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Prospects of Using Oak Wood Integrated Processing Products in Winemaking, Dealing with Shortcomings on the Base of Implementing Oak Wood Derivative Products

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Abstract

Background/Objectives: Our studies were aimed at exploring the use of oak derivative products for treating and correcting wine flaws and faults, in particular, mousy taint. **Methods/Statistical analysis:** To assess condition and stages of disease development in selected wine materials and to define traditional schemes of their processing, which will be used later for control, we conducted research of their physical, chemical and microbiological criteria. Additionally, the wine materials were tested on their susceptibility to turbidity caused by the elevated content of multivalent metals salts (metal casse) as well as colloidal (reversible and irreversible), crystallic and biochemical turbidity in accordance with generally accepted methods. The authors also tested the wine materials for a fault “mousy taint” with the help of the so called “soda test”. **Findings:** Use of oak derivative products has a positive effect of treating and eliminating mousy taint and improves organoleptic characteristics. Use of oak derivative products for treating faults and correcting mousy taint in dry and fortified wines should be accompanied by preliminary sulfitation and acidification of wine materials to the concentration of titratable acids at least 6.0 g/dm3. To make the processing with oak derivative products even more efficient, it is reasonable to use chips in filtering bags of lavsan or capron fabric, and it is recommended to mix it 2-3 times. **Applications/Improvements:** Obtained results suggest that the proposed method for treating and correcting wine flaws with oak derivative products is efficient and can be implemented into production.

Keywords: Assessment of Oak Primary Resources, Oak Aroma Components, Oak Stave, Oak Wood, Tannin and Tanning Substances, Wine and Cognac Barrels

1. Introduction

Recently, in many countries of the world traditional use of oak wood for making barrels or bouts, goes hand in hand with a rapidly developing new trend of using oak wood derivative products – adding to young wine and cognac spirits oak chips, i.e. fragmented oak wood (micro-staves, chips, micro-chips) both natural and heat treated as well as oak extract[1-5]. Due to large specific surface area and ability to control and adjust oak chips properties (oak type, degree of crushing and toasting), these oak derivative products are effective for optimizing wine aging and its distillates[6].

Heat-treated oak wood gives the drinks a special aroma and flavor. Thermally hydrolyzed lignin, cellulose and hemicelluloses are responsible for this. In France several types of chips are produced: chips and micro-chips. Depending the toasting degree oak chips acquire aroma of a newly cut tree – coconut, hazelnut, spices, toasted bread, almond, coffee, biscuit etc[7-8].

It is also reported about production of wines having diseases and cognacs with faults and flaws. Among them the most common is excessive oxidation of wine or brown casse, sulphurous tone, metal casse etc., passing on to cognac spirits after wine material distillation[9]. The most common wine fault still remains “mousy taint” which is mostly spread among ordinary table and fortified wines, passing on to cognac spirits after faulty wine material distillation[10,11].

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2. Review

"Mousy taint" is double-natured by its origin: physical and chemical changes, and – microbial: lactic acid bacteria (*Bacterium mannitopoeum Muller-Thurgau*, *Osterwaldera* and *Lactobacillus*), vinegar bacteria, and yeast-like fungi *Pichia*, *Brettanomyces*[10-12]. The wines with such diseases are often too oxidized, have low titratable acidity, high concentrations of volatile acids, iron ions which is a catalyst for reductive-oxidative processes (RO)[9]. In the presence of ethyl acetate contained in wine acetamide may appear and it is known to have an after-taste of mice droppings. If wine contains an optimal amount of tannins (red wines) then reaction of oxidative deamination of amino acids is performed only with generating aldehydes without ammonia (amino phenols are formed) and for this reason a mousy taint appears in such wines not that often[10]. Among the main reason for mousy taint appearance are: violation of process discipline and manufacturing sanitation – seeding of mash and wines with microflora, low oxidation and high RO wine potential. Action of active oxygen or aeration, high pH level >3,4 may also account for mousy taint. Mousy taint is also observed under the value of RO potential rH_2 in a range of 20 – 26.

Physical and chemical conditions of mousy taint occurrence depend on the grape harvest year. During the best time of grapes gestation it accumulates sugars, the level of titratable acids is low and pH level is high enough, amino acids such as proline, glycine, ornithine, lysine are also accumulated in grapes; the level of inhibitors (glutamic amino acid and its amide) is decreasing. Insignificant content of glutamic amino acid and phenolic substances contributes to mouse taint occurrence immediately after fermentation.

Studies by Unguryan, Ponomarchenko, Parfientiev, Nemtsev showed that mouse taint occurs as the result of a complex RO reaction under a high value of RO potential (460 – 550 mV). However we should not exclude the possibility of its occurrence during fermentation as a result of vital activities of microorganisms. Adding 1-2% alkali solution into wine enhances the mouse taint and enables to identify it with a rapid method[9]. Traditional techniques to eliminate wine and cognac spirit faults are characterized by their complexity and time-consuming processing, loss of wine materials, energy and resource consumption that increases the prime cost of end products, however positive effect is not always obtained[9,13].

The use of oak wood derivative products for eliminating faults and consequences of wine diseases, particularly mouse taint, has not been studied before. That is why this is a promising direction for technological winemaking processes. The main hypothesis is that enriching the wines with phenolic substances and aroma producing components of oak wood with possible absorption of components refines the wine and eliminates unpleasant tone and after-taste.

The study materials were oak staves and young wine distillates of a grape variety Rkatsiteli produced in manufacturing environment and aged in new and old (depleted) barrels and in reservoirs with storage capacity of 1500 dl and with oak woodblocks and oak wood derivative products.

Also the materials for the study were oak wood derivative products (oak chips) from *English oak* (*Q. robur* L., *Q pedunculata Ehrh.*), *durmast oak* (*Q. petraea* L.), *chestnut-leafed oak* (*Q. castaneifolia* C.A.M), and *Georgian oak* (*Q. iberica* Stev.) with various degree of processing and chopping – woodblock, micro-woodblock, chips, micro-chips, natural and heat-treated.

In studies on eliminating the consequences of mouse taint in wines, the authors used wine materials from the grape variety Rkatsiteli, Sauvignon, and white Port. The studies were repeated three times. Organoleptical method was used for selecting and evaluating faulty wine materials.

