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Chemical and Sensory Comparison of Classical and Alternative Systems for the Ageing of Wine Distillate

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Abstract

The chemical components were quantified in artificially aged (with oak chips) and barrel-aged wine distillate. These components belong to various chemical families, including aldehydes (acetaldehyde, propionic aldehyde, furfural, coniferaldehyde, synapaldehyde), higher alcohols (methanol, ethanol, 1-Propanol, butanol, isobutanol, amyl alcohol, isoamyl alcohol, 1-hexanol), volatile acids (ethanoic acid, propionic acid, 3-methylbutanoic acid, hexanoic acid, heptanoic acid, octanoic acid) and esters (methyl formate, ethyl acetate, butyl butyrate, methyl butyrate). Chemical analysis was performed by classical methods of analytical chemistry. During the seven month aging process all the chemical components were affected by ageing systems. The analysis of alcoholic strength by volume, aldehydes and volatile acids showed a great discrimination of the brandies based on the ageing system. The loss of alcohol was lower in a glass vessel with oak chips than in oak wood barrel. Thus, artificial ageing is cost-efficient method than the classical one. Moreover, the ageing system affected the sensory profile of the wine distillates as well. The present study demonstrated that alternative ageing up to five months is the most promising technology to get desirable colour. However, traditional wine spirit ageing method is preferable to produce high quality brandy compared to alternatives as spirit aged in Limousin oak barrels are more matured than the one aged with oak chips.

Keywords: wine distillate; oak barrel; artificially aged; chemical components; oak chips.

1. Introduction

Brandy is produced from wine spirit, which is matured for at least six months with oak [1]. During the aging period, slow physicochemical changes involving both brandy and wood take place.

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These changes result in radical modifications of the product, producing well-known changes in colour, taste and flavor. In this evolution, there are changes in both the composition and concentration of many compounds related to the sensory characteristics of the brandy [2, 3]. The process of ageing spirit reduces both its volume and the alcoholic content. The time required for satisfactory maturation varies, depending on the characteristics of the raw distillate, the origins of the wood, the size and treatment of the cask and the environment in which the spirit is matured [4]. The oak wood, especially from the French region of Limousin, is traditionally used in the ageing of wine spirits. Recently study has been done to identify different aging technics and materials as alteration of high cost oak barrels. Additionally, financial attractions are in reducing storage capacity and reducing the large evaporation losses that result during maturation [5]. In recent years the addition of oak fragments has been used to introduce desirable oak aromas and flavours into beverages and accelerate and decrease cost of the aging process [6,7]. Considering the overall quality of the brandies, the results of recent study suggests the use of wood fragments to be an interesting alternative technology [8]. The wooden barrels promote greater enrichment in the majority of the compounds. However, gallic acid, ellagic acid and syringaldehyde, and vanilla and 5-methylfurfural, which are strong antioxidants and key-odourant compounds, respectively, present higher contents in the brandy aged with the alternative technologies (wood fragments) [9,10]. The results suggest that the use of alternative systems allows producing brandies with slightly different sensory profile [11], the most discriminating factor is the colour. Wood tablets cause the fast evolution of the colour of brandies [12]. There is not enough information about the alternative aging systems for the wine distillate, while growing brandy industry needs additional studies. The present research will show a difference between two systems for the ageing of wine distillate. The novelty of this study is that comparison of wine distillates aged in Limousin oak barrel and with oak chips was performed for the first time. The aim of this work is to compare alternative wood ageing system (oak chips) with Limousin oak barrels in the ageing of the wine spirit, during 7 months. The spirits from different ageing systems are evaluated by chemical composition and sensory quality.

2. Materials and Methods

2.1 Raw material

The raw material used was a wine distillate with an alcoholic strength by volume of 68.63 %, produced on an industrial scale by a Georgian brandy factory, JSC Sarajishvili (Tbilisi, Georgia). Grapes were from Kvareli, Kakheti region, Georgia. Grape variety was Rkatsiteli. Wine was made at Graneli wine factory (Kvareli, Kakheti region, Georgia). Fermentation was initiated with wild yeast and temperature range was 20-30 °C. The distillation was done in JSC Sarajishvili distillation factory (Gremi, Kakheti region, Georgia). The wine was distilled with classical technology, in French Charentaise alembics. The latest distillation was done by the end of January. Maturation of distillate samples was performed using new Heavy Toast Limousin French oak barrel (400 L) and glass vessel (20 L) with Heavy Toast French oak chips (producer for both barrel and chips - Tonnellerie Allary, France). The chips size was 5/12 mm, dose - 6 g/L.

