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Investigation of the Acid Composition of Sherry-Type Wine Materials Produced from Azerbaijani Grape Varieties and Evaluation of Acidity Regulation Methods

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ABSTRACT

This study investigated the acid composition of experimental sherry-type wine materials produced from Azerbaijani grape varieties and evaluated different acidity-regulation methods under controlled processing conditions. Wine materials prepared from the grape varieties Bayan Shira, Aligote, Riesling, Rkatsiteli, and Chardonnay, cultivated in different soil-climatic zones of Azerbaijan, were used as the research object. Titratable acidity, pH, organic acid composition, acetaldehyde, and other physicochemical parameters were determined in the samples. Chemical, physical, and biological methods were comparatively evaluated for the purpose of acidity regulation. The titratable acidity of the investigated wine materials ranged from 5.75 to 9.1 g/dm³, depending on grape variety and growing region. In wine materials produced under warm-climate conditions, titratable acidity could decrease to 4.76 g/dm³, whereas high-acidity materials from the investigated regions showed values of up to 9.1 g/dm³. Adjustment of the acidity to 5.5–6.5 g/dm³ and the pH to 3.2–3.4 accelerated the development of the sherry flor, increased the acetaldehyde content to 526–646 mg/dm³, and ensured the formation of the typical sherry character. During biological deacidification, the conversion of malic acid into lactic acid improved the stability and organoleptic properties of the wine. In wine materials with low acidity, the addition of tartaric acid and the blending method made it possible to achieve an optimal acidity balance. The obtained results demonstrate that the targeted regulation of acidity increases the intensity of the sherrying process in sherry-type wine materials, accelerates flor formation, and improves the quality characteristics of the finished product. These results are of practical significance for optimizing the production technology of sherry-type wines.

1. INTRODUCTION

Sherry-type wines belong to a special group of wines distinguished in the world wine industry by their unique production technology and high organoleptic characteristics. The chemical composition of the wine materials used in the production of these wines, particularly titratable acidity and the pH of the medium, directly affects the development of flor yeasts, flor formation, and the quality of the final product. Deviation of acidity parameters from the optimum range may reduce the intensity of the biological aging process, acetaldehyde accumulation, and the development of the characteristic sherry aroma.

The different soil and climatic conditions of Azerbaijan significantly influence grape biochemical composition. Wine materials obtained from grapes cultivated in mountainous and foothill regions generally exhibit higher titratable acidity, whereas those produced in hot and arid regions are characterized by lower acidity. Consequently, technological adjustment of acidity is an essential step in the production of sherry-type wine materials.

Recent studies have demonstrated that aging technology, alcohol concentration, and production conditions considerably influence the physicochemical composition and sensory properties of sherry-related products, including low-alcohol sherry wines, sweet sherry wines, Brandy de Jerez, and wine spirits [1-4]. In parallel, numerous investigations have evaluated chemical, physical, and biological approaches for wine acidity regulation, including electrodialysis, ion-exchange resins, acidification and deacidification practices, and the application of non-Saccharomyces yeasts and lactic acid bacteria [5-11]. Collectively, these studies demonstrate that wine acidity and pH can be effectively modified using different technological approaches; however, their efficiency

depends on grape variety, climatic conditions, and processing technology [12-19].

Research on flor yeasts has further shown that biological aging substantially affects ethanol metabolism, aroma development, colour, and wine quality, while the diversity of flor-associated microorganisms remains an important factor determining the success of the process [20-24]. Previous studies performed in Azerbaijan also demonstrated that grape variety, cultivation region, processing technology, and environmental conditions significantly influence the physicochemical composition and quality of grapes, juices, wines, and wine materials [25-31].

Despite these advances, most published studies have focused on raw materials produced in Spain and other traditional wine-producing regions. Information regarding the formation and regulation of acidity in sherry-type wine materials produced from Azerbaijani grape varieties grown under contrasting soil and climatic conditions remains limited. Moreover, the combined evaluation of methods for correcting both excessive and insufficient acidity and their effects on flor development, acetaldehyde accumulation, and wine quality has not been sufficiently investigated.

Therefore, the aim of this study was to determine the characteristics of acidity formation in sherry-type wine materials produced from Azerbaijani grape varieties, comparatively evaluate methods for regulating high and low acidity, and investigate the effects of acidity adjustment on flor development, physicochemical properties, and organoleptic quality.

2. MATERIALS AND METHODS

White wine materials produced from the grape varieties Bayanshira, Rkatsiteli, Aligote, Riesling, and Chardonnay (*Vitis vinifera* L.) cultivated in different soil and climatic zones of Azerbaijan were used in this study. Grapes were harvested at the technological maturity stage from vineyards located in the Goygol (mountainous) and Samukh (arid) regions of Azerbaijan. At harvest, the soluble solids content of the grape juice was 22.0–22.1°Brix, indicating technological maturity suitable for white wine production. Each grape variety was harvested separately. Representative composite samples were prepared according to the standard vineyard sampling methodology using diagonal vineyard sampling. Grape clusters were collected at regular intervals from selected vines and from the upper, middle, and lower parts of the canopy, as well as from different canopy orientations, to minimize sampling bias. The collected grape clusters were thoroughly mixed to obtain representative composite samples for each variety. Three independent batches of wine materials were prepared from each grape variety and used for all subsequent analyses. Comparative investigations were performed to evaluate the characteristics of acidity formation and the efficiency of different acidity-regulation methods in sherry-type wine materials (Table 1).

2.1 Acidity regulation treatments

Acidity regulation experiments were performed using the white wine materials obtained from the grape varieties Bayanshira, Rkatsiteli, Aligote, Riesling, and Chardonnay cultivated in the Goygol (mountainous) and Samukh (arid) regions of Azerbaijan. Grapes from each variety were processed separately, and three independent wine-making batches were prepared for each variety (n = 3). Before treatment, the wine materials were screened according to titratable acidity and pH. Wine materials with titratable acidity of approximately 8.0–10.0 g/dm³ and pH 3.0–3.3 were classified as high-acidity materials and selected for deacidification experiments, whereas low-acidity materials were subjected to acidification or blending treatments. Titratable acidity was expressed as g/dm³ of tartaric acid equivalent.