Wine materials with faults were processed with oak wood shredded in accordance with "Technological instruction for applying shredded oak wood in production of wines, cognacs (brandies) and strong beverages" KSVP-5-2000. The efficiency of processing was detected by the main physical, chemical and organoleptic criteria. Reference samples were the samples of diseased and faulty wine materials. Additional materials for processing were both national and German, produced by the company "Döhler"

3. Research Methods of Physical, Chemical and Organoleptic Criteria in Wine Materials and Cognac Spirits

- sampling for analysis – GOST 14137–74;
- ethyl alcohol volume ratio – GOST 13191–73;
- mass concentration of reducing sugars – GOST

3192–73;

- mass concentration of titrable acids – GOST 14252–73;
- mass concentration of volatile acids– GOST 13193–73;
- mass concentration of free and total sulfurous acid – GOST 14351–73;
- mass concentration of phenolic substances – GOST 4112.41:2003;
- active acidity (pH) – potentiometer method with pH–meter “Ionomer I–130”;
- iron mass concentartion – GOST 13195–73 and GOST 26928–86;
- organoleptic assessment of wine materials and cognac spirits – method of tasting on a 100 point system[14-17];

The testing of wine materials having diseases and faults was performed in relation to turbidity caused by heavy metals (metal casse), colloidal turbidity (reversible and irreversible), crystallic and biochemical turbidity with generally accepted methods[18,19]. The testing was performed before and after wine material processing.

To identify the presence of mouse taint in wine materials we performed the so-called “soda test”. We added 5-8g of baking soda into a glass of wine (white or red) and stirred. If the wine is sound and fault-free, then it does not have excessive amount of iron, its color and transparency do not change and after releasing CO_2 the wine remains of the same quality. If the wine is sick with mousy taint or have excessive amount of iron, then it gets darker with black sediment and unpleasant flavor of acetamid or mice droppings. Wines with a fault and a positive soda test should not be consumed due to cancerogenic substances and these wines need a special processing[10,20,21].

Minimal concentrations of major aromatic oak components of hydroalcoholic extracts were measured by the method of odorometry – the use of sensation of smell to detect the strength of aromas of vanilla, spices and coconut in minimal concentrations[22].

After tasting, we designed an aromagram of fixed differences, measuring in seconds the length of sensation and aroma strength in major groups of aromatic components of a fresh wood. To compare aromatic balances of the samples, we presented obtained results in percentage terms[17,22-24].

The research was conducted with the methods of multiple-factor experiment design. Reliability of obtained results was supported by conducting the experiment three times. Statistical data processing was performed in Excel.

4. Results and Discussion

The main property of oak wood in winemaking is enhancement of qualitative indicators of wines and cognacs due to their enrichment with aromatic and flavor components. In the result their aroma, flavor, completeness, delicacy and balance are refined[25-29].

The results of our research of technological evaluation of Azerbaijan oak wood derivative products in winemaking showed one more unique ability of oak wood – correction of flaws, faults and alien tones in wines and cognacs.

Our further studies were aimed at exploring the use of oak derivative products for treating and correcting wine flaws and faults, in particular, mousy taint.

To assess condition and stages of disease development (faults) in selected wine materials and to define traditional schemes of their processing, which will be used later for control, we conducted research of their physical, chemical and microbiological criteria (Tables 1-3).

Additionally, the wine materials were tested on their susceptibility to turbidity caused by the elevated content of multivalent metals salts (metal casse) as well as colloidal (reversible and irreversible), crystallic and biochemical turbidity in accordance with generally accepted methods (Table 3). We also tested the wine materials for a fault “mousy taint” with the help of the so called “soda test”.

As we can see from the obtained results (Figure 1), the wine materials under research are characterized by low concentration of titratable acids, sulfurous acid, high level of pH and high concentration of iron ions and volatile acids.

Relying on the review of literature, this complex of physical and chemical indicators of wine materials (in particular, elevated content of iron ions) contributes to acceleration of reductive-oxidative processes and may be a precursor for flaws and faults.

Table 1. Physical and chemical parameters of the wine materials with flaws and faults

Wine material	Alcohol by volume, % об.	Mass concentration							pH
		sugars, g/100 cm^3	Titratable acids, g/ dm^3	Volatile acids, mg/ dm^3	iron, mg/ dm^3	Phenolic substances, mg/dm^3	H_2SO_3, mg/dm^3 free	total	
White Port	17,24	9,4	3,9	0,73	25,4	420	5,0	88,96	3,83
Rkatsiteli	11,6	0,3	3,6	0,62	8,8	190	8,9	124,96	3,77
Sauvignon	11,2	0,2	4,8	0,56	14,1	140	7,36	111,36	3,82

Table 2. Microbiological parameters of wine materials with faults and flaws

Number of wine samples	Number of cells in native material	Amount in 10 cm^2		Groups of microorganisms in 1 cm^3		
		Yeast cells	Potentially patho-genic enterobacteria	CFU[1] of lactic acid bacteria	CFU of yeast cells	CFU of acetic acid bacteria
White Port						
4	0	0	0	0	0	0
Rkatsiteli						
3	0	2	0	+	0	+
Sauvignon						
4	1	12	0	+	0	+

[1]CFU – colony-forming units

Presence in these wine materials the signs of mousy taint is also evidenced by the results of organoleptic test.

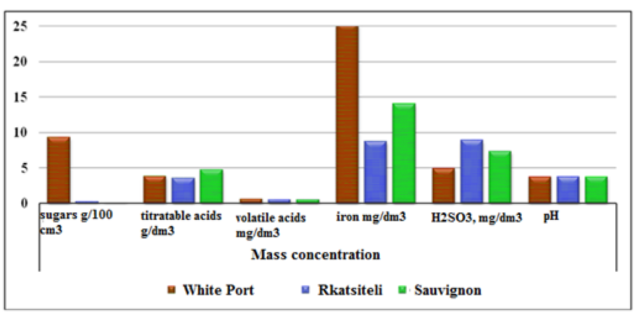

Figure 1. Physical and chemical parameters of the wine materials with flaws and faults.

Tests on wine materials resistance to turbidity (Table 2) proved the tendency of Rkatsiteli and Sauvignon wine materials to albuminous cloudiness. Also we found the presence of metal casse in Sauvignon and white Port wine materials which is supported by the results of physical and chemical research on the exceeding permissible concentration of iron ions in these wine materials – 14.1 and 25.4, respectively under the norm of ≤ 10 mg/dm^3.

The same results were obtained from the “soda test” – after adding 5 g of baking soda into a glass of wine material all the theree samples discoored to black and acquired unpleasant tone of mice droppings. In the taste and aftertaste of white Porto was acetamide tone which is characteristic of the wines with the fault “mousy taint”. Additionally, we found “sugariness” which is typical for low acid wines.