2.2 Experimental design

The same distillate was placed in heavy toast Limousine oak barrel (the first sample) and in a glass vessel with heavy toast French oak chips (the second sample). The prepared samples were aged for 7 months. The conditions for ageing in the storeroom were as follows: temperature was 19 °C, the humidity was 78%. The ageing process was held by static system (second sample was stirred for only 2 min per day, 3 times per week). Both samples were analyzed at the start of the ageing process and after 3, 5 and 7 months. The analyses were: global parameters by classical methods, such as alcoholic strength by volume (%), total aldehydes (mg/L), total esters (mg/L), total higher alcohols (mg/L), total volatile acids (mg/L) and organoleptic analysis (sight, smell, taste) by the experts. The control sample for each variant was the wine distillate before ageing.

2.3 Identifying alcoholic strength by volume

Ethanol concentration (*i.e.* alcoholic strength by volume; ASV (%)) was measured with an alcoholmeter using the percentage scale (by volume of ethanol) at 20 °C. If the distillate temperature diverged from 20 °C, then a correction was applied, using alcohol metric tables [13].

2.4 Identifying aldehydes

This method was used to identify total aldehydes (acetaldehyde, propionic aldehyde, furfural, coniferaldehyde, synapaldehyde). Prior to titration, 25 mL of sample was transferred to the glass flask with adding 20 mL pH 7 and 5 mL potassium metabisulfite. The mixture was then cooled in icy water for 15 minutes. Then was started titration by adding 1 mL of starch solution and 5 mL of hydrogen chloride. In the next step, 0.1 N iodine was added till sample got light pink colour, than was added thiosulfate till getting colourless. Then was added 3 drops of phenolphthalein solution and titrated with sodium borate. After adding 1 mL of starch solution, 0.01 N iodine was added to sample till it got violet colour. The amount of aldehydes (mL) is calculated by Equation 1.

$$X = \frac{V \times 88}{C} \quad (1)$$

Where: V is spent amount of 0.01 N iodine (mL) and C is alcoholic strength by volume of sample (%).

2.5 Identifying esters

This method was used to identify total esters (methyl formate, ethyl acetate, butyl butyrate, methyl butyrate). 50 mL of sample was transferred to the glass flask. Then was added 3 drops of phenolphthalein solution and titrated with 0.1 N sodium hydroxide. The next step is to add 15 mL of 0.1 N sodium hydroxide and left the sample at ambient temperature for 7 hours with periodically mixing. Then sample is titrated with 0.1 N sulfuric acid. The amount of esters (mL) is calculated by Equation 2.

$$X = \frac{(15 - V) \times 1760}{C} \quad (2)$$

Where: V is spent amount of 0.01 N sulfuric acid (mL) and C is alcoholic strength by volume of sample (%).

2.6 Identifying higher alcohols

This method was used to identify total higher alcohols (methanol, ethanol, 1-Propanol, butanol, isobutanol, amyl alcohol, isoamyl alcohol, 1-hexanol). 20 mL of sample was transferred to glass flask and added the deionized water. Water amount (mL) is calculated by Equation 3.

$$X = \frac{C \times 20}{40} - 20 \qquad (3)$$

Where: C is alcoholic strength by volume of sample (%).

10 mL of mixture was transferred to 50 mL glass flask and filled with 40% ethanol solution. Then from this flask 0.5 mL of sample was transferred to 25 mL glass flask. As control sample 0.5 mL of 40% ethanol solution was transferred in second 25 mL glass flask. In the next step, both samples were placed in icy water and 10 mL of para-dimethylaminobenzaldehyde was added to both flasks. After this both samples were placed in boiling water for 30 minutes. Then cooled samples were transferred to 3 mm cuvettes and measured at 490 nm in photoelectric colorimeter.

