Influence of Ageing Process on the Chemichal Composition and Some Aromatic Compounds on Muschat Hamburg Distillate Produced in Albania

Klotilda Marku, Renata Kongoli, Vlash Mara
Faculty of Biotechnology and Food
Agricultural University of Tirana
Tirana, Albania

e-mail: klodimarku@gmail.com

Abstract:- **Under study has been taken the variety Muscat Hamburg cultivated in Durres- Albania area in 2009, according to established agro-techniques. The variety taken in the study is characterized from the pronounced aroma flower. As an alcoholic distillate it can be highly aromatic. Muscat grapes contain a number of compounds that give muscat wines and distillates their distinct flavor. The study aims to identify these compounds after distillation process and after three years of ageing in Q.robur oak barrels.The analytical control for determining the aromatic compounds was conducted with the official methods of OIV and REG-CE 2870/2000 GC-MS and GC-FID. According to the analytical results performed respectively to the fresh distillate and the aged distillate, we see that some compounds increase in level such as: superior alcohols have a significant decrease from 221gr/hl (at the fresh distillate) to 282gr/hl anhydrous alcohol (at the three aged distillate), the acetic aldehydes increase from 13.3 gr/hl (at the fresh distillate) to 17 gr/hl anhydrous alcohol after three years of ageing some others increase such as linallol from 8.8 gr/hl a. a in the 19.5 gr/hl a. a, citronellol from 2.7 gr/hl a. a in the 4.3 gr/hl a. a among the compounds analyzed results that geraniol have an significant increased from 2.1 to 6.8 gr/hl a. a. By organoleptic evaluation results that the distillate produced shows pronounced aroma flower that is attributable to the variety and the highter changes of the aromatic compounds under the ageing process.**

Keywords: Muscat Hamburg; flower aroma; linallol; higher alcohols; geraniol.

I. INTRODUCION

The aromatic potential inherent in grapes can either be realized or destroyed during the alcoholic fermentation. The preferable flavors of wine depend on a balance of volatile constituents such as acids, alcohols, aldehydes, ketones and esters, with yeast bouquet resulting largely from higher alcoholsand esters (Swiegers et al., 2005) [1]. The normal methods of monitoring the fermentation used are measurement of sugar (or, more accurately, dissolved solids) by hydrometry, measurement of temperature and sensory evaluation. One mole (180 g) of glucose yields about 103 kJ (24.5 kcal) as heat, and each percent of sugar (1˚Brix) in the must generates enough heat during fermentation to raise its temperature by 1.3 ^{o}C/l, if no heat is lost. (Jackson, 2000b) [2]. Although alcohol is a major component of wine, there are many by-products of fermentation which will have a major impact on quality. The characteristic fruit flavors of wine are primarily due to a mixture of hexyl acetate, ethyl caproate, ethyl caprylate, isoamyl acetate and 2-phenylethyl acetate, that have specific functions in the yeast cell, while others are still speculative (Pretorius and Lambrechts, 2000) [3]. Higher alcohols are produced during alcoholic fermentation through the conversion of the branched chain amino acids present in the medium (valine, leucine, isoleucine, threonine and phenylalanine), and are important precursors for the formation of esters, which are associated with pleasant aromas. They can also be produced de novo from a sugar substrate (Clemente-Jiminez et al., 2005) [4]. The temperature of fermentation has a considerable effect on the aromatic character of the wine. Low temperatures and slower fermentations are well known to help retain the fruit character of the juice. These aromatic compounds are lost at high temperatures. The influence of fermentation temperature on the production of yeast-derived aroma compounds at 15˚C and 28˚C was investigated. Higher concentrations of compounds related to fresh and fruity aromas were found at 15˚C, while higher concentrations of flowery related aroma compounds were found at 28˚C. The formation rates of volatile aroma compounds varied according to growth stage.

II. MATERIAL AND METHODS

A. Variety and wort production

The variety is cultivate in field area where the soil is a clay-loess and lime is added to improve. In this ecosystem characterized as part of the Mediterranean climate the annual temperature is 11ºC, the average temperature of vine vegetation is 17˚C, the amount of active temperatures 3705°C, relative humidity 76-80%. This variety has its plant time during the months April-August and it should not be watering before harvest. (K. Marku and R. Kongoli 2012) [5] The variety taken in the study is characterized from the pronounced aroma flower. After the harvest the selected raw material is in good condition, the grape undergo to the mechanical analysis, the determination of acidity, the sugar content and then is suppressed according to the raki production. The determination of the percentage of sugar with refractometer is base on the relationship between the concentration of soluble dry matter and the coefficient of the refraction of light, the used refactometre shows Brix degree. Fermentation is monitored daily by measuring the density and temperature, after preparing the fermentated material it goes to fractional distillation. The study aims to identify these compounds in the fermented must (wine without using SO_2) before distillation process and in the distillate obtained from this wine. The distillation is carried out by a distiller allowing a high alcoholic degree without need for redistillation.