An important aspect for defining the schemes for wine

Table 3. Resistance of wine materials with flaws and faults to various types of turbidity

Test results on tendency to turbidity	Turbidities					
	Caused by metals (metal casse)	Irreversible Colloidal	Reversible Colloidal	Crystallic	Biochemical	Test on “mousy taint” (“soda test”)
White Port	+	-	-	-	-	Wine darkening, clear “mousy taint”
Rkatsiteli	-	+	-	-	-	Wine darkening, “mousy taint”
Sauvignon	+	+	-	-	-	Wine darkening, “mousy taint”

Table 4. Organoleptic evaluation of wine materials with flaws and faults

Evaluation	Wine materials		
	White Port	Rkatsiteli	Sauvignon
Color	**Golden amber**	**Straw**	**Light straw**
Aroma/Bouquet	inharmonious, mousy taint is noticeable	Week aroma, disbalanced	Aroma is not developed enough
Flavor	disbalanced, inharmonious, mousy taint is noticeable	ordinary, blank, inharmonious, disbalanced, mousy taint is slightly noticeable	grassy, mousy taint is slightly noticeable
Tastings, scores	8,1	7,1	7,4

materials processing is their micribiological conditions. As microbiological researches showed (Table 2), the growth of lactic acid and acetic acid bacteria was found in wine materials Rkatsiteli and Sauvignon. Thus, we can make a conclusion that mousy taint in white Port wine materials has physical and chemical nature, supporting low concentration of volatile acids and physical, chemical and organoleptic chanes in Rkatsiteli and Sauvignon wine materials have microbiological nature. So in the first case mousy taint is a flaw and in the second case it is a fault (Table 4).

Summarizing obtained data we developed major schemes for processing wine materials with mousy taint, involving acid concentration increase due to adding citric and tartaric acids (to accelerate acetamide hydrolysis) and sulfitation (for binding the products of hydrolysis), processing with sorbents (bentonite, activated charcoal, bioxsine and herbinol super) with decantation and filtration after the processing[10].

At the same time, it is important to study the process of increasing concentration of titratable acids of wine materials by adding citric and tartaric acids, because the use of the last one is an important factor for forming organoleptic properties of wine materials. Also, to ensure a stable quality of the wines, i.e. to reduce their oxidizing properties, it is necessary to reduce the concentration of iron – which is a catalyst of reductive-oxidative processes – if its content in wine materials is increased up to 10 mg/dm^3 (required for white Port and Sauvignon).

Iron content reduction in white Port and Sauvignon wine materials was performed in laboratory (experimental processing) and only then we performed production processing with the phosphorous cellulose ether[30]. In the result, concentration of iron in these wine materials reduced and was 6.7 and 4.4 mg/dm^3, respectively.

On the other hand, to evaluate the impact of oak wood on eliminating wine flaws and to determine optimal amount of oak chips as well as duration of their interaction with wine materials, we process analyzed wine materials with oak derivative products. For the experiment we chose middle fraction oak chips in amount of 0.5 – 2.0 g/dm^3 in various ratio N:T (Natural : Heat-treated) (5:1 – 1:5). Duration of interaction and contact between wine materials and chips was 40 days and every 7-10 days the wine materials were stirred and the samples were tasted. Criteria for determining the optimal amount of chips and duration of interaction was disappearance of the fault in aroma, bouquet, flavor and better quality of wine materials with possible pleasant tones of wood oak aging (vanilla, cloves, coconut etc.). Further worsening of organoleptic parameters after increasing the chips amount or duration of interaction supported our conclusions about optimal chips amount in wine materials.

Preliminary contact of analyzed wine materials with oak derivative products showed that, in general, shortcomings in these samples were eliminated. Wine materials acquired clear and harmonious bouquet, full, pleasant and complicated flavor with noticeable tones of aging (vanilla, cloves, coconut etc.). Organoleptic parameters showed that the best amount of chips for all wine materials was 1.0 g/dm^3 with the ratio N:T = 1:1 (sample 15) and duration of interaction is 60 days for white Port and 30 days for Rkatsiteli and Sauvignon.

Further studies were aimed at comparing the quality of wine materials with mousy taint using traditional processing methods and a proposed method which is based on using oak wood derivative products.

The main criteria to identify the impact and efficiency of oak derivative products on the quality of wine materials and in particular on treatment and elimination of flaws and faults, were indicators of chemical content – mass concentration of titratable and volatile acids, phenolic substances, iron, pH and organoleptic evaluation.

The results of impact of different types of wine materials processing on their acidity and pH value are shown in Figures 2 and 3.

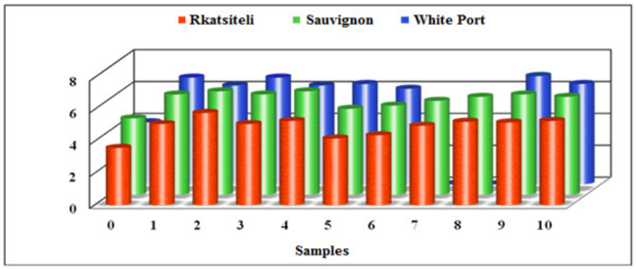

Figure 2. Concentration of titratable acids under various types of processing.

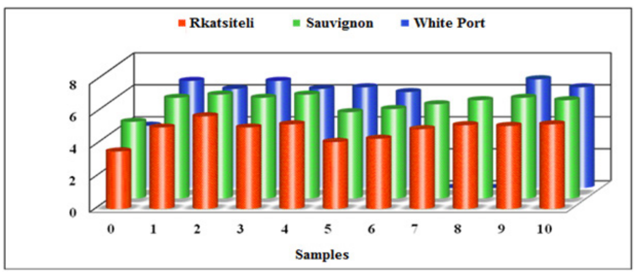

Figure 3. pH value of wine materials under various types of processing.

Unlike reference samples in analyzed samples of wine materials processed with oak chips (with addition of citric and tartaric acids in equal amount), the value of titratable acids was higher than in reference samples undergone different types of processing without using oak chips. It is explained by the fact that chips release oak components, primarily phenolic substances, having acid nature. The same tendency, though to a lower degree, may be observed in all the cases with using oak derivative products[31]. In white Port wine material, interacting with chips for 60 days, concentration of titratable acids was higher that acid increase in wine materials in Rkatsiteli and Sauvignon, interacting with chips for 30 days.

