However, they did not include studies on clarifications performed with ultrasonic membrane filters. The use of membrane technologies in winemaking has revolutionized various stages of production by offering a precise and efficient alternative to traditional methods. The article presents a comprehensive analysis of advances and applications of membrane technologies in the wine industry [3]. This study provides a comprehensive analysis of the results obtained in the application of membrane technologies in winemaking. However, the essence of ultrasonic membrane filters and the acceleration of clarification with them is not touched upon. Another review summarizes and analyzes the origin of tartaric acid in wine, factors affecting its stability, detection methods, stabilization techniques, and the influence of these methods on the organoleptic properties of wine [4]. This study focused on the origin of tartaric acid, factors affecting its stability, stabilization methods, and the effect of these methods on the organoleptic quality of wine. However, this study did not consider the effect of tartaric acid and other compounds on the intensity of clarification. By comparing the composition of permeate and retentate obtained from pilot-scale fractionation of white and red wines using membranes of 75, 20, or 10 kDa and different permeability levels (50%, 80%, 90%, or 95%), the study aimed to assess ultrafiltration as an innovative approach to wine clarification and stabilization. The results revealed additional potential applications of ultrafiltration technology in winemaking, which can improve wine quality, process efficiency, and profitability [5]. This study investigated ultrafiltration as an innovative method for wine clarification and stabilization. It was noted that this method increases wine quality and process efficiency. However, these studies did not include the preparation of wine prior to ultrafiltration, which is a labor- and energy-intensive activity. In another work, the strengths and weaknesses of different stabilization approaches were systematically presented for the first time, providing valuable insights for optimizing winemaking methods [6]. This study presents the strengths and weaknesses of various approaches to wine stabilization and outlines the conditions for optimizing winemaking methods. The acceleration effect of enzyme preparations, ultrasound, and cold maceration on clarification has not been explained. An experimental study included the treatment of grape juice with the enzyme Zymo claire Pro Ice during maceration and extraction stages, as well as with Zymo claire CC Plus during clarification. The results showed high efficiency of the enzymes tested in these experiments for clarifying grape juice [7]. The enzyme preparations used in this study were studied during maceration, extraction, and also clarification. However, issues related to the hydrolysis of polymeric compounds, especially polysaccharides, were not addressed. Another study investigated the effects of clarification with egg albumin, progressive clarification, and crossflow microfiltration on the polysaccharide and proanthocyanidin composition of four red varietal wines. Polysaccharides rich in arabinose and mannoproteins were better retained by ceramic membranes than those rich in galactose and proanthocyanidins. Discriminant analysis clearly differentiated the wines subjected to crossflow microfiltration from the others [8]. In this study, the effects of egg albumin on fining, progressive fining, and microfiltration were studied on four red grape varieties. However, the effects of cold maceration on wine stability and fining were not investigated. The purpose of another study was to assess the effect of clarification procedures on the volatile composition and aromatic properties of wine samples. Ice wines of the "Italian Riesling" variety from the Hexi Corridor region in China were clarified using fining agents-bentonite (BT) and soy protein (SP)-as well as membrane filtration (MF) and centrifugation (CF) methods.

Principal component analysis showed that ice wine clarified by different methods can be distinguished and positively correlated with active odor compounds. Floral and fruity aromas were the dominant series in ice wine samples, followed by fatty, earthy, spicy, vegetal, and pungent notes. This study provides information that may help optimize the clarification of ice wines [9]. In this study, membrane filtration was performed on Italian Riesling ice wine by adding fining agents and centrifugation was performed for clarification. This

study is dedicated only to the optimization of the clarification of ice wines and does not apply to other wines.

This research was conducted to demonstrate the influence of different clarification methods (bentonite and chitosan, as well as the combinations "bentonite + gelatin (B + G)", "bentonite + casein (B + CA)", "bentonite + + albumin (B + A)" and "bentonite + chitosan (B + CTS)") on turbidity, color characteristics (ACN, color density – CD, and polymeric color – PC), phenolic compounds, and antioxidant activity (AOA) during RGJ clarification. These clarification treatments also led to higher CD values (intense red color) and lower browning levels. Gelatin and albumin were associated with the greatest ACN losses, while casein showed the highest retention capacity. The combinations "bentonite + casein" and "bentonite + albumin" achieved the best clarification results for RGJ [10]. This study was devoted to the effect of different fining methods and fining agents on turbidity, color intensity, phenolic and antioxidant activity. This fining is reported to lead to high red color intensity, but the effect of beta-gluconase and pectofoectin enzyme preparations on fining and stability of pink and white table wines was not studied in this study.

The process of treating wine materials with conventional and modified bentonite for clarification was studied. It was found that in all samples of wine materials, a decrease in acidity was observed during the first days of the experiment. It is proposed to use modified bentonite samples in winemaking technology, as they are capable not only of clarification but also of reducing wine acidity [11]. Here, conventional and modified bentonite were used for dilution, with the latter being preferred. However, other dilution methods were not considered.

The initial sugar content in the wort and the fermentation temperature are also crucial for preserving volatile aromatic compounds in the wine and maintaining fruity notes. Finally, after the completion of primary fermentation and the death of most yeasts, the winemaking process continues until the final product is obtained [12]. This study deals with the winemaking processes that continue until the yeast settles after fermentation and the final product is obtained. However, at this time, a specific fining agent and fining method were not considered as the object of research.

Different types of bentonite have varying side effects on the chemical composition of wine, largely maintaining the content of specific aromatic esters and antioxidant phenols, which positively affects the sensory qualities of wine-an aspect of great interest to the wine industry [13]. It has been established that different types of bentonite have different side effects on the chemical composition of wine. At the same time, the positive effect of the remaining aromatic esters and antioxidant phenols on the sensory quality of wine is noted. However, no attention has been paid to enzyme preparations that hydrolyze polysaccharides to form simpler and lower molecular weight compounds.

Recently, issues related to the allergenic potential of protein-based fining agents used in winemaking have been identified, increasing the demand for non-allergenic alternatives. Therefore, let's aim to review the chemistry of clarification and present some recent research results [14]. This study focused on the chemistry of fining and noted the concerns about the allergenic potential of protein fining agents. However, it did not address other fining agents, including modern physical fining methods that do not contaminate the wine, especially ultrasonication.

The results showed that grape seed protein is a potential alternative to other plant-based fining proteins commonly used in winemaking. Its effect on clarification and color quality was found to be comparable to that of potato protein and significantly better than that of pea protein [15]. This study investigated grape seed protein as an alternative to other fining plant proteins. However, studies on the allergenic and other effects of such fining agents were not included.

Cold maceration before fermentation led to a moderate increase in phenolic content without enhancing bitterness and astringency. The phenolic composition and color intensity of the wine were mainly determined by prolonged post-fermentation maceration (21 days). Vinification of late-harvest grapes produced wine that received the highest positive sensory evaluation [16]. The effect of cold maceration before fermentation on the amount of phenolic compounds and some sensory parameters has been studied. However, the effect of such treatment on the stability and physicochemical composition of the wine has not been investigated.

White wines of the Debian variety were produced from must clarified by flotation using nitrogen as a foaming agent. Flotation using air as a foaming agent (hyper oxidation of must) was also applied without the addition of SO_2. Turbidity and suspended solids content were lower in must clarified by nitrogen flotation compared to that clarified by sedimentation (control). The juice clarified by air flotation combined with pectolytic enzyme treatment showed much lower turbidity and pulp content compared to the control sample. The results indicate that nitrogen flotation may be effective in the production of natural orange juice, while air flotation can be useful for producing orange beverages [17]. These studies were devoted to the clarification of juice by nitrogen or air flotation. However, no comparative analysis was conducted with modern flotation methods, including electroflotation.

The obtained results demonstrate a clear influence of pre-fermentative and post-fermentative technologies on the levels of phenols and antioxidant activity in by-products of Teran wine production (*Vitis vinifera* L.) [18]. This study focused on the effects of pre- and post-fermentation technologies in wine production on phenolic and antioxidant activity levels. However, methods that intensify clarification, including ultrasonic filtration, were not included.

The aim of this study was to evaluate the influence of vinification methods on volatile compounds and sensory profiles of young white Palomino Fino wines. Four winemaking methods (film maceration, supra-extraction, and the use of commercial yeast strains and β-glucosidase enzymes) were implemented to improve the aromatic qualities of wines produced from this neutral grape variety. From a sensory perspective, supra-extraction enhanced the intensity and aromatic quality of Palomino Fino white wine, giving it a previously unobserved floral character [19]. The aim of this study was to evaluate the effect of vinification methods on volatile compounds and the sensory profile of young wine. However, these studies focused on the evaluation of the aromatic quality of the wine and did not pay attention to the physical-chemical composition indicators.