B. Analitical method

The analytical control for determining the aromatic compounds in the fresh distillate and the aged distillate was conducted with the official methods of OIV and REG-CE 2870/2000 GC-MS and GC-FID. Gas chromatographic assays of volatile compounds may prove particularly interesting as a means of determining both the origin of the raw material used in the distillation processs and the actual conditions of distillate. Some spirits contain other volatile components, such as aromatic compounds, which are characteristic of the raw materials used to obtain the alcohol, of the aroma of the spirit drink and of the special features of the preparation of the spirit. These compounds are important for evaluating the requirements set out in Regulation (EEC) No 1576/89. Congeners in spirit drinks are determined by direct injection of the spirit drink, or appropriately diluted spirit drink, into a gas chromatography (GC) system. A suitable internal standard is added to the spirit drink prior to injection. The congeners are separated by temperature programming on a suitable column (6ft x 2mm i.d, packed 5% Carbowax 20 M on 80 – 100 Carbopak B. Gase flow:He, 300- 30 ml/min) and are detected using a flame ionisation detector (FID). The concentration of each congener is determined with respect to the internal standard from response factors, which are obtained during calibration under the same chromatographic conditions as those of the spirit drink analysis.

C. Chemicals and standards

As recomanded from REG-CE 2870/2000 for this work were used exclusively reagents with purity of more than 97% purchased from a provider accredited to ISO 3696 and have a certificate of purity, free from other compounds at test dilution. Acetal and acetaldehyde must be stored in the dark at a temperature lower than 5° C; all other reagents should be stored at room temperature. Absolute Ethanol (CAS 64-17-5). methanol (CAS 67-56-1). Propan-1-ol (CAS 71-23-8). 2-methylpropan-1-ol (CAS 78-33-1-). Acceptable internal standards: pentan-3-OL (CAS 584-02-1), pentan-1-ol (CAS 71-41-0), 4-methylpentan-1-ol (CAS 626-89-1) and methyl nonanolo (CAS 1731-84-6). 2-methylbutan-1-ol (CAS 137-32-6). 3-metibutan-1-ol (CAS 123-51-3). ethyl acetate (CAS 141-78-6). Butan-1-ol (CAS 71-36-3). Butan-2-OL (CAS 78-92-2): Acetaldehyde (CAS 75-07-0). Acetal (CAS 105-57-7): Ethanolic 40% v/v. To prepare an ethanolic to 400 ml/l, put 400 ml of ethanol in a 1 litre volumetric flask and make up to volume with distilled water by mixing thoroughly. Preparation and storage of calibration solutions (procedure used for the validated method). All calibration solutions should be stored at temperature below 5° C and renewed once a month. The masses of the components and solutions should be recorded with the accuracy of 0.1 mg. Calibration Solution-A Pipette the following reagents into a 100 ml volumetric flask, containing approximately 60 ml ethanol solution in order to limit losses of components for evaporation; make up to volume with ethanol solution and mix thoroughly. Record the weight of the flask, of each component added and the total final weight of contents. Volume (ml) of methanol 3.0, Propan-1-ol 3.0, 2-methylpropan-OL 3.0, 2-methylbutan-OL 3.0, 3-methylbutan-OL 3.0, ethyl acetate 3.0, 3.0 Butan-1-ol, 3.0 Butan-2-OL, Acetaldehyde 3.0 Acetale 3.0.

D. Analytical procedure

Volatile compounds were analysed using an gas chromatograph equipped with a flame ionization detection (FID) system. And GcMS Determination of volatile compounds by GC is a way to define the characteristics of raw materials and the product emerge after distillation. Most compositions are distinguished in chromatogram are aromatic compounds and flavor. These compounds are significant for the evaluation of requirements defined in Regulation (EEC) No 1576/89 Chromatographic conditions GC-MS, used for the analysis of distillates, are as follows:

- Samples are injected directly after injections of internal standard.
- Chromatogram acquired represent the decoded a few score points, especially after 24 minutes, the unwritable to present organic acids, especially acetic acid and butyric.
- Initially are evident in the base line, specifically in the first few minutes, owing to ethanol and water.
- Quantitative assessment of analitëve are in extract ion, while identification is based on Retention time and by comparison with the total ion spectra of the analyte in the study and the spectrum of pure ingredients (fit threshold> 80%).