The results of reference sample with mousy taint research showed that mass concentration of volatile acids was in norm and did not exceed 1.2 g/dm^3. However various types of processing had a different impact on volatile acids concentration which is shown on Figure 4.

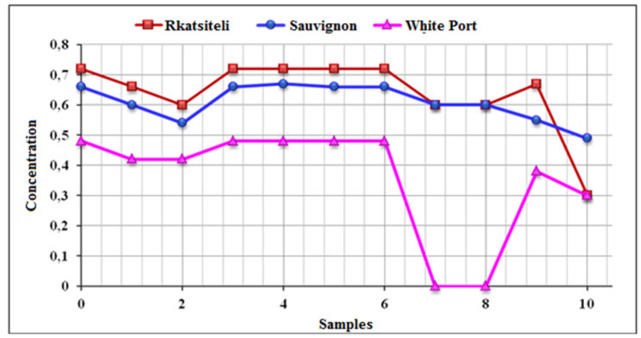

Figure 4. Concentration of volatile acids in wine materials after various types of processing.

In all analyzed samples of wine materials interacting with oak chips, we observed reduction of volatile acids concentration.

Obtained data on the reduction of volatile acids concentration in the samples after processing with chips are indicative of efficiency of using chips as a sorbent agent for components undesirable in wine materials, in particular, for the most common fault "mousy taint" which is supported by the results of organoleptic evaluation (section 4.7.5).

Reduction of iron ions concentration in analyzed samples of white Port and Sauvignon wine materials with mousy taint was reached by preliminary processing with Phosphorous Cellulose Ether (PCE) with further processing in accordance with the accepted schemes[30]. The results of testing the analyzed wine materials on their tendency to turbidity caused by the ions of multivalent metals (metal casse) are given in Table 5.

Table 5. Resistance of wine materials with flaws and faults to turbidity

Turbidity	The results of testing the analyzed wine materials on their tendency to turbidity after processing		
	White Port	Rkatsiteli	Sauvignon
Caused by metals ("metal casse")	-	-	-

Results of the impact of various types of processing of reference and analyzed wine material samples on iron concentration are given in Figure 5.

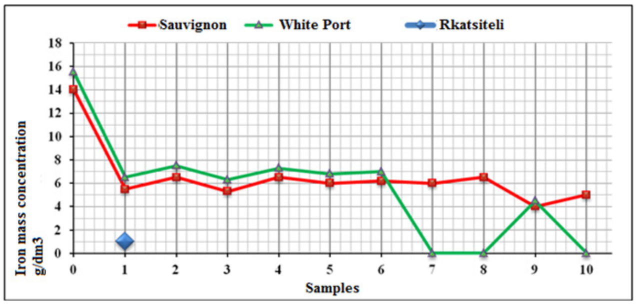

Figure 5. Changes in iron ions concentration in wine materials under different types of processing.

After processing the analyzed wine materials with faults, we observed the common trend in relation to the changes in iron ions concentration (Figure 5).

We have found that tartaric acid added to faulty wine materials for balancing acidity does not change the concentration of titratable acids and iron in wine materials, and citric acid causes insignificant (1.1 – 1.2 mg/dm^3 on average) reduction of iron concentration after all types of processing and in all the samples. The reduction of iron ions concentration (Fe^{3+}) is explained by the complexable capabilities of citric acid under interaction with iron ions.

In analyzed samples of wine materials contacting with oak chips, iron ions concentration was lower than in reference samples. It is explained by the generation of complexes between oak phenolic substances and citric acid during the interaction with iron ions Fe^{3+} with deposition of dark blue sediment under pH = 3 – 5.

Low concentrations of iron ions contributed to the resistance of wine materials to turbidity caused by multivalent metals (metal casse) (Table 5).

We also came to conclusion that a mix of crushed oak wood in chips composition 1:1 = N:T after a special preparation – natural drying and ageing during 3 years and heat treatment – acquires absorption properties. As a result, when interacting with wine materials or cognac spirits with flaws or faults, at first physical and chemical absorption processes take place as well as transformation of undesirable components to their minimum concentration and then occurs enrichment and saturation of wines and spirits with aromatic oak components. At the same time, we can observe insignificant increase of beverage extractivity due to phenolic oak substances, and increase of the content of titratable acids due to a higher concentration of gallus acid. All this helps to correct flaws and faults of wines and cognac spirits.

The results of initial samples of wine materials with "mousy taint", showed that mass concentration of phenolic substances was low: in Rkatsiteli – 190 mg/dm^3, in Sauvignon – 140 mg/dm^3, and in white Port – 420 mg/dm^3.

The impact of various types of wine materials processing on phenolic substances concentration is shown in Figure 6.

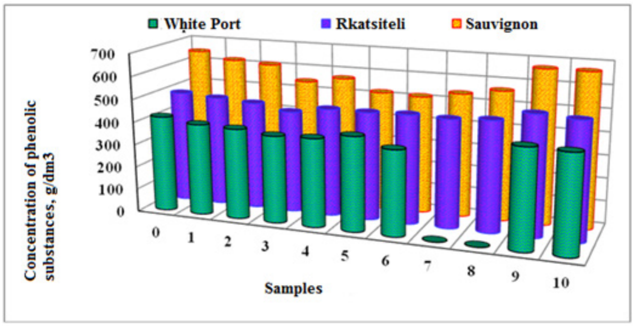

Figure 6. Concentration of phenolic substances in wine materials after different types of processing.

During the interaction between wine material and chips (samples 9 and 10), we observed increase of phenolic substances concentration of 10-30 mg/dm^3, on average. It occurs due to the extraction of tannins form oak wood with their intensive oxidation. These are the tannins that give the wine materials the depth, roundness and softness. Except for tannins, in the process of interaction between the wine materials and oak wood due to ethanolysis, hydrolysis of oak lignin occurs; its products give the wine materials complex tones in the bouquet (vanilla, cloves, coconut and others) and harmony, softness and fullness in taste and after taste.

Results of wine materials tastings processed by different schemes are given in Figure 7.

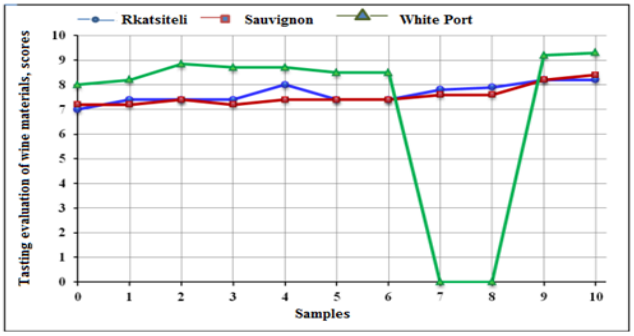

Figure 7. Tasting evaluation of wine materials under different types of processing.