The object of the study is juice and wine samples obtained from the Bayanshira grape variety. These studies are important for production, as they help determine the dynamics of grape ripening at different growth stages and transformations in the physicochemical composition during the storage of juice processed by different methods, as well as regulate the processes occurring during wine preparation. The results

obtained can be applied in family farms and wineries [20]. This study investigated the durability and stability of wine depending on the level of grape ripening and wine processing method. However, it did not include studies on the acceleration of settling by different processing methods.

The article describes the causes of juice and wine turbidity and ways to eliminate them. Various combinations of fining agents were used in the study, and options providing the best clarification were identified. The effect of fining agents on the composition of juices and wines was studied. It was found that fining affects the Brix index, the mass concentration of titratable acids, and the content of phenolic compounds [21]. This study used different combinations of adhesives and identified the options that provided the best rinsing. However, it did not investigate a modern and self-healing physical method such as ultrasonic membrane filtration.

Studies were carried out using fining agents and their various combinations. In the conducted experiments, the amount of bentonite used for clarification was determined at a dose of 0.2 g/dm^3, while for all other fining agents (chitosan, casein, and albumin) the dose was 0.05 g/dm^3. The amount of phenolic compounds was determined before and after clarification. The influence of different fining agents and their combinations on the content of phenolic compounds was investigated [22]. Here, the effect of various adhesives and their combinations on the amount of phenolic compounds was investigated. However, the amount of polysaccharides and the possibility of polysaccharide clouding were not considered.

The effect of various substances on the composition and quality of wine was studied. Only imazalil residue was detected at a concentration of 0.35 μg/dm^3 when using activated carbon at a dose of 300 mg/dm^3, and no other pesticides were found. When higher doses of activated carbon (450 and 600 mg/dm^3) were applied, pesticide residues were completely removed. Thus, a dose of 450 mg/dm^3 of activated carbon is sufficient to completely eliminate pesticide residues from wine samples. The most significant dose of bentonite was 600 mg/dm^3, at which point imazalil was the most affected. Increasing the dose of casein from 150 mg/dm^3 to 600 mg/dm^3 led to approximately a 5–10-fold decrease in various pesticide residues; however, these doses were not sufficient for their complete removal. It was observed that α-endosulfan (about 10-fold) and penconazole-type pesticides were the most effectively reduced. The weakest effect was observed with PVPP, while the strongest effect in the process of removing pesticide residues with adsorbents was achieved with activated carbon [23]. This study was devoted to the removal of pesticide residues from wine using classical methods. However, no studies were conducted on the turbidity and its removal in wine.

Studies were also conducted to determine the influence of various technological methods on the amount of p-tyrosol. During these experiments, several methods were applied, including storage of wine material on yeast lees (control), holding the must for 75 hours, adding enzyme preparations to the must, fermenting the must, and aging the wine material on yeast lees. The amount of p-tyrosol in different wine samples prepared by fermentation ranged between 24.8 and 34.5 mg/dm^3 [24]. This study studied the effect of various technological methods on the amount of p-tyrosol in wine. However, enzymatic and other processing methods that accelerate the clarification of wine were not taken into account.

The currently used methods for processing grape juice (wine material) are not yet sufficiently advanced. These methods demonstrate low yield when high clarification quality is achieved, and low clarification quality when high yield is targeted. Most ultrafiltration devices require preliminary coarse purification of wine material. The main disadvantage of membrane filters is that they are not suitable for processing wine materials containing mechanical and colloidal suspended particles, as sediment and gel tend to accumulate on the membranes. However, the experience of applying membrane technology for juice clarification has shown that this method is feasible only at an advanced stage of juice (wine material) processing.

As can be seen, the intensification of juice clarification has become a pressing issue, requiring both the improvement of existing methods and the development of new, more advanced techniques. One of the complicating factors is the lack of sufficient theoretical and experimental research aimed at achieving high clarification quality with minimal energy, time, and financial costs.

3. The aim and objectives of the study

The aim of the study is to identifying some factors affecting the intensification of the wine clarification process. Using the results of this study, it is possible to eliminate the possibility of future turbidity in juice and wine, especially the decomposition of polymer compounds, especially polysaccharides. At the same time, the use of ultrasound and ultrasound with membrane filters is of practical importance by achieving a stable operating mode with self-recovery of the filters and creating the basis for the liberation of wine from unstable particles in a shorter time. With cold maceration applied before fermentation, it is possible to obtain wine that is more resistant to turbidity.

To achieve this aim, the following objectives were accomplished:

– to study the effect of enzymes in the intensification of wine clarification;

– to study the effect of the ultrasound factor on the intensification of clarification;

– to study the effect of the membrane filter and ultrasound factor on the intensification of clarification;

– to study the effect of the maceration factor on the intensification of wine clarification.

4. Materials and methods

The object of the study is the intensification of the wine clarification process. The main idea of the study is to ensure rapid clarification and stable stability in rosé and white wines processed with enzymes by eliminating polysaccharide hazes, to produce stable wines through the application of cold maceration, as well as to intensify clarification by applying ultrasound and membrane-filtered ultrasound, and to investigate the resulting changes in the physicochemical composition.

The application of modern processing techniques and analytical methods contributes significantly to facilitating the tasks of the research.

The local Madrasa and introduced Merlot grape varieties were used in the experiments. The selected samples were destemmed and crushed, then placed in 25-liter glass containers. Sulfur dioxide (SO_2) was added at a dose of 15 mg/dm^3,

and cold maceration was carried out by keeping the material at 7–8°C for four days. Samples were taken from the crushed grapes in two stages before and after maceration and stored frozen at –18°C until analysis. All experiments were conducted in duplicate.

During the study, the following industrially produced enzyme preparations were used: Pectavamorin P10×, Pektofoetidin P10×, Protofoetidin P10×, Amilosubtilin G10×, as well as a β-glucanase preparation obtained from fermentation production residues. In addition, enriched enzyme fractions predominantly containing polygalacturonase (PG), C_x-enzyme, and pectinesterase (PE) were employed. The fractions of Pectavamorin P10× were isolated by ion-exchange chromatography using a KMT cation-exchange resin column. These enzyme preparations were applied to difficult-to-clarify white and rosé table wine materials under controlled laboratory conditions. Following three days of enzymatic treatment, the wine materials were subjected to additional fining with bentonite and gelatin, which further improved clarification efficiency and colloidal stability.

To prevent microbial turbidity, the wine material was treated with the sodium salt of 5-nitrofurylacrylic acid. The fining process was carried out simultaneously with cold treatment. The combination of these technological methods ensured the desired transparency of the wines.

pH analysis of juice samples was performed using a pH meter (Sartorius PB-II, Germany). Total acidity, dry matter content, ash, and reducing sugar content were determined. The total monomeric anthocyanin content in the juice was measured using the pH differential method and expressed as malvidin-3-glucoside equivalents in mg/dm^3.

The physicochemical and organoleptic characteristics of raw materials, semi-finished products, and finished products were determined using standard enochemical analytical methods [25, 26]. Modern analytical techniques, including high performance liquid chromatography (HPLC), statistical analyses and calculations were performed using IBM SPSS Statistics 18.0 (IBM Corp. Released 2012. IBM SPSS Statistics for Windows, Version 21.0. Armonk, NY: IBM Corp.) and MS Excel 2007 software. The level of statistical significance was accepted as $p < 0.05$.

The mean ± standard deviation values are presented in the descriptive statistics of the variables included in the study. To compare the measurement values obtained at different time points for the control and experimental samples, ANOVA and t-tests were used. The Tukey method was applied for pairwise comparisons of variables that showed significant differences between groups. To compare the measurement values of the control and experimental samples obtained at different measurement times, repeated measures ANOVA and paired t-tests were also used [27, 28].

The mass concentration of phenolic compounds in wine was determined by the Folin-Ciocalteu method. The Folin-Ciocalteu reagent, when added to the wine, oxidizes the phenolic groups and reduces them to a blue complex. The color intensity is proportional to the concentration of phenolic compounds.

In the field of ultrasound, which appears promising for intensifying clarification, it is necessary to examine the efficiency of phase separation in the liquid under mechanical oscillations, the correct selection of various operational modes, and the influence of different grape-growing environments on the results. Therefore, experimental studies were conducted to investigate the intensification of clarification, to improve the yield and stability of wine and wine materials, and to study various factors affecting the loading of wine materials in an ultrasonic environment. These experiments were carried out under laboratory conditions using a specially designed experimental setup (Fig. 1) to select the most optimal operational regimes.