GC-FID method is specified by the current European norms REG-CE 2870/2000 and in particular paragraph III.
Methode GC-MS (GC Perkin Elmer Autosystem XL):

-HP-innowax (30m x 0.320mm x 0.50 mm);
-Oven: 40°C (3 min) @ 2.5°C/min to 165°C (0 min) @ 10°C7min to 250°C (10 min);
-Flow: He, 2.70mL/min (4.5 min)@ 0.1mL/min to 1.70mL/min;
-Injector: 250°C, splitless (0.0-1.5min), split 50:1, 0.5 mL injected;
-MS Perkin Elmer Turbomass Gold
-Scan: 33-300 m/z;
-Source: 200°C, Electron Energy: 70eV, solvent delay: 0.9 min;
-Transfer line: 250°C;

The identification of volatile compounds was based on the matching of mass spectra of the compounds with the reference mass spectra of the NIST library. The identification of chromatographic peaks was also confirmed by comparing retention times with those of pure compounds. Quantitative analyses were made by employing the corresponding response factor in the reference solution, according to the internal standard method. Determinations were made in triplicate.

E. Sensory analysis

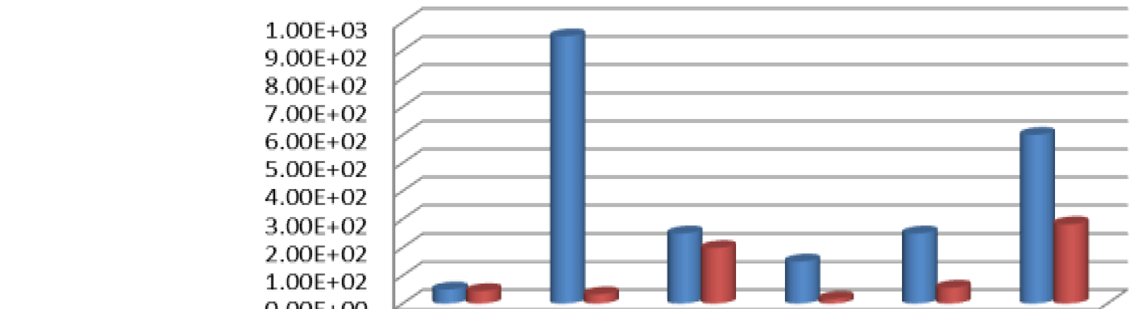
The sensory analysis is based on evaluated the intensity of the descriptive parameters and the qualifying parameters (in visual, aroma, taste, aftertaste and general impression) acording to the evaluation form used in the sensory analysis of the aged grape distillates, all the samples have been tasted in tasting glasses, at room temperature.

The evaluation has been made by using a structured scale (10, no perception; 20, very low; 30, low; 40, middle; 50, high and 60, very high intensity).

III. RESULTS AND/OR DISCUSSION

The results obtained for the chemical composition of the aged grape distillates analysed are shown in Chart 1. The results showed significant differences amongst the samples in the study. The presence and concentration of these parameters was related to the raw materials used, the conditions of fermentation, the distillation technique employed and the aging process.

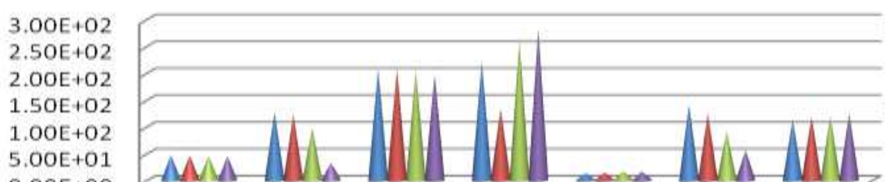
Chart 1. Chemical parameters of 3 yeas aged MH 2009 AL compared with the standard default values of reg. council for aged grape marc distillates.



	Alcohol degree (% v/v)	Methanol (gr/hl a. a	Total acidity (gr/hl a. a)	Acetaldehyde (gr/hl a. a .)	Ethyl acetate (gr/hl a. a)	Higher alcohols (gr/hl a. a)
Maximum standarte	50	950	250	150	250	600
Muschat Hamburg 3 years aged distilate	44.6	33	198	17	56.5	282

As seen from the results given in chart1 the chemical parameters of the 3 years aged Muschat Hamburg distillate (MH 2009 AL) shows that all the parameters are within standarte default values of reg. council for aged grape distillates. Muschat Hamburg (MH 2009 AL) aged distillate is presented with very low levels of methanol 33 g / hl a.a and acetaldehidit 17 g / hl a.a. This indicates the best quality of the distillate aged for 3 years Q.robur.

Chart 2. The compared resultes obtained from GC-FID method for all the samples taken for analysis.