2.7 Identifying volatile acids

This method was used to identify total volatile acids (ethanoic acid, propionic acid, 3-methylbutanoic acid, hexanoic acid, heptanoic acid, octanoic acid). 20 mL of sample was transferred to the glass flask and 50 mL of hot deionized water was added. In the next step 3 drop of phenolphthalein solution was added to sample and titrated with 0.05 N sodium hydroxide. The amount of volatile acids (mL) is calculated by Equation 4.

$$\mathrm{X} = \frac{1500 \times \mathrm{V}}{\mathrm{C}} \qquad (4)$$

Where: V is spent amount of 0.05 N sodium hydroxide (mL) and C is alcoholic strength by volume of sample (%).

2.8 Sensory evaluation

Sensory evaluation of wine spirits was done by expert panel, which consisted of the 5 member (2 men and 3 women). Sensory evaluation of the samples occurred in a degustation room and the environmental conditions were kept as constant as possible. Each expert was supplied with mineral water. The experts scored different characteristics that described the sight, smell and taste of each sample on a scale divided from 0 to 10, with 0 being absence of the described attributes and 10 - the presence of the one.

3. Results

3.1 Changes in chemical components

In this study, the same wine distillate ASV=68.63% was stored in glass vessel with oak wood chips and in oak wood barrel. The loss of alcohol after 7 months of aging was higher with 1.1% in oak wood barrel than in glass vessel with oak chips (see Figure 1). Alternative aging method does not produce the changes in concentration that occur due to evaporation during cask ageing [5]. This proves that alternative methods for ageing is cost efficient than classical methods.

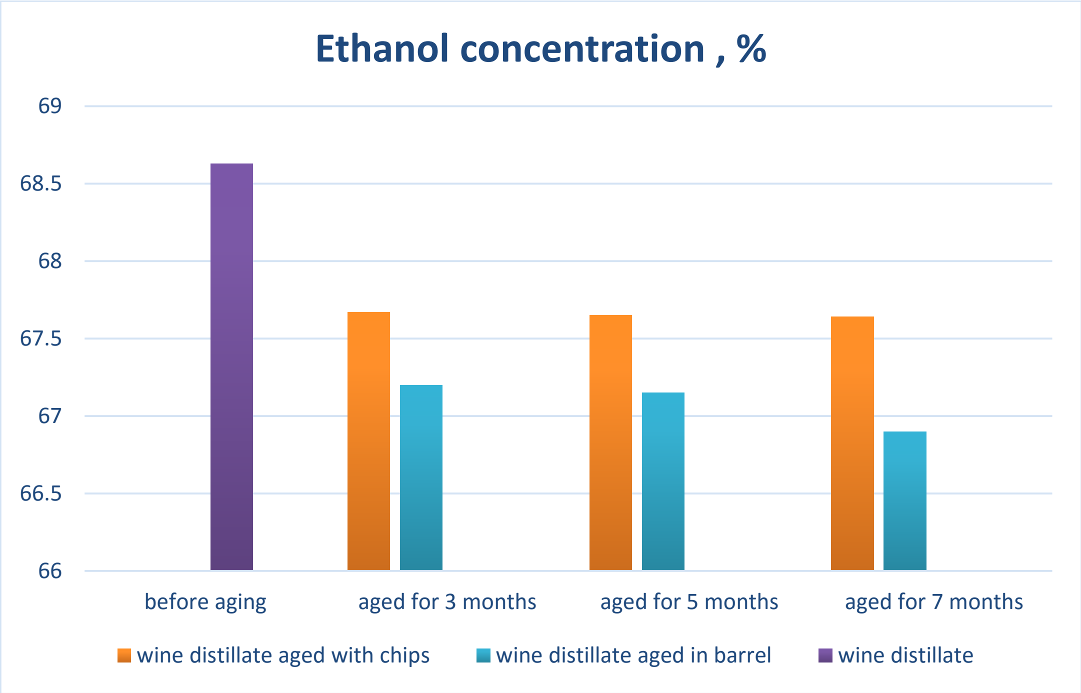

Figure 1: Ethanol concentration, % in wine distillate, barrel-aged wine distillate and artificially aged wine distillate before ageing and after 3, 5 and 7 months of ageing.