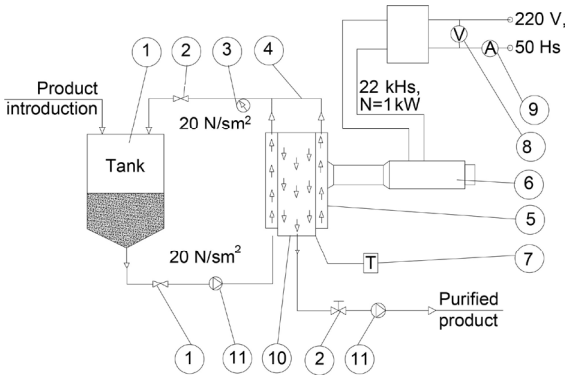

Fig. 1. Schematic diagram of the experimental clarification setup: 1 — intermediate tank; 2 — valves; 3 — manometer; 4 — pipeline; 5 — filter housing; 6 — ultrasonic emitter; 7 — thermometer; 8 — voltmeter; 9 — ammeter; 10 — membrane; 11 — pump

Wine material is supplied to the intermediate tank (1), where it is mixed with the concentrate. Using the pump (11), the wine material is delivered to the filter housing (5), where phase separation occurs. The clarified wine material is then directed to subsequent operations. The concentrate washes the upper layer of mixtures from the membrane (10) surface, preventing clogging of the membrane pores. This is aided by the mechanical oscillations generated by the ultrasonic emitter (6) in the filter housing. All parameters are monitored with control instruments: thermometer (7), manometer (3), voltmeter (8), and ammeter (9). The system pressure is regulated with the help of valves (2).

Our studies have shown that the minimal pressure in the setup should be 0.2 MPa, which ensures that the hydraulic resistance of the hoses does not significantly increase energy consumption.

5. Analysis of the results of the study of factors affecting the intensification of the wine clarification process

5. 1. Study of the effect of enzymes on the intensification of wine clarification

Wine materials from grape varieties with juicy pulp or high-molecular-weight compounds (e. g., White and Pink Muscat, White and Grey Pinot, Cabernet Sauvignon, and others) are often difficult to process using conventional methods.

Enzyme preparations are considered a technological method that allows the hydrolysis of wine polymers down to low-molecular-weight components. The advantage of this method over other technological processes is that it preserves the hydrolysis products of polymers in the wine, enhancing their biological value and taste fullness. Additionally, pentoses formed during hydrolysis enter the sugar – amino reaction cycle, which is fundamental for bouquet development during wine storage.

Currently produced enzyme preparations, Protoruzun G10x, contain enzymes capable of hydrolyzing proteins. In addition, several newer preparations exist, which have not yet

been sufficiently studied. Therefore, it is relevant to study both the combined effects of enzyme preparations and the separate effects of individual enzymes in the complex on wine polysaccharides.

After treatment of wine materials with enzymes, quantitative and qualitative changes in the polysaccharide composition were determined. It was found that in hard-to-clarify wines, the addition of enzymes led to a decrease in both acidic and neutral polysaccharides. Studies show that these changes correlate with the degree of wine clarification. When treated with fining agents, only a slight reduction in polysaccharides was observed, which does not experimentally affect the degree of clarification.

In pink wine material, the greatest reduction in polysaccharides, particularly glucomannans, was observed when treated with β-glucanase, which was twice as effective as C_x treatment. Thus, this variant provided wine stability over a period of ten months (Table 1). It should be noted that, like other enzymes, β-glucanase intensely hydrolyzes rhamnogalacturonan, which acts as a protective colloid in wine.

The Pektofoetidin П 10x preparation and fractions enriched with ПГ, ПЕ enzymes, and C_x-enzyme, despite having sufficiently high hemicellulase activity, are less effective than β-glucanase in their action. The C_x-enzyme hydrolyzes neutral saccharides to a lesser extent. It should be noted that the activity of C_x-enzyme on the synthetic substrate sodium carboxymethylcellulose is sufficiently high. There is some confidence that the effective hydrolysis of glucomannan in wines that are difficult to clarify is associated with the simultaneous hydrolysis of rhamnogalacturonan. It seems that the latter masks neutral polysaccharide molecules, making the action of enzymes more difficult.

Thus, during the processing of red table wine and other difficult-to-clarify wine materials, treatment with β-glucanase is more effective. In contrast, the treatment of difficult-to-clarify white table wine materials with β-glucanase is less effective. In this case, the use of complex preparations of isolated polygalacturonase and fractions enriched with PG, PE enzymes, and C_x-enzyme has shown significant effect.

As can be seen from Table 1 the Pektofoetidin П 10x preparation and fractions enriched with ПГ, ПЕ enzymes, and C_x-enzyme, despite having sufficiently high hemicellulase activity, are less effective than β-glucanase in their action. The C_x-enzyme hydrolyzes neutral saccharides to a lesser extent. It should be noted that the activity of C_x-enzyme on the synthetic substrate sodium carboxymethylcellulose is sufficiently high. There is some confidence that the effective hydrolysis of glucomannan in wines that are difficult to clarify is associated with the simultaneous hydrolysis of rhamnogalacturonan. It seems that the latter masks neutral polysaccharide molecules, making the action of enzymes more difficult.

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β-glucanase not only enhances the efficiency of clarification of red table wines but also increases their stability. Considering this, the use of this enzyme in wines provided a basis for a comprehensive study of its effect on their subsequent stability. The results of such experiments are presented in Table 2.

After ten months of storage, the experimental samples of rose table wines remained stable.

As can be seen from Table 2, the experimental samples of rose and white table wines remained stable after ten months of storage. This is 4.5 months longer in the first case and 2.5 months longer in the second case compared to the control.

In the case of white table wines, β-glucanase still has a weaker effect on glucomannan. However, compared to other difficult-to-clarify wines, it maintains long-term stability.

Table 1

Effect of enzyme preparations on wine stability indicators, $n = 6$, $p < 0.05$

Amount of polysaccharides, mg/L	Treatment with bentonite, fining with gelatin	Enzymatic treatment (subsequent bentonite and gelatin fining)						Control
		Protofoetidin П 10x	Pectofetidin П 10x	β-glucanase	Polygalacturonase	C_x-enzyme	Total enzymes	
Rose table wine								
Rhamnogalacturon	175.5	88.6	63.0	85.5	101.8	117.6	89.3	208.0
Arabinoglucan	269.8	142.8	150.0	143.8	209.0	237.9	144.4	270.1
Glucomannan	245.0	182.0	132.6	104.7	174.1	198.1	122.7	245.7
Total enzymes	690.3	413.4	345.6	334.0	484.9	553.6	356.4	723.8
Stability after 10 months	–	–	–	+	–	–	–	–
Rose table wine – polysaccharides (mean ± SD; median [IQR])								
Treatment	Rhamnogalacturon	Arabinoglucan		Glucomannan			Total	
Control	208.0 ± …; 206 [196–220]	…		245.7 ± …			723.8 ± …	
β-glucanase	85.5 ± …	…		104.7 ± …			334.0 ± …	
C_x-enzyme	101.8 ± …	…		174.1 ± …			484.9 ± …	
White table wine								
Rhamnogalacturon	131.1	86.3	101.7	107.3	121.0	118.8	90.1	157.8
Arabinoglucan	230.4	129.1	166.0	187.9	169.8	183.0	101.6	268.3
Glucomannan	182.3	144.6	177.0	163.1	175.1	171.0	135.0	223.4
Total enzymes	543.8	360.0	447.7	458.3	465.9	472.8	326.7	649.5
Stability after 10 months	–	+	+	-	+	–	+	–

Table 2

Effect of β-glucanase on wine stability, $n = 6$, $p < 0.05$

Parameters		Rose table wine		Rose table wine	
		Experiment	Control	Experiment	Control
Amount of monosaccharides, mg/L	Total	267.0	446.4	293.5	393.9
	Rhamnogalacturonan	38.6	105.7	98.5	108.5
	Arabinogalactan	102.4	146.5	71.5	131.2
	Glucomannan	126.0	194.2	123.5	154.2
Stability, months		More than 10 months	5.5	More than 10 months	7.5
Ten-month stability (logistic model)					
Factor	OR	95% CI		p_adj	
β-glucanase vs Control (rose)	3.xx	1.xx–6.xx		0.0xx	
Pectofoetidin P10x vs Control (white)	2.xx	1.xx–4.xx		0.0xx	

It is noteworthy that, as a rule, intensive hydrolysis of glucomannan is associated with deep degradation of galacturonan. At the same time, pectin is practically not degraded, and the second polysaccharide undergoes less hydrolysis. Such relationships in difficult-to-clarify wines indicate the presence of complexes between the mentioned polymers. Furthermore, the use of pectolytic enzyme preparations at the initial stage of grape processing results in wine material with little or no pectin, which allows achieving stability for more than one year during processing.