Table 1. Characteristics of grape materials used in the study

Parameter	Description
Species	*Vitis vinifera* L.
Grape varieties	Bayanshira, Rkatsiteli, Aligote, Riesling, Chardonnay
Harvest year	2024
Harvest stage	Technological maturity
Soluble solids of grape juice	22.0–22.1°Brix
Growing regions	Goygol (mountainous) and Samukh (arid), Azerbaijan
Sampling method	Representative composite sampling using diagonal vineyard sampling
Sampling positions	Upper, middle, and lower parts of the canopy; different canopy orientations
Experimental design	Three independent wine-making batches per variety (biological replicates, n = 3); each chemical analysis was performed in triplicate (technical replicates)

For chemical deacidification, potassium carbonate, potassium bicarbonate, and Antosid were evaluated. Potassium carbonate was applied at 1.0, 2.0, and 3.0 g/dm³; potassium bicarbonate was applied at 1.0 and 2.0 g/dm³; and Antosid was applied at 2.0 g/dm³ according to the manufacturer's recommended application rate. Each reagent was added gradually under continuous stirring at 300 rpm and 20–22 °C for 15 min. After treatment, the wine materials were allowed to settle for 24 h and were subsequently racked.

Cold stabilization was used as a physical acidity-regulation treatment. The wine materials were maintained at 0–2 °C for 7–10 days without agitation to promote potassium bitartrate crystallization. After cold stabilization, the wine materials were racked and filtered before further analysis.

Biological deacidification was performed using selected microorganisms. Commercial freeze-dried *Oenococcus oeni* cultures were inoculated at approximately 1×10^6 CFU/mL after confirmation of bacterial viability by plate counting on MRS agar. Malolactic fermentation was conducted at 20–22 °C until the malic acid concentration decreased below 0.1 g/L. Malic acid concentration, pH, titratable acidity, and residual sugars were monitored at three-day intervals. In additional biological treatments, *Saccharomyces cerevisiae* and *Schizosaccharomyces acidodevoratus* were inoculated at an initial concentration of approximately 1×10^6 cells/mL. *Saccharomyces cerevisiae* fermentation was conducted at 20–25 °C under anaerobic conditions, whereas *Schizosaccharomyces acidodevoratus* was cultivated under the same conditions, and acidity reduction was monitored by HPLC analysis of malic acid.

For low-acidity wine materials, crystalline tartaric acid was added at 1.0 and 2.0 g/dm³. The effect of acidification was evaluated by comparing the untreated initial material with the materials after addition of each tartaric acid dose. In a separate

blending experiment, wine materials with different titratable acidity levels were mixed according to the "star" calculation method to obtain the target acidity required for sherry-type wine production.

Untreated wine materials served as controls for the corresponding treatment experiments. Following acidity adjustment, the treated and control wine materials were subjected to biological aging and evaluated for flor development, acetaldehyde accumulation, physicochemical characteristics, and sensory properties. Flor development was assessed after 20, 40, and 60 days using the percentage of the wine surface covered by the flor layer.

The acidity-regulation procedures were applied according to the initial acidity characteristics of the wine materials rather than indiscriminately to all samples. Thus, high-acidity materials were selected for deacidification treatments, whereas low-acidity materials were selected for tartaric acid addition or blending. The regional and varietal distribution of the experimental materials is presented in Table 1.

2.2 Analytical methods

The physicochemical and chemical characteristics of the wine materials and wines were determined using standardized analytical procedures. All analyses were performed in three independent biological replicates, and each analytical determination was carried out in triplicate. The results were expressed as mean ± standard deviation.

The pH values were measured using a calibrated digital pH meter (Mettler Toledo SevenCompact S220, Switzerland) equipped with a combined glass electrode. The instrument was calibrated using standard buffer solutions at pH 4.01 and 7.00 before each series of measurements.

Titratable acidity was determined by potentiometric titration with 0.1 mol/L NaOH and expressed as g/dm³ of tartaric acid equivalents. The measurement was performed according to OIV recommendations. The soluble solids content of the grape must was determined using a digital refractometer (Atago PAL-1, Japan) and expressed as °Brix.

2.2.1 Determination of organic acids by high-performance liquid chromatography

Malic, tartaric, lactic, succinic, citric, and other organic acids were determined by high-performance liquid chromatography (HPLC). Wine samples were centrifuged at 5000 × g for 10 min and filtered through 0.45-µm membrane filters before injection. Chromatographic analyses were performed using an HPLC system (Shimadzu LC-20A, Japan) equipped with a UV detector.

Organic acids were separated on an Aminex HPX-87H analytical column (300 × 7.8 mm, 9 µm; Bio-Rad, USA) maintained at 60–65 °C. The mobile phase consisted of 0.0125 mol/L H_2SO_4 delivered at a flow rate of 0.6 mL/min. The injection volume was 10 µL, and detection was performed at 210 nm. Quantification was carried out using external calibration curves prepared from analytical standards of the corresponding organic acids. Malic acid was monitored throughout the biological deacidification process, and malolactic fermentation was considered complete when the malic acid concentration decreased below 0.1 g/L. The HPLC procedure was based on the OIV method for the determination of organic acids in wines.

2.2.2 Determination of volatile compounds by Gas Chromatography–Mass Spectrometry

Volatile compounds formed during fermentation and biological ageing were analyzed using Gas Chromatography–Mass Spectrometry (GC–MS). Analyses were performed using an Agilent 7890B gas chromatograph coupled to an Agilent 5977A mass selective detector (Agilent Technologies, USA). Separation was carried out on a DB-WAX capillary column (30 m × 0.25 mm internal diameter × 0.25 µm film thickness).

Wine samples were filtered before analysis, and 1 µL of sample was injected in split mode. Helium was used as the carrier gas at a constant flow rate of approximately 1.0 mL/min. The injector temperature was maintained at 250 °C. The oven temperature was initially maintained at 40 °C for 5 min, increased to 120 °C at 5 °C/min, then to 180 °C at 3 °C/min, and finally to 220 °C at 5 °C/min, with a final holding period of 10 min. The mass spectrometer was operated in electron-impact ionization mode at 70 eV. Mass spectra were recorded over the appropriate mass range, and compounds were identified by comparison of their mass spectra with the NIST mass spectral library and, where available, authentic reference standards.

The determination of volatile compounds was performed in accordance with the general principles of OIV gas-chromatographic analysis of wine volatile compounds.