	Alcohol degree (% v/v)	Methanol (g/hl a.a.)	Total acidity (g/hl a.a. Acetic acid)	Higher alcohols (g/hl a.a.)	acetaldehide (g/hl a.a.)	ethyl acetate (g/hl a.a.)	3-methyl butanol (g/hl a.a.)
MH 2009 AL fresh distillate	47.5	128	208	221	13.8	141.1	113.9
MH 2009 AL fresh distillate MH 2009 AL 1 year aged	46.36	123	206	134	15	124.4	119
MH 2009 AL fresh distillate MH 2009 AL 2 years aged	46.14	98	204	260	16.2	90.8	120
MH 2009 AL fresh distillate MH 2009 AL 3 years aged	44.6	33	198	282	17	56.5	125

The alcoholic degree of the samples under study was within the margins established by the corresponding Regulation Council (37.5–50% v/v). The fresh distillate with 47.5% (v/v) is introduced into the oak wood at a high alcohol degree. It have been established that the alcoholic concentration will be 55% (v/v) of ethanol in the distillate, to obtain a greater extraction of compounds from the wood and better quality in the final product (Van Jaarsveld et al.) [6]. During the aging process, there are many changes in the analytical and sensory composition of the spirit, favoured by the high alcohol content [7]. During the aging process oscillations occur in the ethanol content of the distillate, a function of the temperature and humidity outside of the barrel, that is, the higher the outdoor humidity, the greater the loss of alcohol (Catão et al.) [8].

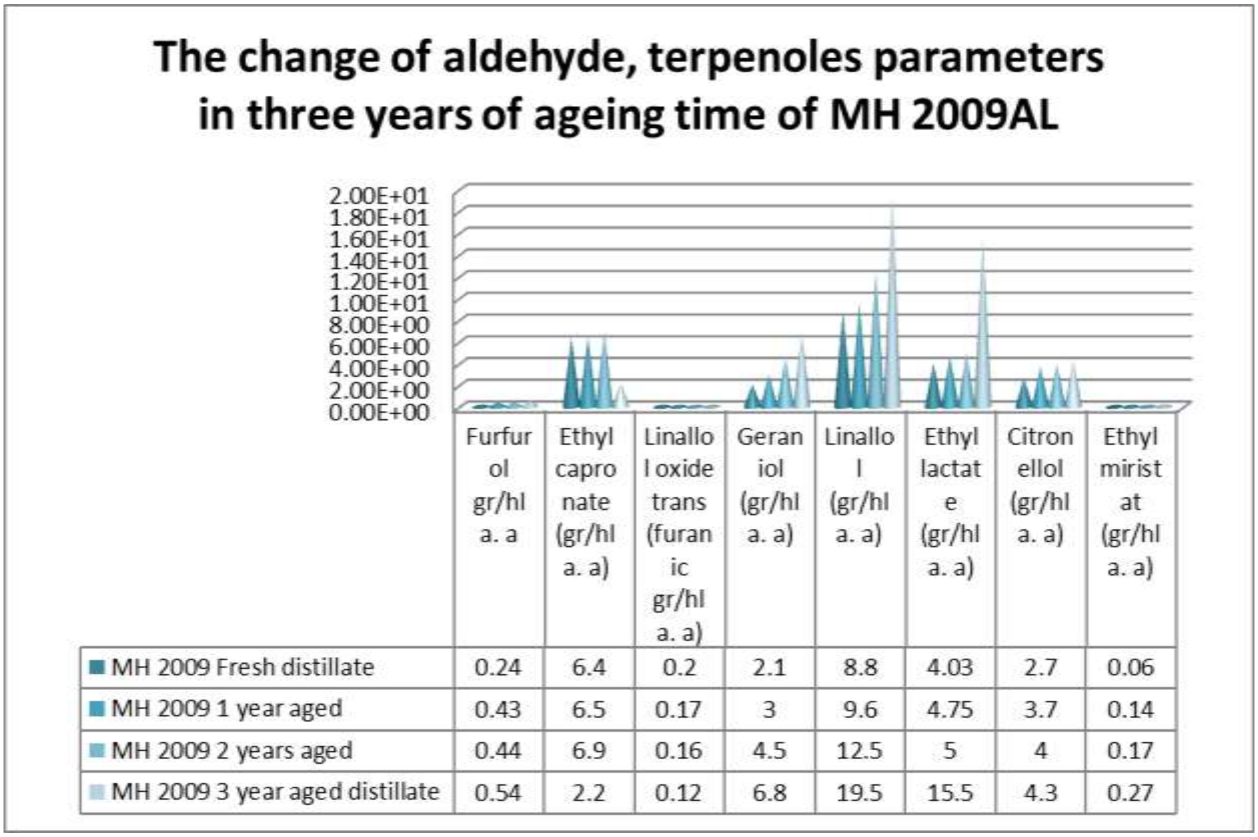
Acetaldehyde is a volatile compound formed during spontaneous or microbial mediated oxidation during the alcoholic fermentation of raw material. Its concentration in the final distillate is also influenced by the distillation system, the wood and the aging time [7]. The acetaldehyde concentration increased in lower levels during aging time it owes to the distillation system of the raw material and when the wood species used was Q. robur. Acetaldehyde in age-distilled spirits increased its concentration nogtably owing to the oxidation process in the barrel. The level of acetaldehyde in the aged Muschat Hamburg (MH 2009 AL) samples ranged between 13.5 to 17 g /hl a.a. It have been reported values of acetaldehyde in whisky of 11.5 g/ hl a.a. (Nascimento et al.) [9] and in other distilled alcoholic beverages (tequila, rum, cognac, grappa and vodka) values of 12.4 g /hl a.a. (Parazzi et al.) [10].
Total acidity values in the samples varied from 208 to 198 g/hl a.a. This parameter decreases significantly during the wood aging process, resulting from oxidation reactions of ethanol and from wood extraction [11].