The concentration of volatile acids in sample aged with chips after 3 and 7 months decreased by 44.8% and 35%, respectively. In the barrel-aged sample after 3 and 7 months the initial concentration increased by 0.4% and 23.3%, respectively (see Figure 2). Remarkable increase of volatile acidity was reported in barrel-aged other distillates and low level was found in oak chips-aged wine [14, 15].

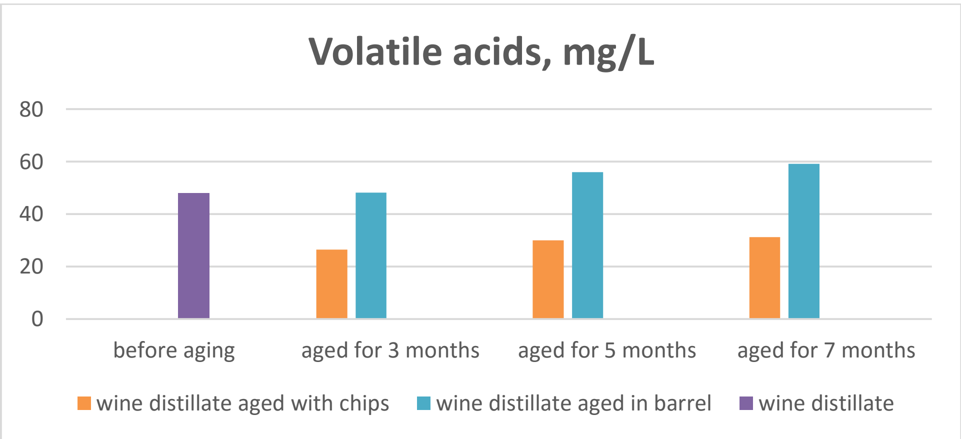

Figure 2: Volatile acids, mg/L in wine distillate, barrel-aged wine distillate and artificially aged wine distillate before aging and after 3, 5 and 7 months of aging.

Total aldehydes content is much lower in wine spirit treated with oak chips than that of barrel-aged one. The concentration of aldehydes increased by 5% in artificially aged and by 16% in barrel-aged wine distillate (see Figure 3). Similar was found in 6 months aged wine-brandy, highest aldehydes levels occurred in the brandies aged in barrels. Aldehydes are the most crucial compound of our study, as mainly defines organoleptic characteristics. According to several researches, total aldehydes presented important correlations with several olfactory attributes like vanilla, smoke, toasted, dried fruits, woody, which influence positively the quality of the brandies [16, 1].

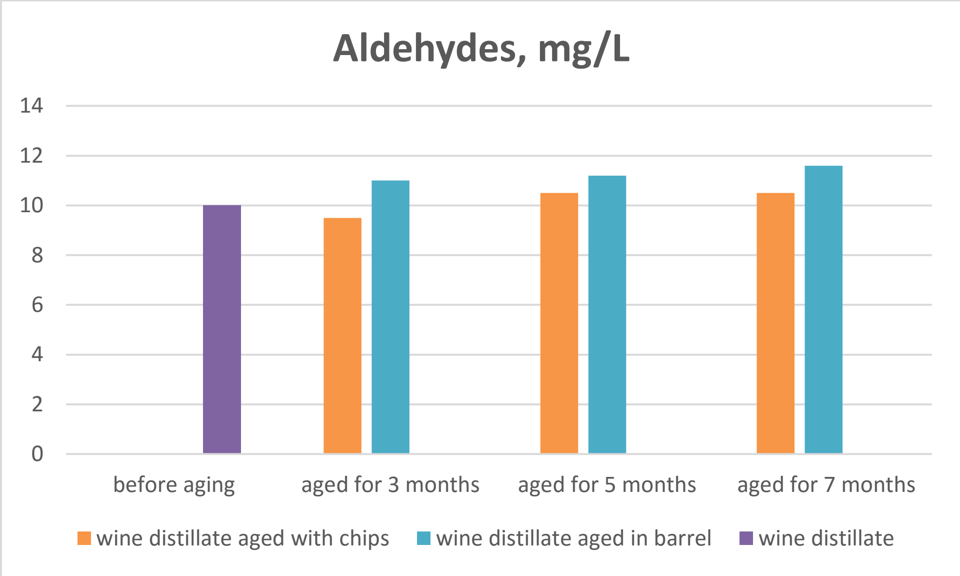

Figure 3: Aldehydes, mg/L in wine distillate, barrel-aged wine distillate and artificially aged wine distillate before aging and after 3, 5 and 7 months of aging.