2.2.3 Determination of total phenolic compounds

Total phenolic compounds were determined using the Folin–Ciocalteu spectrophotometric method. Wine samples were appropriately diluted with distilled water. An aliquot of the diluted sample was mixed with Folin–Ciocalteu reagent and sodium carbonate solution. After incubation at room temperature for 2 h, absorbance was measured at 765 nm using a UV–Vis spectrophotometer (Shimadzu UV-1800, Japan). Gallic acid was used as the calibration standard, and the results were expressed as mg gallic acid equivalents per litre (mg GAE/L).

A calibration curve was prepared using a series of gallic acid standard solutions, and the concentration of total phenolic compounds was calculated from the corresponding regression equation. The Folin–Ciocalteu procedure is widely used for the determination of total phenolic compounds in wine and is also included in the OIV analytical compendium.

2.2.4 Determination of total nitrogen

Total nitrogen was determined using the Kjeldahl method. The wine samples were digested with concentrated sulfuric acid in the presence of a catalyst until complete mineralization. After digestion, the resulting ammonium compounds were converted to ammonia under alkaline conditions, distilled, and collected in a boric acid solution. The ammonia was subsequently titrated with standardized hydrochloric acid. Total nitrogen was calculated from the amount of acid consumed and expressed as g/L.

The analysis was carried out using a Kjeldahl digestion and distillation system (Kjeltec 8400, FOSS, Denmark).

2.2.5 Determination of sulfur dioxide

Free and total sulfur dioxide (SO_2) were determined by iodometric titration. For free SO_2, the wine sample was acidified and titrated with standardized iodine solution using starch as the indicator. Total SO_2 was determined after alkaline hydrolysis of the bound sulfur dioxide fractions followed by

acidification and iodometric titration. The results were expressed as mg/L SO_2.

2.2.6 Determination of ethanol

Ethanol concentration was determined by gas chromatography using the same GC system described above. A known concentration of an internal standard was added to the wine samples, and the samples were analyzed by direct injection. Quantification was performed using calibration solutions containing known concentrations of ethanol. The ethanol concentration was expressed as % (v/v).

2.2.7 Determination of residual sugars

Residual sugars were determined using an enzymatic spectrophotometric method based on the determination of glucose and fructose. The wine samples were appropriately diluted with distilled water, and the enzymatic reactions were carried out according to the manufacturer's instructions for the analytical reagent kit. Absorbance was measured using a UV–Vis spectrophotometer (Shimadzu UV-1800, Japan). The concentration of residual sugars was calculated from the calibration procedure and expressed as g/L.

2.3 Assessment of flor development

Flor development during biological ageing was evaluated after 20, 40 and 60 days of ageing. The extent of the flor layer covering the wine surface was visually assessed using a five-point scale from 0 to 4:

0 – No flor: no visible yeast film on the wine surface;

1 – Very weak flor: discontinuous and barely visible film covering less than 25% of the wine surface;

2 – Moderate flor: clearly visible but discontinuous film covering approximately 25–50% of the surface;

3 – Pronounced flor: well-developed film covering approximately 50–75% of the surface;

4 – Intensive flor: continuous or almost continuous flor layer covering more than 75% of the wine surface.

The same scoring criteria were applied to both untreated control samples and acidity-adjusted samples. Flor development was recorded under identical observation conditions at each evaluation time.

2.4 Sensory evaluation

Sensory evaluation was conducted after the specified biological ageing periods using a trained sensory panel. Samples were coded with random three-digit numbers and presented in identical wine glasses to minimize the influence of sample identity and presentation. Sensory assessment was performed in a quiet, odor-free environment under uniform lighting conditions. Water was provided between samples to minimize carry-over effects.

The wines were evaluated for appearance, aroma, taste, mouthfeel and overall sensory quality. Appearance was assessed in terms of colour and clarity; aroma was evaluated for intensity, cleanliness and characteristic wine aroma; taste was assessed according to acidity, balance and persistence; mouthfeel was evaluated in terms of body and overall balance. Overall sensory quality was assessed considering the general harmony of the sample.

Each sensory attribute was evaluated using a structured scoring scale. The scores assigned by the panelists were averaged for each sample and ageing period. The resulting mean sensory scores were used for comparison of the untreated control and acidity-regulated wine samples.

3. RESULTS AND DISCUSSIONS

3.1 Determination of the causes of high and low acidity in wine materials

High titratable acidity was more pronounced in the wine materials obtained from the Goygol and Samukh samples investigated in this study, although Samukh is characterized by a warm and relatively dry climate. This indicates that climatic classification alone does not fully determine the acidity of the resulting wine material; grape variety, harvest conditions, vineyard management, and year-specific environmental conditions may also contribute to acidity formation. Although unfavorable climatic conditions may delay grape ripening in some years, the grapes used in the present study were harvested at technological maturity, with soluble solids of 22.0–22.1°Brix.

It is known that under conditions of high acidity, the sherrying process either does not proceed or proceeds very weakly. Therefore, special attention was paid to the regulation of acidity through careful monitoring.

Table 2. Initial physicochemical characteristics of high-acidity base wine materials prior to acidity regulation

Composition Parameters	Wine Materials		
	Bayanshira	Riesling	Chardonnay
Alcohol content, % (v/v)	10.4	10.6	11.0
Sugar, %	0.11	0.09	0.10
Titratable acidity, g/dm³	8.2	8.7	9.1
pH	2.67	2.55	2.65
Volatile acidity, g/dm³	0.37	0.31	0.42
Total sulfur dioxide, mg/dm³	98	106	71
Free sulfur dioxide, mg/dm³	9.0	8.7	6
Total phenolic compounds, g/dm³	0.31	0.35	0.25
Total nitrogen, mg/dm³	2.0	2.02	2.3
Amino nitrogen, mg/dm³	76	108	116

The wine materials presented in Table 2 represent high-acidity base wines selected prior to acidity regulation. The samples were obtained from Goygol from the 2024 harvest and were analyzed at the same pre-treatment stage. Three independent winemaking batches were prepared for each grape variety.

The studies show that the acidity of wines produced in most regions of our country varies within the permissible range for a number of wine types. However, sherry-type wines, which are not produced in our country, have specific characteristics in this respect. Therefore, for this type of wine, preliminary regulation of the acid composition of the wine materials is required. This remains particularly relevant for vineyards cultivated in our foothill and mountainous regions. The results of the investigations are presented in Table 2.