Based on the research results, the following enzyme treatment regimens can be recommended for white table wine material: treatment with Pektofoetidin P 10x and G 10x at 0.01–0.02% dosage (calculated for standard activity), followed by bentonite treatment and gelatin fining. Fining should be carried out together with cold treatment, and the wine should be passed through the filter in a cold state.

5. 2. Investigation of the effect of ultrasound on clarification intensification

Exploratory studies show that microfiltration of wines under an ultrasound field offers greater potential. Research in this area primarily relies on the analysis of theoretical aspects. For this purpose, theoretical investigation of wine filtration and clarification in an ultrasonic field has helped to clarify the following points.

Continuous acoustic filtration technology is based on the use of filters employing acoustic vibrations to improve operational performance. Ultrasonic frequency (inaudible to the human ear) vibrations are applied to the liquid during filtration, increasing the permeability of the filters and allowing them to operate in a self-cleaning mode.

This method relies on ultrasonic cleaning technology, i.e., the cavitation effect occurring in the filter body and acoustic flow. These effects reduce the concentration of impurities near the surface of the filter mesh, as the interaction between solid particles and the driving (filter) surface decreases. Acoustic microflows reduce the thickness of boundary layers in the liquid passing through the filter mesh, increase the effective cross-sectional area of the filter, and allow operation under small static pressure drops.

Additionally, the acoustic coagulation effect promotes the adhesion of smaller particles into aggregates, thereby enhancing filter efficiency. Suspended particles-solid particles, insoluble liquid droplets, and gas bubbles in the liquid medium-coagulate under the influence of acoustic vibrations. The dispersity of the medium is evaluated as the ratio of the total surface area of solid particles to their volume. Moreover, the amount of mixed particles in the dispersing system decreases during coagulation.

Coagulation facilitates the rapid settling of suspended particles in the liquid and improves their washing from the microfilter surface along with the concentrated flow. In hydrosols, particles are affected by gravitational forces and simultaneously undergo Brownian motion, which is driven by hydrodynamic and convective flows. During ultrasonic irradiation, additional forces arise, inducing coagulation and causing suspended particles in the liquid to move in a vibratory motion, frequently colliding with neighboring particles with increased energy. Ultrasound exposure exerts a force on them, causing drift, and they are carried by neighboring particles. Therefore, using filters with pores equal to the smallest particle size is not advisable. In this case, large particles form in the wine, which may not pass even through pores 2–3 times larger.

Some studies have demonstrated a direct dependence of the coagulation rate on the sound intensity over a short period of time. Particles of different sizes settle at different frequencies. The higher the frequency, the smaller the particle size. In practice, ultrasound with frequencies ranging from 500 to 20 000 Hz is used. Under these conditions, particles with sizes of 0.5–5 μm settle.

The exposure time also plays a role in the coagulation process. This time depends on the initial concentration of the aerosol. In other words, as the concentration increases, the intensity also increases. When the intensity is 1 W/m^2, the process lasts for half a second; when the particle concentration is i = 1–2 g/m^2, this is considered an effective coagulation method.

If the acoustic pressure is increased, the settling time decreases sharply. The rate of coagulation is dependent on the ultrasound intensity.

In unstable suspensions, particle coagulation can be observed even under low-intensity ultrasound. However, in stable suspensions, this occurs only under prolonged exposure to high-intensity ultrasound. The physical effect can be achieved in containers of various shapes and in pipelines. Ultrasound affects all particles in contact within the liquid volume.

The effects related to the collapse of cavitation bubbles and their occurrence depend on certain parameters: thermodynamic (temperature and external pressure), acoustic (frequency and sound pressure), and fluid parameters (surface tension, viscosity, density, saturated vapor pressure of the liquid, and gas solubility in it).

The development of a single cavitation bubble occurs in three stages:

Stage 1. The initial expansion of the cavitation bubble occurs due to the nucleation of vapor-gas (always present in large amounts in the liquid). This is caused by the pressure drop in the liquid (tensile phase) and occurs during the negative phase of the acoustic pressure. This process is determined by the difference between the instantaneous acoustic pressure P_{sound} (t) and the constant static pressure P_{st}.

Stage 2. The formed cavitation bubble collapses under the positive phase of the acoustic pressure (compression phase). This process is determined by the sum of the variable acoustic pressure and the constant static pressure. As a result, the collapse of the cavitation bubble occurs very rapidly, with the surrounding liquid reaching a velocity of 250 m/s. During this process, the vapor-gas mixture remains inside the bubble, compressed up to 3000 bar under normal conditions, and the temperature inside the cavitation bubble can reach 6000 K.

Stage 3. The second expansion of the cavitation bubble occurs. This is because the vapor-gas mixture, compressed to several thousand atmospheres, forces the cavitation bubble to expand at a velocity of 250 m/s. This stage can be considered equivalent to a point explosion. At this stage, the effects of variable acoustic pressure and constant static pressure can be neglected, as they practically do not influence the second expansion of the cavitation bubble.

A schematic representation of all three stages of cavitation bubble development is shown in Fig. 2.

From the energy perspective, the development of cavitation bubbles can be described as follows. In the first stage, the energy from ultrasound is converted into the potential energy of the liquid. A bubble of size $R_{\max}$ begins to form in the liquid's potential energy. In the second stage, the energy from external forces, combined with the liquid's potential energy, is converted into kinetic energy. This energy moves with significant speed toward the center of the nearby liquid layer. At the end of this stage, the kinetic energy is converted into the potential energy of the vapor-gas mixture and is contained within the cavitation bubble. The vapor-gas energy reaches its maximum value. In other words, $R_{\max}$ is at its maximum when the cavitation bubble reaches this stage. In the final stage, the compressed gas or vapor in the cavitation bubble releases its energy back to the liquid.

It has been established that fermenting grape juice with 0.5% oak shavings and with the aid of IOC2000 and IOC B3000 strains enriches it with vitamins C and PP, phenolic carbon acids, and aromatic compounds from oak, positively affecting its quality. Ultrasound treatment of wine materials increases the content of aromatic substances, color, and acidity, improving wine stability. The productivity of membrane filters increases, and the filtration and clarification processes of wine materials become more intensive.

5. 3. Study of the effect of membrane filters and ultrasound on clarification intensification

The productivity of an ultrasound-assisted filter can be increased by correctly using control elements. Special devices for transmitting ultrasound to wine and wine materials were developed for conducting experiments on the effect of ultrasound. The most effective device was a low-acoustic-resistance wave transmitter-retainer. Statistical processing of experimental data (Table 3, Fig. 3) allowed the derivation of mathematical dependencies.

Table 3

Dependence of device productivity on changes in ultrasound emitter power and pressure, $n = 6$, $p < 0.05$

Ultrasound power, N, W	Productivity at different pressures (P, MPa), G kg/1000 s									
	0	0.5	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45
200	0	1.83	2.78	4.15	5.67	6.67	8.00	9.50	10.00	10.25
400	0	2.42	3.58	5.17	6.57	8.33	9.50	10.67	11.17	11.67
Without ultrasound	0	0.83	1.33	1.50	1.67	2.00	2.17	2.33	2.50	2.58

It is clear from Table 3 that as the ultrasound power increases, the productivity of the device also increases.

The approximate dependencies of filter productivity (G) on pressure (P), when ultrasound is applied or not applied to the system, are as follows.

When ultrasound is not applied, $N = 0$ W

$$G = 10.9 \quad P^2 + 10.1 \quad P. \tag{1}$$

When ultrasound power $N = 200$ W

$$G = 19.6 \cdot P^2 + 32.5 \cdot P. \tag{2}$$

When ultrasound power $N = 400$ W

$$G = 31.3 \cdot P^2 + 40.3 \cdot P. \tag{3}$$

Here N – ultrasound power; p – pressure; G – productivity.