The methanol content in alcoholic beverages is very important because of its toxicity (maximum legal limit 1000 g/ hl a.a. or 100% vol. ethanol) [12]; however, this compound has no specific odour, and thus does not contribute to the aroma of the distillate. This compound is naturally present in distilled grape marc spirits as a consequence of the enzymatic degradation of pectins [13, 14]. Methanol content in the analysed samples decrease from 128gr/ hl a.a of fresh distillate to 33 g/ hl a.a. in the three years aged distillate, this can be attributed to the low pectin content of the raw material employed in the elaboration of the alcoholic distillate. Methanol did not increase in concentration during the aging process (Parazzi et al.)[10].

Ethyl acetate is the most abundant acetate in the distillates derived from the secondary metabolism of the yeast during the alcoholic fermentation of grape pomace. However, it is the product of acetic acid esterification and thus its concentration increases during the aging process [15]. A high content of ethyl acetate in the distillate, above its perception threshold of 180 g/ hl a.a., has a negative impact on sensorial characteristics and is perceived as having a solvent character [16]. In the samples analysed, the content ranged from 141.1 g/ hl a.a of fresh distillate to 56.5 g/ hl a.a. the three years aged distillate, contributing fruity and floral notes to the aroma of the distillate. Ethyl acetate shows a mean content of 1.75 g/hl in whisky [17], lower owing to the storage conditions of the raw material and the distillation process. For Brazilian sugar cane spirits, the value of ethyl acetate was 62.65 g /hl a.a. after 3 years of aging (Parazzi et al.) [18].

The amount of total higher alcohols in the samples analysed varied from 221 g/hl a.a from fresh distillate to 282 g/hl a.a of three years aged distillate. The concentration of amino acids, the yeast strain, the fermentation conditions (pH, temperature, time) and the distillation process are all important factors in terms of the concentration of higher alcohols in the final distillate. Higher alcohols comprise the group that is quantitatively more important in the distillates. These volatile compounds are positively involved in the sensory quality of the distillate, if they are not present in high concentrations. The content of higher alcohols in the cognac after aging have an increasement as a result of the phenomenon of concentration by ethanol evaporation (Snakkers et al.) [19]. This author reported concentrations of higher alcohols of 37.36g/hl a.a and 38.63 g/hl a.a before and after the aging process, respectively. The increasement of the higher alcohols during aging was primarily due to isoamyl alcohol (Parazzi et al.)[18]. In a study on the chemical composition of Calvados (apple brandy), it has been reported a total higher alcohol content in the range of 435.7 to 1441.07 g/ hl a.a., (Guichard et al.) [20], a value significantly higher than the mean concentration for this group of compounds in the Muschat Hamburg (MH 2009 AL) samples analysed.

Chart 3. The compared resultes of aldehydes and terpenoles obtained from GC-MS method for all the samples taken for analysis.



	Furfurol gr/hl a. a	Ethyl capronate (gr/hl a. a)	Linallol oxide trans (furanic gr/hl a. a)	Geraniol (gr/hl a. a)	Linallol (gr/hl a. a)	Ethyl lactate (gr/hl a. a)	Citronellol (gr/hl a. a)	Ethyl miristat (gr/hl a. a)
MH 2009 Fresh distillate	0.24	6.4	0.2	2.1	8.8	4.03	2.7	0.06
MH 2009 1 year aged	0.43	6.5	0.17	3	9.6	4.75	3.7	0.14
MH 2009 2 years aged	0.44	6.9	0.16	4.5	12.5	5	4	0.17
MH 2009 3 year aged distillate	0.54	2.2	0.12	6.8	19.5	15.5	4.3	0.27

Some compounds very important to the quality of alcoholic distillates which have a direct influence on their aromatic profile are also terpenolet, aldehydes and esters. As seen in chart 3, Furfurol as one of the representatives of the aldehydes that comes as a result of overheating the mass of fermented must grape, is present in the fresh distillate 0,24 gr/hl a.a and in the aged distillate after 3 years is 0,54 gr/hl a.a.