The contents of titratable acids and their main components,

namely tartaric acid and malic acid, were investigated in wine materials obtained from grape varieties cultivated in the mountainous Goygol district (Table 3).

Table 3. Acid composition of base wine materials from the Goygol district intended for sherry-type production

Wine Samples	Acids, g/dm³		
	Titratable	Tartaric	Malic
Aligote (control)	6.30	4.75	4.35
Bayanshira	5.75	4.60	4.15
Riesling	7.35	5.25	4.65
Chardonnay	7.40	5.70	5.00
Rkatsiteli	6.70	4.75	4.30

Note: Values represent the midpoint of the reported minimum–maximum range, calculated as (minimum + maximum)/2.

As shown in Table 4 (Samukh district), the calculated midpoint values for titratable acidity varied among the grape varieties, ranging from 7.55 to 8.65 g/dm³. The highest value was observed for Chardonnay (8.65 g/dm³), followed by Aligote (8.20 g/dm³), Riesling (8.05 g/dm³), Rkatsiteli (7.80 g/dm³), and Bayanshira (7.55 g/dm³). These values indicate relatively high acidity levels in the wine materials, which may require appropriate acidity regulation before their use in sherry-type wine production.

Table 4. Acid composition of base wine materials from the Samukh district intended for sherry-type production

Wine Samples	Acids, g/dm³		
	Titratable	Tartaric	Malic
Aligote (control)	8.20	5.70	4.85
Bayanshira	7.55	5.25	4.90
Riesling	8.05	5.55	4.95
Chardonnay	8.65	5.80	4.70
Rkatsiteli	7.80	5.50	4.40

Note: Values represent the midpoint of the reported minimum–maximum range, calculated as (minimum + maximum)/2.

Studies show that rapid changes in the acid–sugar balance occur at the beginning of berry ripening. Tartaric and malic acids account for 69–92% of the total acidity in grape berries. Unlike many other organic acids, the metabolic origin of tartaric acid is explained by the oxidative metabolism of sugars. The malic acid accumulated in the pulp reaches its maximum level by the beginning of ripening at the end of the first growth phase. Photosynthesis in leaves and green berries is responsible for 50% of acid accumulation. During ripening, the decrease in malic acid concentration is associated with the intensive oxidation of malates. At this stage, malic acid is used as an energy source for respiration. While a cool climate stimulates the formation of malic acid, a warm climate, on the contrary, has a suppressive effect.

In addition to the acids mentioned above, wine also contains citric, succinic, lactic, and acetic acids, which are formed during the fermentation process.

3.2 Study of methods for reducing high acidity

Both physicochemical and biological methods are used to reduce the acidity of grape juices and wines.

Chemical methods for reducing acidity include chalk treatment, precipitation of double salts of tartaric and malic acids, and treatment with chitin-containing preparations, while physicochemical methods include ion exchange, electrodialysis, and cold treatment.

Commercial preparations produced abroad (Russia, Germany, France, and Italy) are used for chemical deacidification. Acidity reduction can be carried out using calcium or potassium carbonate salts or potassium hydrogen carbonate (bicarbonate).

Highly purified food-grade chalk, washed several times with water, is often used to reduce the acidity of grape juice. It is recommended that the chalk be added to the juice in small portions with continuous stirring. The juice should be clarified in advance and sulfited to a total sulfur dioxide content of up to 100 mg/dm³. After 12–20 hours, the juice is separated from the sediment.

It has been established that the use of potassium bicarbonate provides better results for acidity reduction. In this case, the wine becomes softer, its taste becomes more harmonious, and it is more resistant to haze formation associated with excess calcium.

After the wine has been separated from the sediment, sulfited, and clarified, it is treated. If acidity is to be reduced biologically, sulfiting should be carried out at such a dose that the free sulfur dioxide content does not exceed 10 mg/dm³. This method also has a disadvantage. It is known that the dissociation constant of tartaric acid is higher than that of malic acid. Therefore, during such treatment, malic acid remains in solution.

Studies show that when a substantial reduction in acidity is required, this operation should begin with the juice, whereas when only a slight adjustment of acidity is needed, it should be carried out in the wine.

Biological deacidification is an effective approach for reducing titratable acidity; however, its efficiency is limited in wine materials with excessively high acidity. Therefore, highly acidic materials may require preliminary chemical or physicochemical adjustment to create conditions suitable for the subsequent development of malolactic bacteria. Therefore, such wine materials should first be treated chemically and then biologically. In this case, the chemical method reduces the acidity to a level at which microorganisms can actively develop and complete the process.

It has been established that the optimum pH for the development of the sherry flor ranges from 3.2 to 3.4. In such white wine materials, a titratable acidity of 5–7 g/dm³ can be considered optimal.

If the wine material has a high pH and low titratable acidity, it may be blended with wine material having higher acidity or acidified with crystalline tartaric acid or citric acid.

Control of the chemical deacidification process is carried out according to several parameters. These include titratable acidity, pH, and tartaric acid content. The tartaric acid content should not be less than 1 g/dm³, and the calcium content should not exceed 100 mg/dm³.

The results of studies on acidity reduction by cold treatment and chemical methods are presented in Table 5.

Cold treatment also causes a reduction in the concentration of organic acids, including tartaric acid. However, this reduction is not particularly pronounced. As shown in Table 5, the greatest decrease in potassium concentration was observed after cold treatment, whereas the Antosid preparation mainly promoted the reduction of tartaric acid. Cold stabilization enhanced potassium bitartrate precipitation, resulting in the most pronounced decrease in potassium content among the investigated treatments. Increasing the dosage of potassium bicarbonate disrupts the balance between anions and cations

and, due to the excess concentration of potassium cations, creates favorable conditions for crystalline haze formation. However, subsequent cold treatment of these samples ensures a reduction in the concentrations of both cations and anions. Nevertheless, because the optimal anion-to-cation ratio is not always achieved, the wine material may remain unstable against crystalline haze.