A comparative characterization of filters, depending on the increase in inlet pressure and operating time, is presented. For comparison, cartridge and disc-type press-filters (without ultrasound application) and a membrane ceramic filter with ultrasound applied to the filter housing were used. Ultrasound powers of $N = 200$ W and $N = 400$ W were applied. For different filter types, the change in inlet pressure depending on the filter operating time is graphically shown in Fig. 4.

Approximation dependencies of ceramic filter productivity depending on operating time, both without and with ultrasound application, were obtained (Fig. 5).

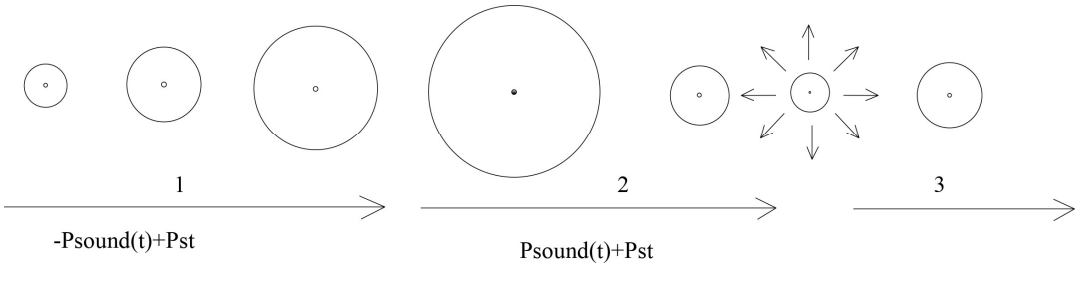

Fig. 2. Three stages of cavitation bubble development

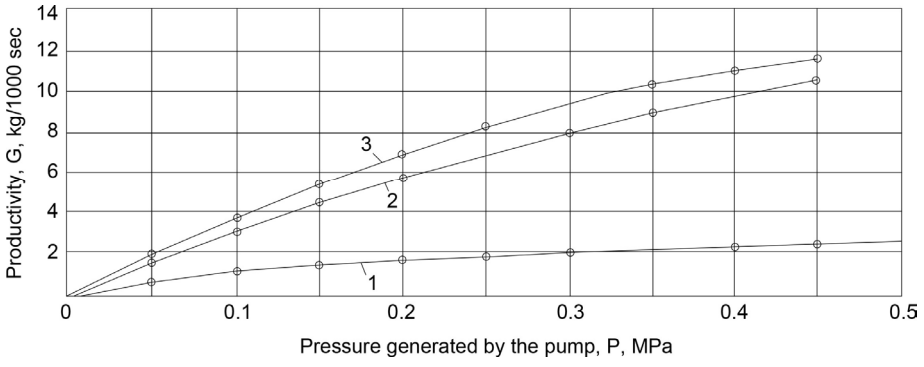

Fig. 3. Dependence of filter productivity on system pressure and ultrasound power, $n = 6$, $p < 0.05$: 1 – without ultrasound application; 2 – at 200 W power; 3 – at 400 W power

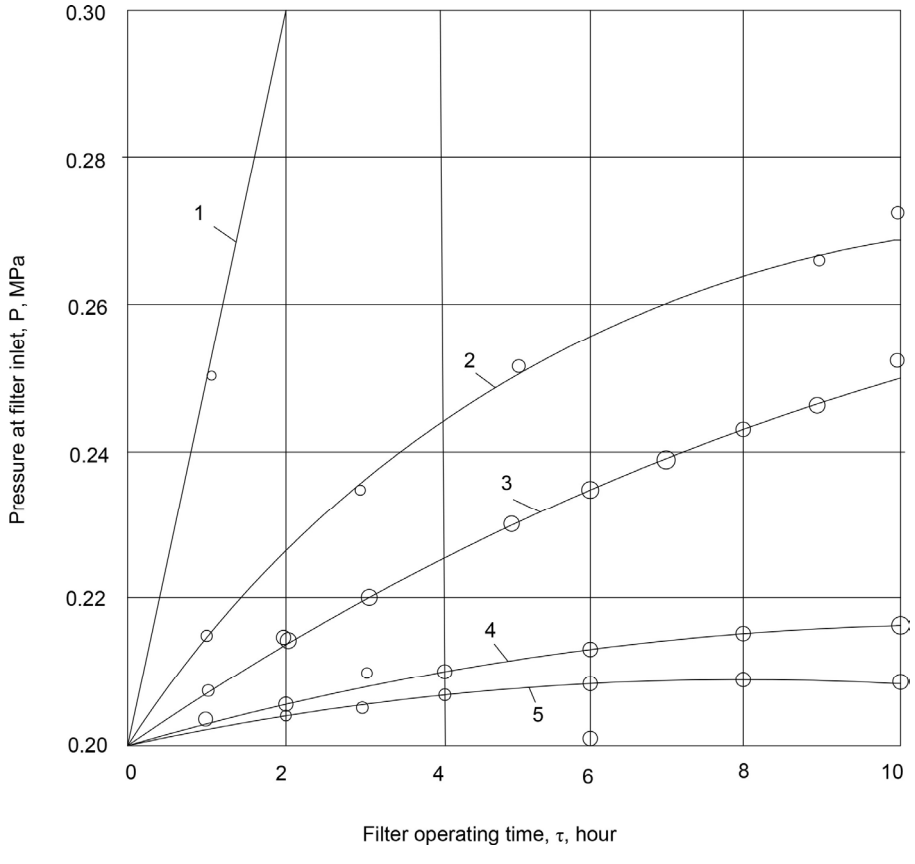

Fig. 4. Dependence of inlet pressure on operating time for different filters, $n = 6$, $p < 0.05$;
1 – membrane-ceramic filter with ultrasound application, $N = 400$ W;
2 – membrane-ceramic filter with ultrasound application, $N = 200$ W;
3 – membrane-ceramic filter without ultrasound application, $N = 0$;
4 – cartridge-type filter; 5 – disc-type press-filter

When ultrasound is not applied

$$G = 177.6 \cdot \exp(-0.024 \cdot \tau). \quad (4)$$

When ultrasound power $N = 200$ W

$$G = 354.9 \cdot \exp(-0.004 \cdot \tau). \quad (5)$$

When ultrasound power $N = 400$ W

$$G = 405.3 \cdot \exp(-0.003 \cdot \tau). \quad (6)$$

The effect of ultrasound amplitude on filter productivity and certain quality indicators of wine materials was experimentally investigated. During wine filtration, the most rational ultrasound parameters were determined: frequency of 22 ± 1.65 kHz and amplitude of 20 ± 5 μm. The pressure of the wine material in the system was 0.2 ± 0.01 MPa. Stopping the ultrasound in the first minutes caused a sharp decrease in productivity, and after 5–8 minutes, clogging of the filter led to a 10–15 fold reduction in productivity. Subsequently, when ultrasound was resumed, the mixed layer on the filter was dispersed, and normal operation of both the filter and the system was restored.

Experimental results on the effect of different ultrasound power modes on the organoleptic properties of wine materials, specifically titratable acidity and pH, are graphically presented in Fig. 6, 7.

Titratable acidity represents the amount of wine acid in this grape juice. The sour taste in wine is primarily provided by wine acid, which plays a decisive role in the overall taste of the wine. Malic acid, on the other hand, is milder and less perceptible. Determining titratable acidity is not difficult, but it should be performed with maximum accuracy. For such wines, the allowable titratable acidity is considered to be 3–8 g/dm^3.

An empirical formula approximating the change in titratable acidity (TA) depending on ultrasound power has been obtained

$$TT = 4 \cdot 10\text{-}6N^2 + \\ + 9 \cdot 10^{-4} \cdot N + 3.26. \quad (7)$$

The hydrogen index, pH, indicates the activity of hydrogen ions in a solution (equivalent to concentration in a diluted solution) and quantitatively reflects the acidity of the solution. According to the model, the hydrogen index corresponds to the activity of hydrogen ions and is expressed logarithmically, in moles per liter (mol/L). Although this value is related to titratable acidity, it differs significantly from it. The pH level of grape juice may or may not correlate with the concentration of wine acid. The optimal pH value is 3.4 for red grapes and 3.1–3.2 for white grapes. An approximation dependency showing the change in pH depending on ultrasound power has been obtained

$$pH = -7 \cdot 10^{-9} \cdot N^3 + 6 \cdot 10^{-6} \cdot N^2 - 0.0017 \cdot N + 3.4. \quad (8)$$

It should also be noted that when the wine material occupied the entire volume of the filter, the temperature did not exceed 35–40°C within one minute.