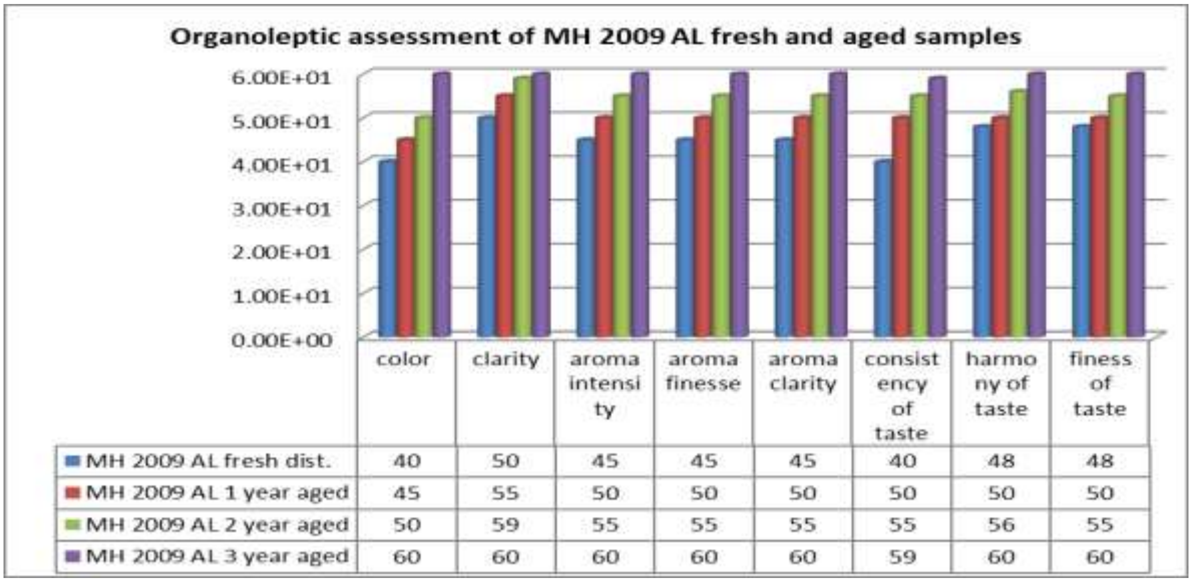
Ethyl capronate show a huge decrease it goes from 6.4 gr/hl a.a of the fresh distillate in 2.2 gr/hl a.a after three years of ageing in Q.robur. Also linallol oxide transfunaric has a little decrease level from 0.2 gr/hl a.a to 0.12 gr/hl a.a after three years of ageing time.

Terpenoles such as linallol, geraniol, citronellol have a huge increasement after three years of ageing time. So linallol goes from 8.8 gr/hl a.a of the fresh distillate in 19.5 gr/hl after three years of ageing time. Geraniol goes from 2.1 gr/hl a.a of the fresh distillate in 6.8 gr/hl after three years of ageing time. Citronellol goes from 2.7 gr/hl a.a of the fresh distillate in 4.3 gr/hl after three years of ageing time. These components are the main responsive for the flower aroma of the distillate.

Ethyl lactate goes from 4.03 gr/hl a.a of the fresh distillate in 15.5 gr/hl after three years of ageing time. ethyl miristate goes from 0.06 gr/hl a.a of the fresh distillate in 0.27 gr/hl after three years of ageing time.