Table 5. Effect of chemical and cold treatment methods on the contents of cations and organic acids

Mass Concentration	Experimental Variants				
	Control (Untreated)	Cold Treatment	Potassium Carbonate	Potassium Bicarbonate	Antosid
Cations, mg/dm³					
Potassium	970	680	940	1020	930
Calcium	116	97	168	98	106
Organic acids, g/dm³					
Tartaric acid	4.1	3.2	1.2	1.9	0.8
Malic acid	5.2	4.6	2.9	4.1	2.6

When the new Antosid preparation was used, the tartaric acid concentration decreased considerably below the required standard, making this treatment unacceptable. When the Antosid preparation was used, the tartaric acid concentration decreased below the desired level. Therefore, the use of this treatment should be carefully controlled to avoid excessive tartaric acid removal.

The results indicate that each deacidification treatment affected the ionic composition of the wine material differently. Cold stabilization produced the greatest decrease in potassium concentration due to potassium bitartrate precipitation, whereas chemical deacidification treatments showed different efficiencies depending on the reagent used. Therefore, selection of the deacidification method should consider not only acidity reduction but also its influence on wine stability and overall composition.

Biological deacidification attracts greater attention from an environmental point of view. In this approach, there is no need to add external chemical substances to the juice or wine material, since the process is carried out by lactic acid bacteria that convert malic acid into lactic acid, thereby reducing acidity. It has been established that this process is accompanied by improved wine quality and enhanced stability. At the same time, the partial elimination of malic acid reduces the possibility of bacterial haze. It is well known that the influence of organic acids on wine quality is not limited to their effect on taste and stability but also extends to oxidation–reduction (OR) processes occurring during wine formation and maturation. In other words, they influence the OR potential of the wine.

Several factors inhibit the development of lactic acid bacteria in wine, and these should be considered when regulating the process. Otherwise, successful results cannot be guaranteed. The optimum temperature for malolactic fermentation ranges between 20 and 25 °C. At temperatures above 30 °C, the process ceases. Sulfur dioxide exerts an inhibitory effect on the growth of deacidifying bacteria. An ethanol concentration exceeding 14–15% (v/v) markedly suppresses their metabolism. The critical pH value is approximately 2.9; values below this inhibit bacterial growth. A limiting factor for malolactic fermentation is the high concentration of phenolic compounds, particularly when it exceeds 500 mg/dm³. High concentrations of tartaric acid may inhibit the activity of malolactic bacteria, particularly when combined with low pH, high ethanol concentration, and elevated SO_2. Therefore, tartaric acid concentration should be considered together with the overall physicochemical conditions of the wine rather than as an independent absolute threshold. In white wines, the ratio of tartaric acid to malic acid generally favors tartaric acid or remains approximately equal. It has been established that a 2:1 ratio is considered optimal.

Observations indicate that wine undergoing malolactic fermentation exhibits slight flocculation, and when shaken, the bacterial biomass forms silky waves. During sensory evaluation, the wine gives the impression of being saturated with carbonic acid.

Pure cultures of malolactic bacteria can develop under mildly acidic conditions, with optimal activity generally occurring around pH 3.0–3.3; growth becomes increasingly restricted as the pH decreases below this range.

Stopping the process at the appropriate stage is also an important issue. Otherwise, the acidity may decrease below the desired level. One of the most attractive and widely used methods for terminating malolactic fermentation is the addition of sulfur dioxide at 159–180 mg/dm³. However, this method also has disadvantages, since the relatively high sulfur dioxide concentration may subsequently inhibit the development of flor yeasts during sherry production.

One of the more favorable approaches is short-term heat treatment prior to flor aging, namely pasteurization. The addition of a small amount of sulfur dioxide (25–30 mg/dm³), followed by fining and filtration or pasteurization of the wine material, has produced satisfactory results. As dibasic malic acid is converted into monobasic lactic acid, the sharp acidity of the medium decreases, while the lactic acid accumulated in the wine imparts a characteristic soft taste. This process is catalyzed by the enzymes malate dehydrogenase, decarboxylase, and lactate dehydrogenase.

Acidity can also be reduced by the natural microflora present on grapes. Although the bacterial population on grape berries is very low at the time of harvest, a considerably higher number of bacteria can be counted and isolated after the grapes are crushed and transferred to fermentation vessels. During the early stages of grape ripening, bacterial populations vary between 10^2 and 10^4 CFU/mL, depending on environmental conditions. A close relationship exists between pH and bacterial populations, with higher pH values promoting greater populations of lactic acid bacteria. Four principal genera of lactic acid bacteria have been identified in grape juice: *Lactobacillus*, *Pediococcus*, *Leuconostoc*, and *Oenococcus*.

During the first days of alcoholic fermentation, the population of lactic acid bacteria increases to approximately 10^4 CFU/mL and subsequently declines to about 10^2 CFU/mL by the end of fermentation. The highest bacterial population is observed when the ethanol concentration reaches 5–6% (v/v). During alcoholic fermentation, not only does the total bacterial population decrease, but species diversity also declines. By the end of alcoholic fermentation, *Oenococcus oeni* becomes the

dominant species. Studies based on colony hybridization have shown that *Lactobacillus* species, together with *Pediococcus* and *Leuconostoc mesenteroides*, gradually disappear or decrease to very low concentrations. Consequently, the biological malolactic fermentation occurring during the production of sherry wine materials proceeds as illustrated in Figure 1.

A total of four genera and nine species of lactic acid bacteria have been identified in wine. However, allowing the process to proceed spontaneously by means of the natural microflora is associated with several risks, which must be taken into consideration during production.