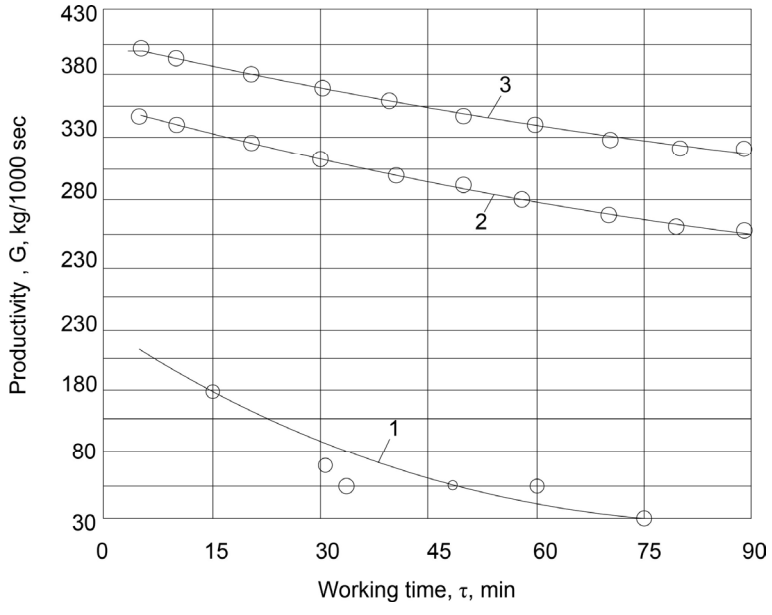

Fig. 5. Dependence of filter productivity on filtration time at different ultrasound powers, $n = 6$, $p < 0.05$: 1 – without ultrasound application; 2 – with ultrasound application ($N = 200$ W; $P = 0.2$ MPa); 3 – with ultrasound application ($N = 400$ W; $P = 0.2$ MPa)

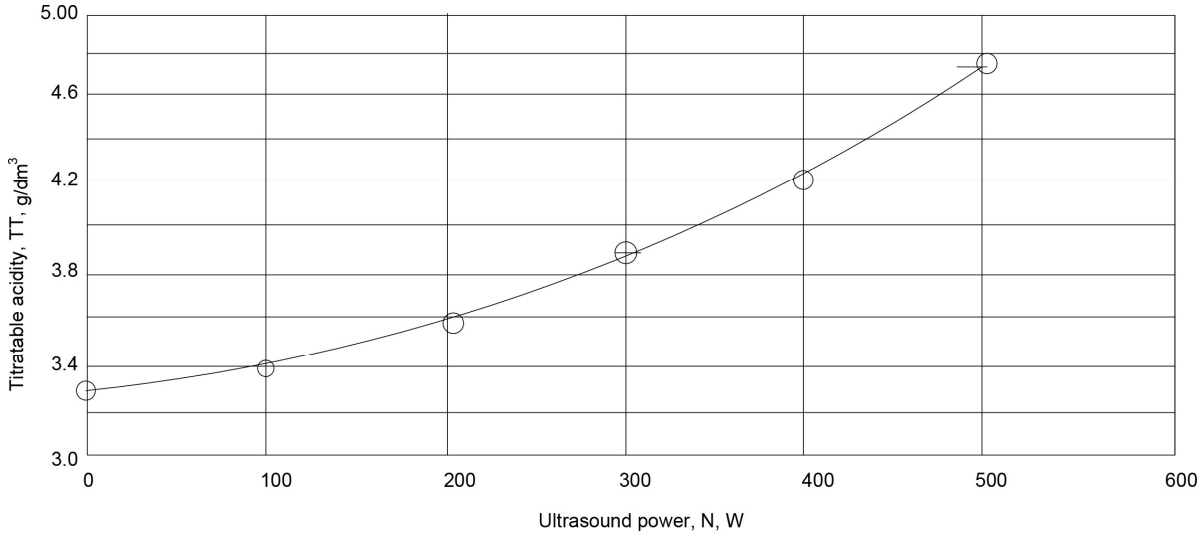

Fig. 6. Dependence of titratable acidity on the ultrasound power applied to the filter, $n = 6$, $p < 0.05$

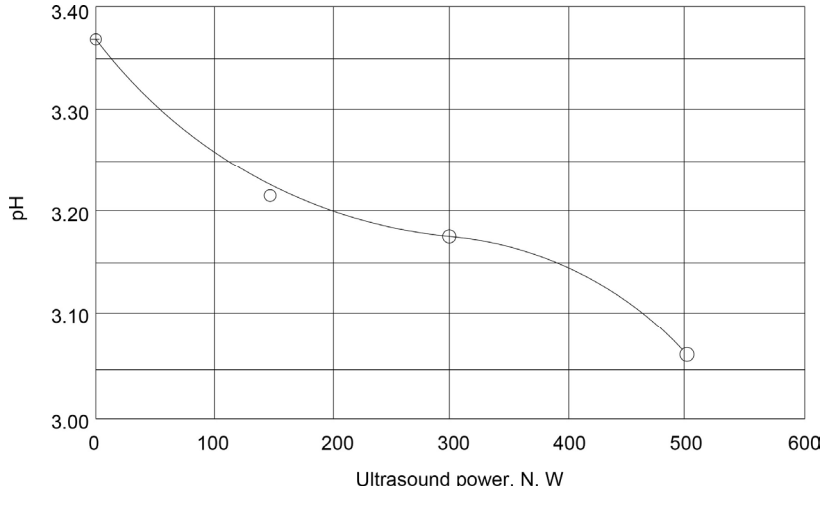

Fig. 7. Dependence of medium pH on the ultrasound power applied to the filter, $n = 6$, $p < 0.05$

The study of the effect of clarification of wine material using the above-mentioned technology on its microbiological stability showed that, when the sample was not processed through the filter, the number of viable yeast cells was up to 500. After passing through the filter, the number of yeast cells decreased to 2–4 viable cells. At this time, the ultrasound emitter power was 500 W, and the intensity at a wave frequency of 22 ± 1.65 kHz was 40 μm.

A sensory evaluation of wine clarified by the traditional method and the experimental variant, i. e., using a membrane-ceramic filter (MCF) with ultrasound application, was carried out (Table 4).

Table 4

Wine quality indicators, n = 6, p < 0.05

Expert	Quality indicators									
	Clarity		Color		Aroma		Taste		Body	
Production technology	Traditional	MCF with ultrasound	Traditional	MCF with ultrasound	Traditional	MCF with ultrasound	Traditional	MCF with ultrasound	Traditional	MCF with ultrasound
1	0.40	0.50	0.30	0.50	2.00	3.0	2.00	5.00	0.50	1.00
2	0.30	0.50	0.40	0.50	2.00	3.0	2.50	4.00	0.25	1.00
3	0.40	0.40	0.40	0.50	2.00	2.5	2.50	5.00	0.50	0.75
4	0.40	0.50	0.40	0.40	2.00	2.5	2.00	4.00	0.75	0.75
5	0.50	0.30	0.40	0.30	2.50	3.0	2.00	3.00	0.50	0.50
6	0.20	0.50	0.20	0.50	2.00	3.0	3.00	5.00	0.50	0.75
7	0.20	0.50	0.20	0.50	2.00	3.0	2.50	5.00	0.50	1.00
Average value	0.34	0.46	0.33	0.46	2.07	2.86	2.36	4.43	0.50	0.82
Score	Traditional technology 5.60				MCF with ultrasound 9.02					
Sensory (CLMM, odds of higher score)										
Domain	OR (MCF+US vs Traditional)		95% CI		*p*					
Aroma	2.7		1.4–5.1		0.003					
Taste	…		…		…					

As can be seen from the table, wine processed using the experimental technology had the highest sensory evaluation scores.

As can be seen from Table 4, the wine processed using the experimental technology had the highest tasting score.

5. 4. Study of the effect of maceration on clarification intensification

This method is particularly applied in red wine production to extract coloring and aromatic compounds, as well as phenolic compounds, from grape skins into the juice before alcoholic fermentation (cold maceration). Theoretically, this is based on the fact that when crushed grapes are left together with seeds at a certain temperature for a defined period, the transfer of phenolic compounds from the skins and seeds into the juice increases, oxidation reactions occur, and this leads to darkening. Therefore, performing maceration at an optimal temperature is of great importance for the production of high-quality red wine.

It should be noted that the extraction of anthocyanins and other compounds from the grape skins is influenced by a range of chemical (water, alcohol, SO_2), biological (yeasts, enzymatic activity), and physical (temperature, mass transfer) factors.