Statistically significant changes of total esters content were observed only in sample of oak barrel, level increased progressively during 7 months ageing. Total esters concentration increase in barrel and artificially aged samples were by 5.5% and 0.8%, respectively (see Figure 4). The typical taste and flavour of aged distillates are mainly attributed to the formation of aromatic esters, compounds that contribute to the formation of the bouquet. Ethyl acetate, the major component of this group, is responsible for the agreeable flavour of aged spirits. These reactions also take place in beverages stored in inert containers, but they are slower owing to a lack of specific reactions that accelerate the oxidation process of the distilled spirits during aging [17,14]. Volatile acids level has impact on esters. The highest levels of acetic acid were found in the brandies aged in the barrels and the lowest level in the brandies aged with tablets. Consequently, this could explain the higher levels of related acetic esters, like isobutyl acetate and isoamyl acetate, found in the brandies aged in the barrels [8].

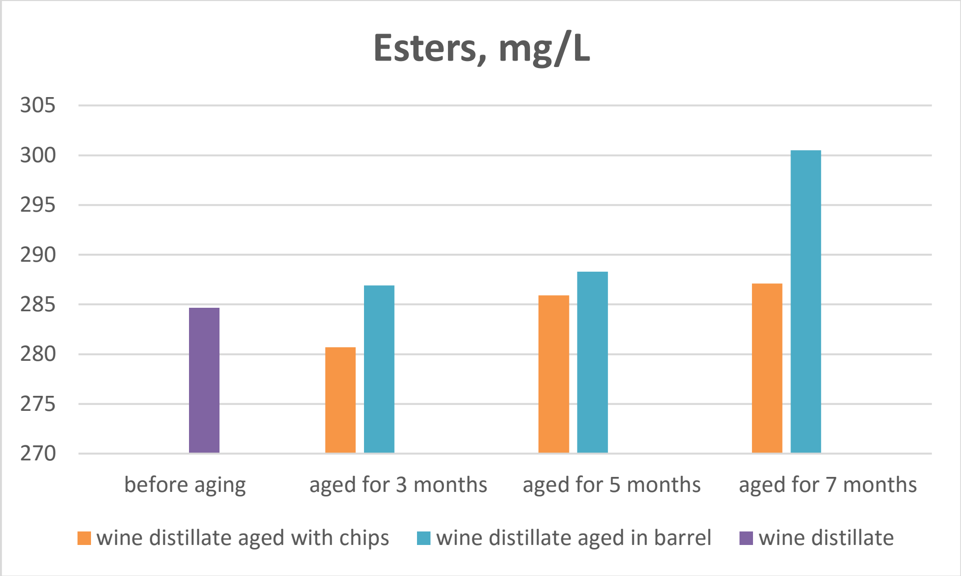

Figure 4: Esters, mg/L in wine distillate, barrel-aged wine distillate and artificially aged wine distillate before aging and after 3, 5 and 7 months of aging.

Higher alcohols levels increased with all ageing systems, but the highest significant levels were found in the brandies aged in barrels. The concentration of higher alcohols increased by 31.8% in barrel-aged wine distillate, whereas in the artificially aged one it increased just by 18.6% (see Figure 5). Research about wine reported that pleasant fresh odours in distilled spirit are correlated with C6 alcohols [18], that is essential factor for sensory analysis.