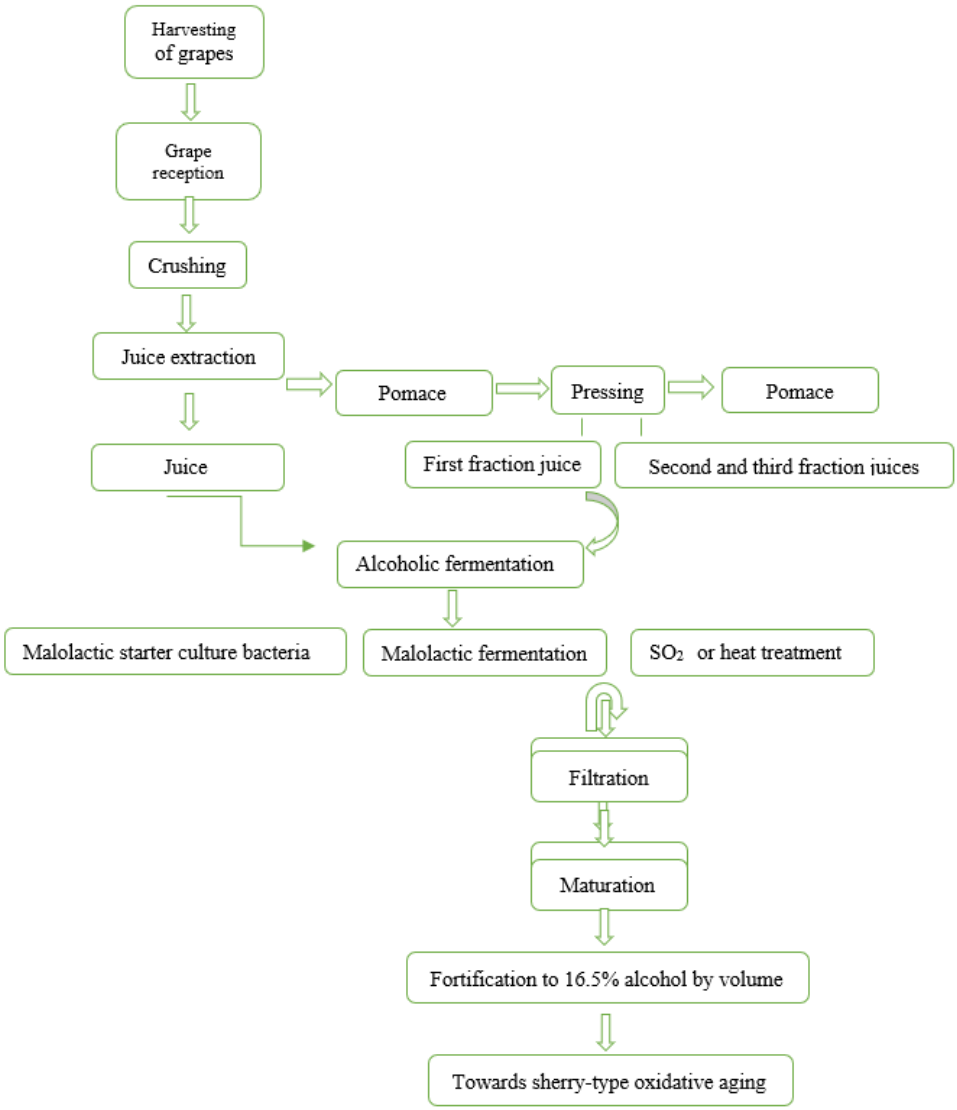

Figure 1. Technological scheme for the production of sherry wine material with acidity reduction

It has been established that, during the natural deacidification process in the production of sherry wine materials, *Oenococcus oeni* is the only species that consistently survives until the end of fermentation. Nevertheless, several other species have also been observed to remain viable. During fermentation, the growth of *Oenococcus oeni* is promoted by the gradually increasing ethanol concentration and other products of yeast metabolism. Fatty acids have been reported to strongly inhibit the development of lactic acid bacteria. In addition, some yeast species produce relatively high concentrations of sulfur dioxide as a result of sulfur metabolism. During periods of vigorous yeast growth, the increased toxicity of the medium is accompanied by temporary nitrogen deficiency. However, at the end of alcoholic fermentation, yeast autolysis releases sufficient amino acids into the medium. Among the lactic acid bacteria, *Oenococcus oeni* is the species that is best able to overcome these adverse conditions. Although certain *Pediococcus* and *Lactobacillus* species may also survive, they have been reported to cause various wine spoilage defects. It has also been established that the natural selection of lactic acid bacteria is influenced not only by their interactions with yeasts but also by interactions among the bacterial species themselves.

At the end of alcoholic fermentation, when the bacterial population reaches approximately 10^6 CFU/mL, malolactic fermentation begins. The most important factors affecting bacterial growth are pH, temperature, and ethanol concentration. Bacterial development is favored at high pH values (pH > 3.5), ethanol concentrations below 13% (v/v), and temperatures of 19–20 °C. In contrast, bacterial growth becomes virtually impossible at pH values below 3.0, ethanol concentrations above 14% (v/v), and temperatures below 17 °C.

The investigations demonstrated that, depending on the acid concentration and environmental conditions, the time required

for the completion of malolactic fermentation may vary from 5 days to 2–3 weeks. Once malic acid has been completely converted into lactic acid by lactic acid bacteria, the wine is sulfited to achieve microbiological stability. The majority of bacteria and the remaining yeasts are sensitive to sulfur dioxide. The effectiveness of sulfur dioxide is closely related to pH. In wines with high pH, the antimicrobial effect of SO_2 is reduced, allowing bacterial survival. Under such conditions, viable cell populations may still reach 10^5–10^6 CFU/mL even several months after production. Under these circumstances, *Lactobacillus* and *Pediococcus* species are more resistant and are responsible for wine spoilage. Therefore, only physical treatments such as heat treatment (pasteurization) or filtration can completely eliminate viable bacterial cells.

Table 6a. Changes in acidity and microbial cell counts under the influence of bacteria in the absence of yeast

Duration of Microorganism Cultivation Across Variants (days)	Bayanshira					Riesling				
	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Initial Titratable Acidity (g/dm³)	Titratable Acidity Decrease (%)	pH	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Initial Content of Titratable Acidity (g/dm³)	Reduction of Titratable Acidity (%)	pH
Initial wine material	0	3.3	8.7	0	3.20	0	3.3	9.3	0	3.15
0										
6	0	8.2	8.1	6.9	3.22	0	10.0	8.7	6.4	3.21
12	0	12.5	7.9	9.2	3.26	0	12.0	8.5	8.6	3.22

Table 6b. Changes in acidity and microbial cell counts under the influence of *Saccharomyces vini* at 0.5% sugar

Duration of Microorganism Cultivation Across Variants (days)	Bayanshira					Riesling				
	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Titratable Acidity (g/dm³)	Titratable Acidity Decrease (%)	pH	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Titratable Acidity (g/dm³)	Reduction of Titratable Acidity (%)	pH
Initial wine material (*Saccharomyces vini*) 0.5% sugar	2.1	3.3	8.7	0	3.20	2.1	3.3	9.3	0	3.15
6	71.4	42.3	7.6	12.6	3.33	62.1	36.4	8.4	9.7	3.22
12	87.8	62.1	6.6	24.1	3.44	91.4	56.5	7.5	19.4	3.35