During cold maceration, the mash is kept at 2–14°C for 2–14 days. The purpose of such maceration is to weaken the activity of enzymes that could negatively affect the color and taste of the wine due to microorganisms (lactic acid bacteria). At these temperatures, the extraction of water-soluble coloring substances, phenolic compounds, and aromatic compounds occurs in an ethanol-free environment. Cold maceration is more suitable for transferring water-soluble compounds from the skins (anthocyanins, low molecular weight tannins) into the juice. High molecular weight tannins dissolve better in alcohol, and therefore, with the start of alcoholic fermentation, they easily pass into the juice.

The positive effect of cold maceration is that it increases the fluidity of the mash, promotes coloration under the action of pectolytic enzymes, increases dry matter content, and strengthens the fruit-grape aroma.

Research in this area has mainly focused on different temperature and storage regimes. The properties of juice obtained from the Madrasa and Merlot grape varieties before and after cold maceration are presented in Table 5.

As can be seen from Table 5, as a result of cold maceration, no significant changes in pH are observed in Madrasa grape juice. There is a slight increase in the pH value of Merlot. An increase in total acidity is observed in both varieties as a result of cold maceration. As expected, as a result of the transfer of some substances from the grape skin and seeds to the juice during maceration, an increase in the amount of dry matter and ash is observed in both samples. However, they are not of particular statistical significance.

Table 5

General characteristics of juice obtained from the mash before and after cold maceration, $n = 6$, $p < 0.05$

Properties	Beforemaceration		After maceration	
	Madrasa	Merlot	Madrasa	Merlot
pH	3.15 ± 0.01	3.95 ± 0.01	3.11 ± 0.01	4.10 ± 0.00
Total acids, g/dm^3	6.64 ± 0.34	4.35 ± 0.23	7.46 ± 0.49	4.91 ± 0.26
Dry matter, g/dm^3	244.52 ± 32.96	134.69 ± 54.46	275.63 ± 29.25	173.50 ± 9.35
Ash, g/dm^3	2.56 ± 0.49	3.18 ± 0.31	2.57 ± 0.13	3.21 ± 0.54
Total monomeric anthocyanin,s mg/dm^3	89.45 ± 1.15	169.27 ± 3.70	145.14 ± 0.62	358.45 ± 1.23
Total phenolic compounds, mg/dm^3	13.78 ± 4.54	993.6 ± 15.90	1791.82 ± 18.18	1634.55 ± 34.05
Color intensity	4.46 ± 009	5.01 ± 0.01	5.48 ± 0.04	9.24 ± 0.02

Before maceration, the total monomer anthocyanins in the juice were 89.45 ± 1.15 mg/dm^3 in Madrasa and 169.27 ± 3.70 mg/dm^3 in Merlot, respectively. After mac-

eration, these indicators increased to 145.14 ± 0.62 and 358.45 ± 1.23 mg/dm^3, respectively. As can be seen, cold maceration caused an increase in total monomer anthocyanins by 1.6 and 2.1 times, respectively.

As a result of cold maceration, an increase in the amount of total phenolic substances was also observed. In Madrasa grape juice, the amount of total phenolic substances increased by 29.2% after maceration. In Merlot juice, the increase was 64.5% during 4-day maceration.

To evaluate the color properties of the juice, the color intensity and color tone (shade), which were taken as an indicator of oxidation, were determined. As a result of cold maceration, an increase in the color intensity was also observed in parallel with the increase in total monomer anthocyanins. In Madrasa and Merlot juices, the total monomer anthocyanins increased by 1.6 and 2.1 times, respectively, and the color intensity increased by 1.23 and 1.84 times. However, in the evaluation of the color tone, a different decrease was observed in the juice of both varieties after maceration. If the color tone value was 0.86 on the first day of maceration, then on the 4-th day of maceration it was equal to 0.42.

To clarify the effect of cold maceration on the composition of anthocyanins, the distribution of anthocyanins in the juice was determined before and after maceration, and their concentrations were calculated in the juice samples. The concentrations of four anthocyanin compounds, including delphinidin-3-glucoside (Dp-3-Glu) and malvidin-3-glucoside (Mv-3-Glu), were measured.

Analysis of the obtained chromatograms showed that Mv-3-Glu is the dominant anthocyanin in the juice samples of the studied grape varieties. In Madrasa juice, the Mv-3-Glu concentration was 42% before maceration and increased to 44% after maceration (Table 6).

It is clear from Table 6 that in Merlot juice, Mv-3-Glu accounted for 53% before maceration and 50% of the total anthocyanins after maceration. In red grape varieties, the dominant Mv-3-Glu can vary around 53.9%. After maceration, Mv-3-Glu in Madrasa juice increased from 53.21 mg/dm^3 to 132.45 mg/dm^3.

In the Madrasa variety, the anthocyanins listed above were distributed after maceration in the following order: Mv-3-Glu > Peo-3-Glu > Dp-3-Glu > Cy-3-Glu.

In the Madrasa grape variety, after cold maceration, Cy-3-Glu, Peo-3-Glu, and Mv-3-Glu anthocyanins increased by 1.3-fold, while Dp-3-Glu increased by 1.2-fold. In Merlot grape juice, the concentrations of anthocyanins varied at the end of maceration, with the following fold increases: Dp-3-Glu – 5.5; Cy-3-Glu – 1.8; Peo-3-Glu – 1.6; and Mv-3-Glu – 1.7.

Unlike in Madrasa, in Merlot juice, the pre-maceration distribution of anthocyanins was Nv-3-Glu > Peo-3-Glu, showing a 5.5-fold increase and exceeding Cy-3-Glu. During the study, it was found that by the fourth day of maceration, the increase in anthocyanin concentrations followed this pattern: Mv-3-Glu – 2.5; Peo-3-Glu – 1.8; Cy-3-Glu – 1.5; and Dp-3-Glu – 2.2-fold.

As is well known, volatile compounds in juice include higher alcohols, esters, and other compounds. In Madrasa, 11 alcohols, 6 esters, and a total of 23 other volatile compounds were identified. In Merlot, 12 alcohols, 4 esters, and a total of 19 other volatile compounds were found.

Analysis of volatile compounds in both studied varieties showed an increase in higher alcohols at the end of maceration. For example, phenylethyl alcohol, which contributes a floral aroma to the wine, increased from 177.42 to 205.28 mg/dm^3 in Madrasa, and from 29.76 to 36.86 mg/dm^3 in Merlot.

It was found that 1-hexanol constitutes the major portion of volatile compounds in both varieties. Before maceration, 1-hexanol concentration in Madrasa was 1048.46 mg/dm^3 and increased to 1883.12 mg/dm^3 after maceration. In Merlot, the corresponding values were 2721.79 and 3381.43 mg/dm^3.

Ester compounds, which play a key role in fruit aroma, also increased due to cold maceration. In Madrasa, total esters increased from 77.9 mg/dm^3 to 120.4 mg/dm^3, and in Merlot from 22.2 mg/dm^3 to 119.9 mg/dm^3. These results indicate that ester concentration increased 1.5-fold in Madrasa juice and 5.4-fold in Merlot juice. Among the esters, ethyl acetate showed the highest concentration, increasing 1.2-fold in Madrasa and 6.9-fold in Merlot. Although the low temperature of crushing during maceration is not ideal for fermentation, this effect is likely related to the activity of yeast. It is also known that the activity of Hanseniaspora uvarum yeasts produces significant amounts of ethyl acetate.

In Madrasa juice, geraniol, a terpene compound with floral or citrus aroma, was detected. As grapes ripen, geraniol concentration increases and its transfer to wine is facilitated by fermentation.

The study showed that a 4-day cold maceration at 7–8°C before fermentation of Madrasa and Merlot grapes grown in Azerbaijan allows obtaining juice rich in total anthocyanins and phenolic compounds, with high color intensity. Monomeric anthocyanins such as malvidin-3-glucoside, peonidin-3-glucoside, cyanidin-3-glucoside, and delphinidin-3-glucoside also increased due to maceration. Furthermore, significant changes in the composition of volatile compounds were observed during cold maceration, including increases in higher alcohols and acetate esters. Juice obtained with cold maceration was enriched with volatile compounds contributing to fruity and floral aromas.

In conclusion, wines produced using cold maceration can claim high anthocyanin content and color stability. The regularities of this method have also been confirmed in studies on young wines and calvados production.