Sensory analysis

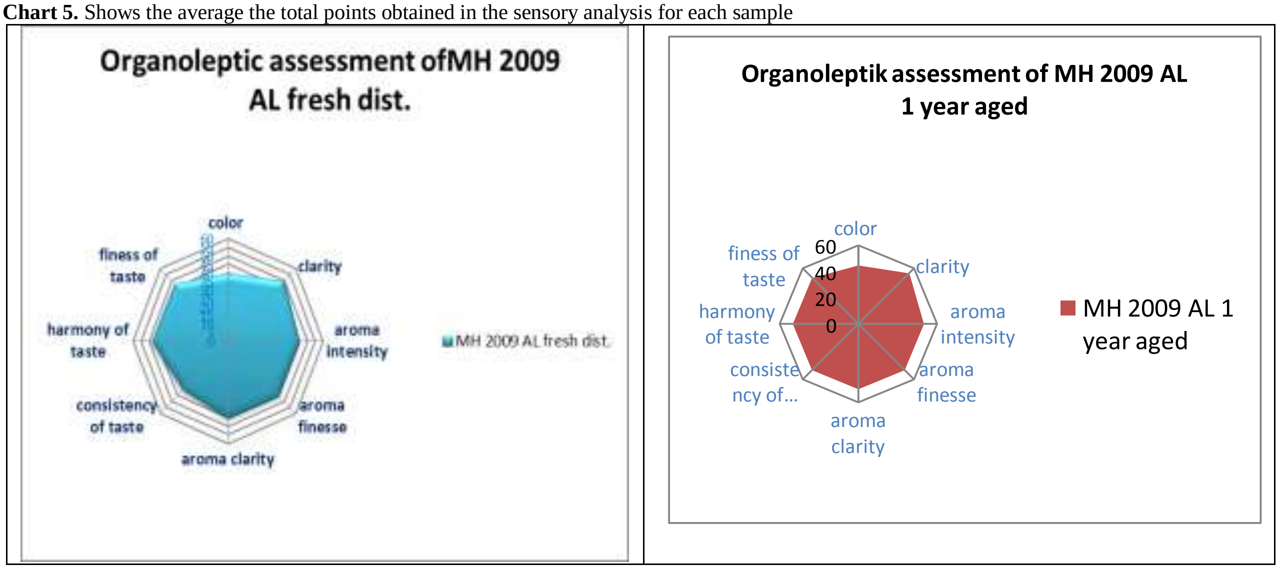
Chart 4. Shows the aroma profile of the fresh and aged samples evaluated according to the qualifying parameters.

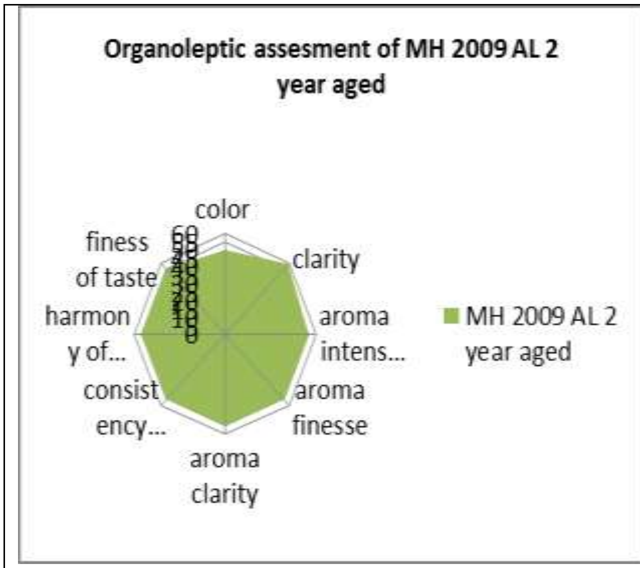


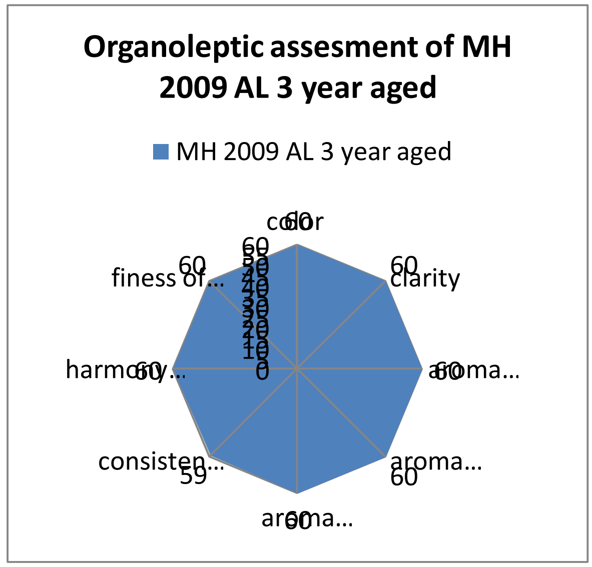
	color	clarity	aroma intensity	aroma finesse	aroma clarity	consistency of taste	harmony of taste	finess of taste
MH 2009 AL fresh dist.	40	50	45	45	45	40	48	48
MH 2009 AL 1 year aged	45	55	50	50	50	50	50	50
MH 2009 AL 2 year aged	50	59	55	55	55	55	56	55
MH 2009 AL 3 year aged	60	60	60	60	60	59	60	60

The results reveal that the samples of MH 2009 AL aged in Quercus robur during 3 years have the best sensory profile and had obtained a greater intensity in all of the qualifying parameters.