Tasting results of reference and analyzed samples of wine materials (samples 1 – 8 and 9 - 10) are indicative of the following.

Reference samples of white Port under various types of processing were characterized by elimination of mousy taint but they kept their weak, blank, inharmonious aroma and flavor as in the initial wine material. It was common after processing with bentonite and activated charcoal - samples 3, 4, 5 and 6 (tastings evaluation is 8.7 and 8.5). The best scores for organoleptic among the reference samples had the sample 2 (tastings evaluation is 8.9), processed by the scheme MBK → VK → Aeration → Filtration. That sample had better, clear and harmonious flavor and bouquet in comparison with the other reference samples. However, unlike the rest samples that one had a slight mousy taint.

Tasting results of reference and analyzed samples of wine materials (samples 1 – 8 and 9 - 10) are indicative of the following.

Reference samples of white Port (samples 9 and 10) processed with oak chips were characterized by elimination of mousy taint, and due to phenolic substances and other aroma oak components, the wine material was harmonious and balanced with full and soft taste and with tones of ageing (vanilla, cloves, coconut etc. The best scores had the sample 10 (tastings evaluation is 9.3), processed by the scheme MBK → VK → Chips → Filtration. That sample had better balance and harmony of organoleptic parameters.

In reference samples of Rkatsiteli processed by various schemes, after processing with bentonite and Bioxsine, we observed gentle alien perfume and paint and coatings tones (samples 2, 3, 5 and 6 with testing score 7.4). Among the reference samples clean aroma and harmonious flavor was found in samples 7 and 8 (tasting scores are 7.8 and 7.9), processed with Herbinole super. Also, sample 4 had good organoleptic parameters (tasting score is 8.0), processes with bentonite and tartaric acid.

The use of oak derivative products (analyzed samples 9 and 10) significantly improved organoleptic parameters of wine material Rkatsiteli (tasting score is 8.2). These sample acquired pleasant fullness, balance, roundness, harmony and clean aroma and flavor with softly expressed characteristics of the grape variety and tones of ageing in an oak barrel.

Taking into account physical, chemical and organoleptic researches, we may come to the following conclusion: the use of oak derivative products does not only eliminate the flaws in wine materials but also improves their quality, enriching them with oak phenolic substances, reduces volatile acids concentration, iron ions and contributes to a slightly higher concentration of titratable acids. To reach even better results, during the interaction between the wine material and chips, it is advised to perform preliminary sulfitation and add tartaric acid (if necessary).

Novelty of the developed method for treating and eliminating faults and flaws in wines using oak derivative products is confirmed by the invention patent of Azerbaijan No. a 20120063[32].

Concentration of aliphatic spirits in young cognac spirits in compliance with Azerbaijan GOST and Russian Federation GOST R 51145-98 must be in a range of 180 – 600 mg/100cm^3 equivalent to isoamyl alcohol. However at some Azerbaijan enterprises cognac spirits have elevated concentration of superior alcohols in the process of grape wine materials distillation on batch type machines and machines of continuous action violating “Major rules for cognac production”. As a result, in the flavor of a beverage unpleasant fusel tone is noticeable. When exporting these beverages, a consumer has to claim for quality and return the product back to the manufacturer[33].

Traditional methods for decreasing concentration of superior alcohols in young cognac spirits are not always efficient enough. While they may give a positive result, they also worsen the main qualitative indicators of distillate.

We conducted experiments on processing young cognac spirits with elevated concentration of superior spirits in a reservoir with capacity of 1.500 dal with specially prepared oak derivative products.

Young cognac spirits were produced by wine materials distillation from the grapes of variety Rkatsiteli using the machine of continuous action K-5M in manufacturing facilities of Geokchaisk Cognac Factory during the season of processing in 2014.

Oak derivative products were the chips (mixture of natural and het-treated chips in ratio 1:1 and in amount of 8 g/dm^3) – experiment, and staves – reference, obtained from the same batch of an English oak of over 120 years old grown in Ismaillinsky region.

The results of our research showed that superior alcohols transformation and absorption on the oak wood surface (*isoamyl and isobutyl alcohol, n-Propanol*) is 61

– 65%, and methanol is 48.4%. Fusil tone in analyzed alcohols is eliminated in 14 months after their active interaction with oak wood composition in ration 1:1 (heat-treated to natural) and in amount of 8 g/dm^3, under the storage temperature over 16°C and with constant oxygen delivery at least 18-20 mg/dm^3, Table 6.

Organoleptic and odorimetric parameters of wine distillates are indicative of the fact that reduction of aroma and flavor in superior alcohols occurs in a distillate in 3, 6 and, finally, in 14 months after the contact with oak chips (scoring is 91.2). Cognac spirits acquire noble tones of ageing in an oak barrel and do not have a fusil flavor, Table 7.

Table 6. Physical and chemical parameters of distillates with elevated content of superior alcohols after their contact with chips and staves