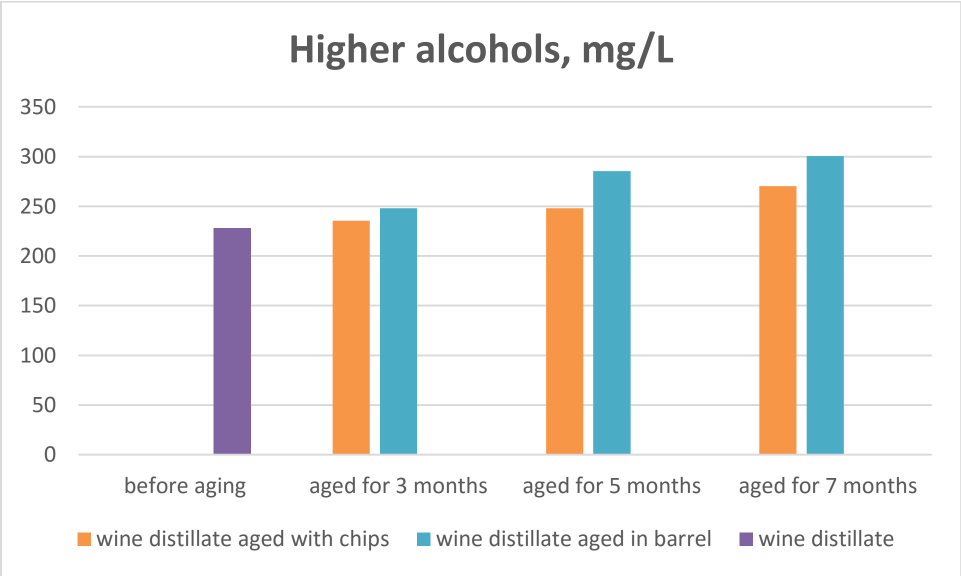

Figure 5: Higher alcohols, mg/L in wine distillate, barrel-aged wine distillate and artificially aged wine distillate before aging and after 3, 5 and 7 months of aging.

3.2 Results of sensory evaluation

In this study, the sensory evaluation of brandy spirit by trained experts gave higher results to spirit samples of the barrel-aged group, in comparison to the oak chips group (see Table 1).

Table 1: Sensory evaluation of samples by experts with 10-point scale.

Sample	**Expert #1**	**Expert #2**	**Expert #3**	**Expert #4**	**Expert #5**	**Total**
Wine distillate	8,5	8,5	8,5	8,6	8,7	42,8
Wine distillate aged for 3 months (barrel)	8,7	8,6	8,8	8,8	8,9	43,8
Wine distillate aged for 3 months (chips)	8,85	8,5	7	8,75	8,85	41,95
Wine distillate aged for 5 months (barrel)	8,85	8,7	9	8,85	8,95	44,35
Wine distillate aged for 5 months (chips)	8,7	8,6	7	8,75	8,85	41,9
Wine distillate aged for 7 months (barrel)	10	8,8	8	8,9	9	44,7
Wine distillate aged for 7 months (chips)	**8,85**	**8,7**	**7**	**8,8**	**8,85**	**42,2**

By all organoleptic characteristics barrel-aged wine distillate was more matured than the spirit aged with oak chips, same result was found in wine-brandy aged for 6 months [8]. Our study demonstrated that wine distillate aging with chips is the most promising for quick result in getting desirable colour. Similar results were reported in research of wines treated with oak chips, which gained colour more quickly than wines aged in barrels and more oak volatile extraction were observed [19]. However even in this component in present study barrel-aged wine spirit gets higher level after 5 months in comparison to artificially aged one. Aldehydes, esters and higher alcohols are chemical markers to distinguish artificially and barrel-aged wine distillate, as they are related to aged brandy aromas [16]. Their levels increased with all ageing systems, but the highest significant levels were found in the distillates aged in barrels. Therefore, chemical composition reflected on sensory evaluation. Consequently, it might be advisable for the brandy industry to age wine spirit in oak barrel to produce high quality product.

4. Conclusions

Considering the ageing period analysed (7 months) these results revealed some important indications about the alternative ageing system (oak chips) for wine distillate. Concerning the chemical composition of the wine spirits, the results showed the possibility of discrimination between spirits based on the ageing system. The more discriminating variables are: alcoholic strength by volume, aldehydes and volatile acids. The ageing system affected the sensory profile of the wine distillates. The spirit aged in Limousin oak barrels seem more matured than the one aged with oak chips. Therefore, the results of our study may be used in practice in the brandy industry.

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