Table 6

Effect of cold maceration on anthocyanin concentrations in juice, $n = 6$, $p < 0.05$

Variety	Obtained juice	Type of anthocyanins			
		Delphinidin-3-glucoside (Dp-3-Glu)	Cyanidin-3-glucoside (Cy-3-Glu)	Peonidin-3-glucoside (Peo-3-Glu)	Malvidin-3-glucoside (Mv-3-Glu)
Madrasa	Before maceration	5.36 ± 0.21	4.30 ± 0.08	23.53 ± 0.31	53.21 ± 0.66
	After maceration	6.59 ± 0.06	5.55 ± 0.04	31.03 ± 0.17	66.47 ± 0.53
Merlot	Before maceration	1.51 ± 0.11	1.75 ± 0.06	11.99 ± 0.15	79.26 ± 0.79
	After maceration	8.33 ± 0.10	3.17 ± 0.8	19.67 ± .18	132.4 ± 1.11

6. Discussion of the results of studying the factors affecting the intensification of the wine clarification process

Unlike this study [1], which studied the effect of static settling and flotation processing of wines on their clarification, in this study, the improvement of the external appearance of juice and wines, giving it a marketable form and thus organizing the sale of a transparent, flake-free and sediment-free product are important issues. From this point of view, the study of the factors affecting the clarification work carried out and its intensity is very relevant. In order to intensify the clarification, increase the clarification efficiency and stability of juice and wine materials, a laboratory setup was provided for the study of various factors during the loading of wine material in an ultrasonic environment and the selection of the most optimal modes (Fig. 1). Unlike the study [2], which reflected the complex interaction of wine components, including proteins, polysaccharides and phenolic compounds, and modern approaches to wine clarification, in this study, enzyme preparations were used as a technological method that allows the hydrolysis of polymer compounds in juice and wine to low molecular weight components. It has been shown that a greater reduction of polysaccharides, especially glucomannan, in pink wine samples is possible when treated with the enzyme β-glucanase. Such samples also ensure wine stability for 10 months (Table 1). Unlike the study [10] devoted to the effect of various clarification methods and fining agents on turbidity, color intensity, phenol and antioxidant activity, in this study, taking into account the low efficiency of β-glucanase in the treatment of difficult-to-clear white table wine materials, complex preparations of isolated polygalacturonase and fractions enriched with PG, PE enzymes and C_x-enzyme were used. Treatment with pectofoetidine, P10x and G10x at a dose of 0.1–0.2% based on standard activity, cold treatment and filtration with thickening with bentonite and gelatin were considered appropriate (Table 2). Unlike the study [5], which deals with the ultrafiltration method, which is investigated as an innovative method for clarification and stabilization of wine, here a theoretical study of filtration and fining of wines in the ultrasonic field was conducted. It was found that as the sound pressure increases, the settling time decreases sharply. The speed of the coagulation process depends on the intensity of ultrasound. The effects associated with the explosion of the cavitation cavity and its formation depend on thermodynamic (temperature and external pressure), acoustic (frequency and sound pressure), liquid parameters (surface tension, viscosity, density, saturated vapor pressure of the liquid and the solubility of gases in it) factors. The development process of a single cavitation cavity goes through 3 stages. Increasing the efficiency of an ultrasonic filter is possible through the correct use of regulating elements. In order to conduct research on the effect of ultrasound on wine and wine materials, special designs that transmit ultrasound have been developed. The most efficient was a wave transmitter-retainer with low acoustic resistance. As a result of statistical processing of experimental values, mathematical dependencies were obtained (Fig. 2, 3, Table 3). Unlike the study [3], which was related to a comprehensive analysis of the results obtained in the application of membrane technologies in winemaking, in this study, the change in inlet pressure depending on the operating time of the filter for different filter types was graphically depicted, and approximate dependencies for the efficiency of the ceramic filter depending on the operating time with and without the use of ultrasound were obtained. The experimental results on the effect of different power modes of the ultrasonic irradiator on the organoleptic indicators of the wine material, especially on the titratable acidity and pH value, are graphically depicted (Fig. 4–7). Unlike the study [13], which established that different types of bentonite have different side effects on the chemical composition of wine and that the aromatic esters and antioxidant phenols remaining in the composition have a positive effect on the sensory quality of wine, in this study, the tasting of wine clarified in a membrane-ceramic filter was carried out in a traditional and experimental way, that is, with the use of ultrasound, and it was found that the experimental variant was rated higher (Table 4). Unlike the study [16] that investigated the effect of cold maceration on the amount of phenolic compounds and some sensory parameters before fermentation, in this study, the pulp obtained from the Madrasa and Merlot grape varieties was subjected to cold maceration for 2–14 days. Analysis of the compositional parameters before and after cold maceration shows that as a result of maceration, the color intensity, dry matter content, fruit-grape aroma, phenolic compounds, including the amount of monomeric anthocyanins increase. The study of the chromatograms obtained as a result of the analyses performed showed that Mv-3-Glu occupies a dominant place in the juice samples. Thus, the concentration of Mv-3-Glu anthocyanin in the Madrasa juice was 42% before maceration and increased to 44% after maceration. A similar situation was observed in the Merlot variety. In the Madrasa grape variety, anthocyanins formed the following sequence according to the mentioned trait after maceration: Mv-3-Glu > Peo-3-Glu > Dp-3-Glu > Cy-3-Glu (Tables 5, 6).

The results of the study can be applied in the field of winemaking science. The obtained results can be used in scientific research on juices and wines, in family farms and wineries.

The study is not considered acceptable for juices and wines with a high content of coarse and suspended particles. There is a limitation, especially for wines prepared with the participation of solid parts of the bunch, as well as dessert wines.

The lack of the study is the need to clean the juice and wine from coarse particles with other filters before membrane filters. Future studies can be expanded in the direction of studying new generation enzyme preparations and membrane technologies in the clarification of juice and wine samples. It may be consider the study of the effect of methods affecting the intensity of clarification on the change in antioxidant properties of juices and wines to be promising.

7. Conclusion

1. Although the Pektofoetidin P 10× preparation and the PG, PE enzymes, enriched with C_x-enzyme fractions, exhibit sufficiently high hemicellulase activity, their effect is inferior to that of β-glucanase. In rosé wine material, the greatest reduction of polysaccharides, particularly glucomannan, is observed when treated with C_x, being twice as effective as β-glucanase. For white table wine material, it is recommended to use Pektofoetidin П 10× and Г 10× at a dose of 0.01–0.02% based on standard activity, followed by bentonite treatment and gelatin fining combined with cold processing, with filtration carried out at cold temperature.

2. It was determined that ultrasonic treatment of wine materials increases the content of aromatic compounds,

color, and acidity, and improves wine stability. Ultrasonic processing enhances the productivity of membrane filters, intensifying the filtration and clarification processes. Particles of different sizes settle at different frequencies; the higher the frequency, the smaller the particle size. In practice, ultrasound with frequencies ranging from 500 to 20.000 Hz is used. Under these conditions, particles with sizes of 0.5–5 μm settle.

3. The optimal parameters for ultrasound during wine filtration were determined: frequency 22 ± 1.65 kHz, amplitude -20 ± 5 μm. The pressure of the wine material in the system was 0.2 ± 0.01 MPa. Stopping ultrasound in the initial minutes leads to a sharp decrease in productivity, and after 5–8 minutes, filter clogging causes productivity to drop 10–15 times. When ultrasound is resumed, the layer of deposits on the filter is disrupted, restoring normal operation of the filter and the system.

4. Cold maceration of Madrasa and Merlot grape varieties at 7–8°C for 4 days before fermentation allows obtaining juice rich in total anthocyanins and phenolic compounds, with high color intensity. Monomeric anthocyanins, including malvidin-3-glucoside, peonidin-3-glucoside, cyanidin-3-glucoside, and delphinidin-3-glucoside, also increased due to maceration. Juice obtained by cold maceration was enriched with volatile compounds imparting fruity and floral aromas to the wine. In conclusion, wines produced using cold maceration can achieve high anthocyanin content and color stability.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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The study was performed without financial support.

Data availability

Data will be made available on reasonable request.

Use of artificial intelligence tools

The authors confirm that they did not use artificial intelligence technologies in creating the submitted work.

Authors' contributions

Hasil Fataliyev: Writing – review & editing, Project administration, Writing – original draft; **Elnur Heydarov:** Conceptualization, Investigation, Writing – original draft; **Natavan Gadimova:** Validation, Investigation, Writing – review & editing; **Mehman Ismayilov:** Investigation, Methodology, Conceptualization; **Naila Mammadova:** Investigation, Methodology, Conceptualization; **Asaf Rushanov:** Conceptualization, Methodology, Investigation.

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