Parameter MC –mass concentration of components	Measuring Unit	MC of analyzed substances in cognac spirits (GOST R 51145-98)						
		Experiment: Oak chips					Reference: Reservoir with staves	
		Initial	3 months	6 months	14 months	%	Staves 6 months	Staves 14 months
Volume Ratio C_2H_50H	% об.	68,4	68, 4	68,3	67,7		68,1	67,9
pH	-	5,34	5,01	4,17	4,15		4,21	4,17
Aromatic components of distillate								
Superior alcohols	mg/100cm^3	1555,01	1275,09	886,35	597,96	38,45	1166,25	995,20
Butanol-1	mg/100cm^3	3,4	2,82	1,93	1,29		2,55	2,17
Butanol -2	mg/100cm^3	9,3	7,62	5,39	3,72		6,97	5,95
n-Propanol	mg/100cm^3	211,01	173,02	122,38	82,29	38,99	158,24	135,04
Isobutyl alcohol	mg/100cm^3	450,37	369,30	256,71	171,14	38,00	337,78	288,24
n-Butanol	mg/100cm^3	9,04	7,38	5,13	3,42		6,75	5,76
Isoamyl alcohol	mg/100cm^3	980,37	794,04	558,77	372,51	37,99	735,23	627,39
N–Amyl alcohol	mg/100cm^3	2,04	1,64	1,14	0,76		1,5	1,28
n -Heptanol	mg/100cm^3	0,31	0,25	0,17	0,11		0,25	0,19
Hexanol	mcg/dm^3	17,32	14,20	10,05	6,58		14,72	11,09
N -Octanol	mg/100cm^3	0,20	0,16	0,11	0,06		0,17	0,13
2-Phenylethanol	mg/100cm^3	13,68	13,45	13,11	12,89		11,63	8,76
Aldehydes	mg/100cm^3	86,30	86,98	87,93	88,82		87,97	88,88
Medium ethers	mg/100cm^3	248,00	247,87	247,63	247,64		247,59	247,51
ethyl acetate	mg/100cm^3	221,12	221,01	221,00	221,01		221,00	221,04
Enanthic ethers	mg/100cm^3	15,02	15,01	15,01	14,99		14,89	14,73
Ethyl lactate	mg/100cm^3	2,24	2,23	2,21	2,17		2,22	2,19
Diethyl succinate	mg/100cm^3	1,06	1,04	1,03	1,00		1,04	1,03
Ethyl capronate	mg/100cm^3	0,24	0,24	0,22	0,24		0,24	0,23
Ethyl miristate	mg/100cm^3	0,02	0,02	0,01	0,01		0,02	0,01
Ethyl lactate	mg/100cm^3	3,62	3,61	3,59	3,57		2,60	3,58
Ethyl caprilate (C8)	mg/100cm^3	0,16	0,15	0,15	0,14		0,15	0,13
Ethyl caprinate (C10)	mg/100cm^3	4,21	4,20	4,17	4,11		4,18	4,15
Ethyl laurate (C12)	mg/100cm^3	3,50	3,49	3,46	3,46		3,47	3,44
Acetic acid	mg/100cm^3	22,00	25,65	27,77	39,01		28,45	39,54
Methanol	g/dm^3	0,91	0,81	0,59	0,47		0,63	0,56
Oak aromatic components								
MC of β-methyl-γ- octa-lactones (total)	mcg/dm^3	0,00	0,091	0,16	0,25		0,20	0,23
MC of vanilla	mcg/dm^3	0,00	0,75	1,36	2,12		1,69	2,04
MC of eugenol	mcg/dm^3	0,00	0,14	0,25	0,39		0,34	0,37

Table 7. Organoleptic evaluation of young distillates with a fusil flavor before and after the contact with oak chips and staves

Names of cognac spirit samples	Sample of cognac spirit and period of ageing					
	Young cognac spirit with a flaw	*Experiment* Oak chips amount - 8 g/dm^3 Ageing in reservoir with chips 1.500 dal			*Reference* Staves – in an amount of 86 cm^2/dm^3, Ageing in reservoir with capacity 1.500 dal	
		3 months	6 months	14 months	6 months	14 months
	-	Medium fraction chips, ratio 1:1=N:T			Oak staves	
Organoleptic characteristics of cognac spirits samples						
Color	Colorless	Light straw with amber undertone	Straw with amber undertone	Amber	Light golden	Light golden with light brown undertone
Bouquet	Noticable light floral tones predominantly with fusel tone, without tones pf ageing	Fusel tones and light tones of ageing: vanilla, coconut, spices	Tones of ageing and light fusel tone	Tones of ageing without fusel tone	Complex, moderately sharp with tones of vanilla, cloves and coconut and with fusel tone	Tones of ageing and light fusel tone
Flavor	Sharp tone of young spirit with fusel tone	Sharp with light pleasant undertones and noticeable fusel tone	Balanced, soft, moderate oak bitterness is not noticeable, light fusel tone	Balanced, moderately harmonious, sharp, fusel tone and oak bitterness are not noticeable	Disbalanced, burning, with light tones of vanilla, coconut, fusel tone is noticeable	Balanced, soft, harmonious with tones of vanilla, spices, light fusel tone
Scorings	77,8	82,4	86,3	90,2	85,6	89,5

For the purpose of reference, we add oak staves into a reservoir with wine distillate of storage capacity 1.500 dal in an amount of 86 cm^2/dm^3 or 8 g/dm^3.

During 6 months of active contact between wine distillate and oak staves, under the same regimes and parameters as with oak chips, reduction of concentration of superior alcohols was on average 25%, methyl alcohol – 38.5% and after 14 months – 64%. However organoleptic parameters showed that fusel tone was still observed even after 14 months of ageing of cognac spirits interacting with oak staves which is indicative of not adequate surface area of staves for interacting with superior alcohols and methyl alcohol, in comparison with processing distillate with oak chips (scorings is 90.5).

Thus, our researches on reducing concentration of superior alcohols in young wine distillates prove the hypothesis about efficiency of using crushed oak wood in eliminating the flaw "fusel tone" in young cognac spirits, produced with the violation of the "Major rules for cognac production". The amount of oak wood is 8 g/dm^3, the time of interaction is at least 14 months. The method of reservoir ageing with oak staves according to professor Agabalyants G.G. does not give the same effect in 14 months. For this reason, to eliminate such a flaw as "fusel tone" in young wine distillates we recommend the method of processing with specially prepared composition of oak chips.

The use of wine materials with mousy taint for further distillation into cognac spirits is prohibited by the “Major rules for cognac production”[19]. It is explained by the fact that substances flavoring mousy droppings contain cancerogenic agents dangerous for health of the consumers. Moreover, cognac produced of such distillate has unpleasant tones in its flavor.

Traditional ways for removing this unpleasant tone in accordance with technological instructions for correcting cognac spirits flaws is the use of activated charcoal, cognac blend fining with protein substances (albumin, gelatin, egg white, fish glue) as well as with absorbents such as bentonite, silicon dioxide etc. In the result of integrated processing with the aforementioned methods, the cognac loses not only its mousy taint but aromatic and flavor characteristics as well. Aroma, flavor, bouquet, taste and aftertaste are also removed. The beverage becomes neutral, moreover the mousy taint does not disappear completely and leaves an aftertaste[9].

The results of our research of oak chips properties in theor interaction with various wine materials and distillates, including those with mousy taint, we noticed elimination of this flaw after beverage contact with oak wood.

With the purpose of exploring the potential of eliminating mousy taint in young cognac spirits, we conducted experiments on processing young cognac spirits with mousy taint with oak-based substances in different ratios of heat-treated and natural wood and in different amounts.

Reference point 1 was activated birch charcoal with “B” marking (acidic) in an amount of 8 g/dm^3 used in food industry for eliminating alien aromas and flavors.