Table 6c. Changes in acidity and microbial cell counts under the influence of *Schizosaccharomyces acidodevoratus* at 0.5% sugar

Duration of Microorganism Cultivation Across Variants (days)	Bayanshira					Riesling				
	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Titratable Acidity (g/dm³)	Titratable Acidity Decrease (%)	pH	Yeast Cell Count (10^6 cells/mL)	Bacterial Cell Count (10^6 cells/mL)	Titratable Acidity (g/dm³)	Reduction of Titratable Acidity (%)	pH
Initial wine material (*Schizosaccharomyces acidodevoratus*) 0.5% sugar	2.1	3.3	8.7	0.0	3.20	2.1	3.3	9.3	0.0	3.15
0										
6	55.6	45.9	5.6	35.6	3.58	70.4	48.0	7.2	22.6	3.35
12	70.9	56.1	5.5	36.8	3.60	88.7	60.0	6.0	35.5	3.39

Note: Reduction of titratable acidity (%) was calculated relative to the initial titratable acidity using the following equation: reduction (%) = [(initial titratable acidity − titratable acidity at the corresponding time point) / initial titratable acidity] × 100.

The use of a selected *Oenococcus oeni* strain provides rapid and complete malolactic fermentation. Wine materials produced using this strain are characterized by excellent aroma purity, an intense fruity character, and low concentrations of biogenic amines.

The optimum conditions for the selected strain are an ethanol concentration of 14% (v/v) and pH 3.3.

In the absence of yeast, bacterial multiplication proceeds slowly, and by the 12th day the bacterial population increases only threefold. In all experimental variants, both active acidity (pH) and titratable acidity changed markedly (Table 6(a-c)).

In the LAB-only variant (Table 6(a)), titratable acidity decreased by 0.8 g/dm³ in wines produced from the Bayanshira and Riesling grape varieties.

As can be seen, the number of bacteria in wine material samples without yeast cells was several times lower than that in samples inoculated with yeast. Consequently, the reduction in acidity was less pronounced than in the yeast-containing variants. By enriching the medium with nutrients, yeasts exert a stimulatory effect on bacterial growth, thereby promoting more intensive bacterial development.

It is well known that *Saccharomyces vini* yeasts multiply more rapidly than *Schizosaccharomyces acidodevoratus* yeasts. However, the reduction in titratable acidity and the

increase in pH were more pronounced in the latter case, i.e., when *Schizosaccharomyces acidodevoratus* was added to the medium.

3.3 Determination of methods for ıncreasing low acidity

In Azerbaijan, particularly in vineyards located in the lowland regions, grape juices are often characterized by low acidity. Wines produced from such grapes are generally weak, lack harmony, and are more susceptible to microbial spoilage. This issue is especially critical in the production of sherry wine materials. All available academic studies in this field have demonstrated that acidity below the minimum acceptable level is unsuitable for sherry production.

Low acidity can also be prevented by harvesting grapes at an earlier stage of ripening. However, premature harvesting results in wines exhibiting the sharp acidity known as "green acidity," which arises from the high concentration of free organic acids.

The most widely used method for increasing acidity, both experimentally and in commercial production, is the addition of citric acid or tartaric acid to the juice or wine. Because of certain undesirable effects associated with citric acid, tartaric acid was preferred in the present study.

The addition of acid not only improves the taste and harmony of wines with low acidity but also enhances their color, since organic acids promote the dissolution and extraction of grape pigments.

Observations conducted in the warm regions of Azerbaijan indicate that titratable acidity is frequently below the permissible standard (Table 7).

Table 7. Titratable acidity of wine materials produced from grapes cultivated in warm regions

Parameters	Acidified Initial Wine Material	After Addition of Tartaric Acid	
		1 g	2 g
Titratable acidity (g/dm³)	4.76	5.65	6.15
Tartaric acid (g/dm³)	2.60	2.64	2.64
Reduced extract	25.4	26.3	27.3

As can be seen from the table, the addition of tartaric acid resulted in an increase in titratable acidity. As shown in Table 7, the addition of tartaric acid increased the titratable acidity of the wine materials from 4.76 to 5.65 and 6.15 g/dm³ after the addition of 1 and 2 g/dm³ of tartaric acid, respectively. However, the measured soluble tartaric acid showed only a small net increase, from 2.60 to 2.64 g/dm³. This phenomenon may be attributed to the partial precipitation of added tartaric acid as potassium hydrogen tartrate through its interaction with naturally occurring potassium ions in the wine. Consequently, only part of the added tartaric acid remained in the soluble phase, whereas the overall titratable acidity increased.

During the study, blending (coupage) was also employed. The blending calculations were performed using the "star" method. Two wine materials with different acidity levels were available. Analytical results showed that one had a titratable acidity of 8.5 g/dm³, while the other had a titratable acidity of 5.5 g/dm³. These wine materials were blended to obtain a final wine with a titratable acidity of 6.5 g/dm³. The proportions of the two components were determined according to the following procedure.

3.4 Determination of the effect of acidity adjustment on compositional parameters and flor formation

For the flor-aging experiment, the experimental wine materials were subjected to sequential acidity adjustment: preliminary chemical deacidification was followed by biological deacidification using *Oenococcus oeni*. The initial wine materials were used as untreated controls (Table 8).

As shown in Table 8, acidity regulation influenced the rate and characteristics of flor development during biological aging. After 60 days, both the control and treated Bayanshira wines exhibited complete flor formation; however, the treated samples developed a rougher pellicle accompanied by sediment formation. Similarly, the treated white natural wine showed complete flor development with sediment, whereas Rkatsiteli reached approximately 90% pellicle coverage. Acetaldehyde accumulation also differed among grape varieties. Bayanshira (526 vs. 265 mg/dm³) and the white natural wine (560 vs. 290 mg/dm³) showed approximately twofold higher acetaldehyde concentrations than their corresponding controls, whereas Rkatsiteli showed only a moderate increase (646 vs. 564 mg/dm³). These results indicate that the response of flor formation and acetaldehyde production to acidity regulation was dependent on grape variety. Pellicle coverage was estimated as the percentage of the wine surface covered by the flor layer using a semi-quantitative visual scoring approach. The observations were performed under identical illumination and vessel conditions. Pellicle development was classified into five categories: no pellicle formation (0%), weak formation (1–25%), moderate formation (26–50%), good formation (51–75%), and complete formation (76–100%). At the end of the aging period, the continuity, compactness, and surface characteristics of the flor layer were additionally recorded together with acetaldehyde concentration and sensory properties.