As oak derivative products we used oak chips chips (mixture of natural and het-treated chips in ratio 1:1 and in amount of 8 g/dm^3) – experiment, and staves – reference, obtained from the same batch of an English oak of over 120 years old grown in Ismaillinsky region. Oak staves were placed in a form of a well inside a reservoir84 cm^2/dm^3.

The results of the experiments showed that the most optimal ratio of composition was 1:1 of heat-treated wood to natural wood in an amount of 8 g/dm^3.

Organoleptic parameters of wine distillates with a flaw “mousy taint” before and after interaction with a specifically toasted chips are indicative that significant changes in organoleptic parameters occur in 6-7 months after their active contact with oak wood, however the flaw remains in aftertaste.

It was found that complete elimination of a flaw occurs in 11-12 months after the contact. The tone of mice droppings is eliminated in aroma, flavor and bouquet, and the distillate acquires organoleptic parameters that are characteristic of sound, aged cognac spirit.

After processing cognac spirit with activated birch charcoal, we found that in the spirits the majority of undesirable chemical components (acetamide) were eliminated due to the absorption together with the components responsible for aroma and flavor. Thus, reference cognac acquired neutral tones which are not typical for cognac spirits.

The results of the experiment showed that it is not possible to eliminate mousy taint from cognac spirits, Table 8.

The changes in organoleptic parameters of distillates in the process of eliminating the flaw with oak chips in 12 months are evidenced by the changes in their physical and chemical parameters. Mass concentration of aroma-producing components in spirits is reduced in superior alcohols by 60%, aldehydes – 13%, medium ethers – 8%, methanol – 48%. Besides, after eliminating mousy taints as the result of contact with oak wood cognac spirits acquire noble tones of ageing typical for cognac spirits aged with oak wood for at least 1-3 years, Table 8.

In other experiments we did not manage to obtain a positive result for eliminating a flaw and for this reason it is not recommended to use it for producing cognac or brandy.

Mechanism for eliminating the flaw “mousy taint” has not been studied up to the end. Potential mechanism for eliminating mousy taint in wine distillates may be divided into two stages. At the *first stage* oak wood with a maximal toasting degree absorbs organic and highly molecular substances with non-polar structure, for example carbohydrates, superior alcohols, medium ethers, ethyl acetate (solvents), organic acids etc. Absorption capabilities increase with lower solubility in water, higher non-polarity of the structure and higher molecular mass of the components. Heat-treated wood better absorbs vapors of the substances with high boiling temperatures than volatile compounds.

At the *second stage*, cognac spirit is enriched with aroma-producing components of oak wood: phenolic substances, fragrant lactones, eugenol etc.

The result of the researches on eliminating mousy taint in young wine distillates resulted from the violation of the “Major rules for cognac production” prove our hypothesis

Table 8. Physical and chemical parameters of distillates with mousy taint after their contact with oak chips and oak staves

Parameter MC – mass concentration of components	Measuring Unit	MC of analyzed substances in cognac spirits (GOST R 51145-98)					
		Experiment: Oak chips				Reference: Staves and activated birch charcoal B	
		Initial	3 months	6 months	12 months	Charcoal B 12 months	Staves 12 months
Volume Ratio C_2H_50H	%	64,8	64,6	68,6	64,5	64,4	64,4
pH	-	4,98	4,83	4,76	4,55	4,41	4,68
Aromatic components of distillate							
Superior alcohols	mg/100cm³	366,81	297,10	240,54	150,39	146,18	234,75
Aldehydes	mg/100cm³	16,65	16,68	17,24	17,33	14,54	18,96
Medium ethers	mg/100cm³	127,00	127,75	127,63	148,44	117,02	148,32
Ethyl acetate	mg/100cm³	77,26	77,68	77,76	78,14	66,85	77,99
Enanthic ethers	mg/100cm³	17,54	17,51	17,67	17,68	17,53	17,56
Acetic acid	mg/100cm³	11,30	11,35	11,90	12,87	11,89	12,75
Methanol	g/dm³	0,23	0,19	0,17	0,14	0,12	0,17
Aromatic components of oak							
MC of β-methyl-γ- octalactones (total)	mg/100cm³	0,00	0,15	0,21	0,33	0,00	0,19
MC of vanilla	mg/100cm³	0,00	0,98	1,74	2,76	0,00	1,95
MC of eugenol	mg/100cm³	0,00	0,17	0,24	0,40	0,00	0,41
Tasting scores							
Color		colorless	Light straw	Straw-amber	Light amber	Colorless	Light straw
Aroma and flavor		++	+	++	+++	-	++
Taste		++	+	++	+++	-	+
Aftertaste		+	+	+	++	-	+
Flaws, presence of mousy taint		++++	++++	+	-	-	+++

*) +) – presence, -) absence

about efficiency of using oak wood for eliminating this flaw form young cognac spirits. The amount of oak wood is 8 g/dm³, the time of interaction is at least 12 months. We recommend this method for industrial use.

For manufacturing testing we used white Port wine material with mousy taint. For a contact with wine material we used oak derivative products (chips) (staves, micro-staves, chips and micro-chips of different sizes and crushing degree), both natural and heat-treated and produced in compliance with TU B 19412998.001-99. We used the chips in accordance with "Technological instruction for using dispersed oak wood in wine and cognacs production" TS (technical specification) U 19412998.001-99 (supplement L). Evaluation and wine materials sample selection before and after processing was performed by organoleptic method by identifying flaws and faults in aroma and flavor, then we conducted studies on their physical, chemical and microbiological parameters (Table 10).

The scheme of wine material processing with mousy taint was traditional (reference) and included the following technological operations: - white Port wine material was sulfitated by adding 50 mg/dm³ of sulfurous acid and acidified with tartaric acid in an amount of up to 2 g/dm³, and then processed with bentonite (the amount of bentonite 1.0 g/dm³ was defined by laboratory trial processing).After retention processed wine material was decanted by filtration.

Wine materials were processed using the following technological scheme (experiment): - faulty white Port wine material was sulfated by adding sulfurous acid in an amount of 50 mg/dm³ and acidified with tartaric acid in an amount of up to 2 g/dm³. Then we added oak chips in an amount of 1.0 g/dm³ and the ration of chips was 1:1 = N:T. The period of ageing the wine material with the chips lasted for 3 months.