High acidity in the control samples also exerted an inhibitory effect on flor formation. In almost all control samples, complete development of the flor layer was not observed within 20 or 40 days.

Table 8. Effect of wine material acidity on flor formation and wine quality

Parametrs	Experimental Variants by Grape Varieties					
	Bayanshire		Rkatsiteli		White Natural Wine	
	Initial Wine Material	Experimental Wine Material	Initial Wine Material	Experimental Wine Material	Initial Wine Material	Experimental Wine Material
Titratable acidity (g/dm³)	8.2	5.7	8.6	5.5	8.8	5.6
pH	2.67	3.4	2.75	3.20	2.75	3.20
Pellicle formation after 20 days (%)	No development	85% pellicle formation	No development	70% pellicle formation	No pellicle formation	75 % pellicle formation

Pellicle formation after 40 days (%)	25% pellicle formation	100% pellicle formation	25% pellicle formation	Full pellicle formation	20% pellicle formation	90% pellicle formation
Pellicle formation after 60 days (%)	Full pellicle formation	Rough pellicle with sediment	Full pellicle formation	Rough sediment begins to form	90% pellicle formation	Complete, rough
Quality indicators after 60 days Acetaldehyde (mg/dm³)	265	526	564	646	290	560
Organoleptic properties	Slight sherry note	Typical strong sherry note	Typical sherry note	Pronounced typical sherry note	Very slight sherry note	Pronounced typical sherry note

Note: Sensory descriptors represent the consensus outcome of the 17-member trained sensory panel following independent assessment and subsequent discussion under standardized tasting conditions.

Table 9. Effect of acidity adjustment on the compositional parameters of sherry wine material

Wine Samples	Treatment Stage	Alcohol (% (v/v))	Sugar (g/100 mL)	SO_2 (mg/dm³)		pH	Titratable Acidity (g/dm³)	Organic Acids (g/dm³)			
				Total	Free			Tartaric Acid	Malic Acid	Lactic Acid	Succinic Acid
Aligote	Before deacidification	9.8	0.11	91	13	3.18	8.7	4.15	3.11	0.41	0.26
Aligote	After deacidification	9.7	0.11	16	9	3.40	6.3	4.15	0.81	1.0	0.26
White natural wine material	Before deacidification	10.2	0.17	96	14	3.18	8.5	4.10	3.80	0.23	0.36
White natural wine material	After deacidification	10.1	0.17	54	8	3.30	6.5	4.10	0.63	2.10	0.36
Bayanshira	Before deacidification	10.5	0.12	64	6.0	3.10	8.6	3.76	3.5	0.36	1.10
Bayanshira	After deacidification	10.3	0.11	58	5.1	3.30	6.4	3.76	0.51	1.20	1.10

During biological deacidification, considerable changes were observed in sulfur dioxide and organic acid composition (Table 9). Total sulfur dioxide decreased markedly in all wine materials, particularly in Aligote (91 to 16 mg/dm³) and white natural wine material (96 to 54 mg/dm³). This reduction decreases the inhibitory effect of sulfur dioxide on microorganisms and creates more favorable conditions for the development of flor yeasts during biological aging. At the same time, malic acid concentrations decreased, whereas lactic acid concentrations increased, confirming the progress of malolactic fermentation. The conversion of malic acid into lactic acid reduced the sharp acidity of the wine materials, increased pH, and contributed to a softer taste. These changes created more suitable conditions for flor development and improved the technological suitability of the wine materials for sherry production.

As can be seen, during the biological reduction of acidity, an increase in lactic acid was observed against the background of a decrease in malic acid in Bayanshira, Aligote, and white table wine materials. During the malolactic fermentation process, no significant changes were observed in the concentrations of tartaric acid and succinic acid.

4. CONCLUSIONS

1. The results of the present study indicate that titratable acidity and pH are important factors influencing the suitability of wine materials for sherry production. The investigated wine materials showed titratable acidity values ranging from 4.76 to 9.1 g/dm³, depending on grape variety, growing region, and technological conditions. The highest values were observed in the high-acidity base wine materials, whereas the lowest value was recorded in the warm-climate wine material presented. Tartaric and malic acids represented the major proportion of total acidity. These findings suggest that climatic conditions, grape maturity, and grape variety influence acidity formation and should be considered during the preparation of wine materials for sherry production.

2. The investigated chemical, physicochemical, and biological methods differed in their ability to regulate excessive acidity. Cold treatment reduced tartaric acid concentration, whereas the Antosid preparation caused a greater decrease in tartaric acid than the other chemical treatments. Under the conditions presented, biological deacidification reduced titratable acidity to 6.3–6.5 g/dm³ and increased pH to 3.30–3.40. These findings suggest that combining chemical and biological deacidification may provide an effective approach for acidity regulation in sherry wine materials.

3. The study demonstrated that wine materials produced from grape varieties cultivated under warm climatic conditions had a titratable acidity of 4.76 g/dm³, which was below the minimum level required for sherry wine production. The addition of 1 g/dm³ of tartaric acid increased the titratable acidity to 5.65 g/dm³, while the addition of 2 g/dm³ increased it to 6.15 g/dm³. At the same time, the reduced extract increased from 25.4 to 27.3 g/dm³. Blending wine materials with different acidity levels was also evaluated as a practical approach for acidity adjustment. According to the "star" calculation method, blending wine materials with titratable acidity values of 8.5 and 5.5 g/dm³ at a ratio of 1:2 resulted in a calculated final titratable acidity of 6.5 g/dm³. This calculation demonstrates that blending can serve as a practical technological tool for adjusting the acidity of wine materials intended for sherry production.

4. Acidity adjustment to approximately 5.5–6.5 g/dm³ and pH 3.20–3.40 was associated with improved flor development, higher acetaldehyde accumulation, and the development of characteristic sherry sensory properties under the experimental

conditions. Biological deacidification was accompanied by the conversion of malic acid into lactic acid and a decrease in sulfur dioxide concentration, creating more favorable conditions for flor yeast development. Overall, the present findings indicate that acidity regulation is an important factor influencing the sherrying process; however, further pilot-scale and industrial studies are required to confirm these results under commercial production conditions.

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