The scheme of processing wine materials with mousy taint by the traditional method (reference) included the following stages: white Port wine material was sulfitated by adding 50 mg/dm³ of sulfurous acid and acidified with tartaric acid in an amount of up to 2 g/dm³, and then processed with bentonite (the amount of bentonite 1.0 g/dm³ was defined by laboratory trial processing). After retention processed wine material was decanted by filtration.

Processing of wine materials by the proposed method was performed by the following scheme: faulty white Port wine material was sulfitated by adding 50 mg/dm³ of sulfurous acid and acidified with tartaric acid in an amount of up to 2 g/dm³. Then we added oak chips in an amount of 1.0 g/dm³ and the ration of chips was 1:1 = N:T. The period of ageing the wine material with the chips lasted for 3 months.

Efficiency of processing of wine materials was established on the base of studying its physical and chemical and microbiological parameters shown in Tables 9 and 10. We also tested the sample of white Port on its tendency to turbidity caused by metal ions (metal casse), colloidal (reversible and irreversible), crystallic and biochemical turbidities in accordance with generally

Table 9. Physical and chemical parameters of white Port wine material with mousy taint after processing by traditional and proposed methods

No. of sample	Processing type	Alcohol volume ratio, %	Mass concentration in wine materials						iron, mg/dm³	pH	Assessment,
			sugars, g/100 cm³	Titratable acids, g/dm³	Volatile acids, g/dm³	фенольных веществ, mg/dm³	SO_2, mg/dm³ free	SO_2, mg/dm³ total			
	Initial wine material	17,2	9,4	3,9	0,48	420	5,0	8,96	25,4	3,83	8,0
Traditional method of processing											
1	Sulfitation→ acidification→ processing with bentonite→ filtration	17,2	9,35	6,7	0,42	395	24,32	107,52	7,4	3,6	8,9
Proposed method of processing											
2	Sulfitation→ acidification→ chips processing → filtration	17,2	9,35	6,7	0,3	425	25,6	115,2	4,4	3,42	9,3

Table 10. Microbiological parameters of white Port wine materials with mousy taint after processing

Amount		Amount in 10 ml		Groups of microorganisms in 1 cm³		
Samoles of wine	Cells in native material	Yeast cells	Potentially pathogenic enterobacteria	CFU[1] of lactic acid bacteria	CFU of yeast cells	CFU of acetic acid bacteria
Initial wine material						
4	0	0	0	0	0	0
Reference (traditional method)						
3	0	0	0	0	0	0
Experiment (proposed method)						
4	0	0	0	0	0	0

[1]CFU – colony-forming units

Table 11. Tendency of white Port to turbidities and mousy taint

Test results on tendency to turbidity	Type of turbidity					
	Caused by metals (metal casse)	Irreversible Colloidal	Reversible Colloidal	Crystallic	Biochemical	Test on "mousy taint" ("soda test")
Initial wine material	+	-	-	-	-	Wine darkening, mousy taint
Reference (traditional method)	-	-	-	-	-	Very slight mousy taint
Experiment (proposed method)	-	-	-	-	-	Mousy taint is absent

accepted methods. We also tested wine materials on the presense of mousy taint – the so-called "soda test". Organoleptic parameters were identified by the method of tastings (Table 11).

The results of physical, chemical, and microbiological studies of white Port wine material with mousy taint processed by traditional and proposed method, we found the following.

Reference sample of white Port processed by traditional method was characterized by better qualitative parameters in comparison with initial wine material. It had higher concentration of titratable acids and higher organoleptic scores (8.8 in comparison with initial – 8.0). In the processed sample the mousy taint was almost completely eliminated (Figure 8).

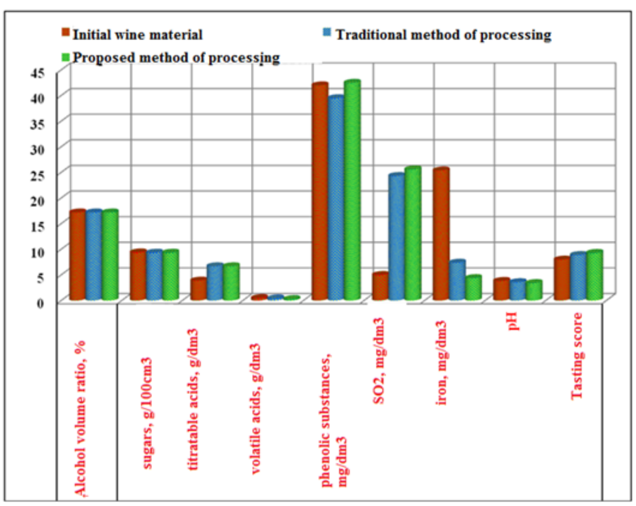

Figure 8. Physical and chemical parameters of white Port with mousy taint after processing by traditional and proposed methods.

Unlike reference sample, the sample under analysis processed with proposed method with the use of oak chips, was characterized by better organoleptic properties and physical and chemical parameters. We observed not only higher concentration of titratable acids (due to tartaric acid), but also reduction of concentration of volatile acids by 0.18 g/dm^3 and iron ions by 2.1 – 3.4 mg/dm^3 as well as increase of phenolic substances concentration. By 5-15 mg/dm^3 and higher pH value by 0.2-0.3 units. Such changes in chemical content supported the possibility and practicability of using oak wood as a sorbent. It is especially beneficial for organoleptic parameters of wine materials (scoring 9.3 in comparison with 8.0). The sample acquired clean and complex bouquet with noticeable fruit tones and harmonious, clean, soft, full and balanced flavor with ageing tones (oak wood – vanilla, coconut etc.). The feeling of mousy taint disappeared and that was confirmed by the results of the "soda test".

Manufacturing testing showed efficiency and practicability of using oak derivative products for treating or eliminating wine faults, in particular mousy taint.

5. Conclusions

Conducted researches and manufacturing testing of the methods for treating flaws and faults let us make the following conclusions:

- Use of oak derivative products has a positive effect of treating and eliminating mousy taint and improves organoleptic characteristics;
- Use of oak derivative products for treating faults and correcting mousy taint in dry and fortified wines should be accompanied by preliminary sulfitation and acidification of wine materials to the concentration of titratable acids at least 6.0 g/dm^3;

- To make the processing with oak derivative products even more efficient, it is reasonable to use chips in filtering bags of lavsan or capron fabric, and it is recommended to mix it 2-3 times.

Obtained results suggest that the proposed method for treating and correcting wine flaws with oak derivative products is efficient and can be implemented into production.

Novelty of the developed method for treating and eliminating faults and flaws in wines using oak derivative products is confirmed by the invention patent of Azerbaijan

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