Chart 5. Shows the average the total points obtained in the sensory analysis for each sample







IV. Conclusions

By organoleptic evaluation resultes that the alcoholic distillates of Muschate Hamburge MH 2009 AL grape aged in Q. robur for 3 years,shows pronounced aroma flower that is attributable to the variety and the changes of the aromatic compounds under the effect of the ageing process. Some compounds are very important to the quality of alcoholic distillates which have a direct influence on its aromatic profile such as terpenoles, aldehydes and esters. During the aging process, there are many changes in the analytical and sensory composition of the distillate the main change is reported for the volatile compounds such as increasing of geraniol, linallol, citronellol, that are the main responsive for the flower aroma of the distillate. It have been reported increasement also in ethyl lactate, etyl miristate, acetaldehyde, furfurol, the higher alcohols that are positively involved in the sensory quality of the distillate,
The ageing process in the presence of Q. robur is associated with the reduction of the toxicity that was attributed to the presence of methanol in the fresh distillate and decreasing of total acidity resulting from oxidation reactions of ethanol and from wood extraction, Ethyl acetate contributing in fruity and floral notes to the aroma of the distillate.

References

[1] Molina, A.M., J.H. Swiegers, C. Varela, I.S. Pretorius and E. Agosin. (2007) Influence of wine fermentation temperature on the synthesis of yeast-derived volatile aroma compounds. Appl. Microbiol. Biotechnol., 77, 675–687.

[2] Jackson, 2000b Wine science Principals, practice, Percepition, 2nd edition. Academic press. Elsevier New york p.315-et seq.

[3] Pretorius and Lambrechts, 2000 Yeast and its importance in wine aroma. Review S. Afr. J Enol vitic 21, 79, 127

[4] Clemente- jimeney et al.J.M.L Mingorance Cazorla, S. Martinez Rodrigues, F. J.Las Heras- Vazgues-Vico; Influence of sequential yeast mixture of wine fermentatio. Int. J. Food Microbi 98, 301-308.

[5] Marku K., Kongoli, Assessment of quality parametres of alcoholic distillate produced bz aromatic varietz cultivated in Durres – Albania. ICRAE 2012

[6] Van Jaarsveld, F.P., Hattingh, S., Minnaar, P., and Blom, M. (2009) Rapid induction of ageing character in brandy products. Part I. Effects of extraction media and preparation conditions. S. Afr. J. Enol. Vitic., 30(1), 1–15.Web of Science® Times Cited: 4

[7] Rodríguez Madrera, R., Blanco Gomis, D., and Mangas Alonso, J.J. (2003) Influence of distillation system, oak wood type and aging time on volatile compounds of cider brandy. J. Agric. Food Chem., 51(27), 7969–7973.CrossRef,PubMed,CAS,Web of Science® Times Cited: 19

[8] Catão, C.G., Paes, J.B., Gomes, J.P., and Araújo, G.T. (2011) Quality of wood of five forestall species for aging of ‘cachaça’. R. Bras. Eng. Agric. Ambiental, 15, 741–747.

[9] Nascimento, R.F., Marques, J.C., Lima Neto, B.S., Keukeleire, D.D., and Franco, D.W. (1997) Qualitative and quantitative high performance liquid chromatographic analysis of aldehydes in Brazilian sugar cane spirits and other distilled alcoholic beverages. J. Chromatogr. A, 782, 13–23. CrossRef, PubMed, CAS, Web of Science® Times Cited: 47

[10] Parazzi, C., Arthur, C.M., Lopes, J.J., and Borges, M.T. (2008) Determination of the main chemical components in Brazilian sugar cane spirit aged in oak (Quercus sp.) barrels. Cienc. Tecnol. Aliment., 28, 193–199. CrossRef, CAS, Web of Science® Times Cited: 6

[11] Caldeira, I., Anjos, O., Portal, V., Belchior, A.P., and Canas, S. (2010) Sensory and chemical modifications of wine-brandy aged with chestnut and oak wood fragments in comparison to wooden barrel. Anal. Chim. Acta, 660, 43–52. CrossRef,CAS,Web of Science® Times Cited: 5

[12] EEC (1989). Council Regulation 1576/89 on the definition, description and presentation of spirit drinks. Off. J. Eur. Commun., L160, 1–17.

[13] Andraous, J.I., Claus, M.J., Lindemann, D.J., and Berglund, K.A. (2004) Effect of liquefaction enzymes on methanol concentration of distilled fruit spirits. Am. J. Enol. Vitic., 55, 199–201.CAS,Web of Science® Times Cited: 7

[14] Zocca, F., Lomolino, G., Curioni, A., Spettoli, P., and Lante, A. (2007) Detection of pectinmethylesterase activity in presence of methanol during grape pomace storage. Food Chem., 102, 59–65.CrossRef,CAS,Web of Science® Times Cited: 11

[15] Onishi, M.J., Guymon, J.F., and Crowlell, A. (1977) Changes in some volatile constituents of brandy during aging. Am. J. Enol. Vitic., 28, 192–198.

[16] Silva, M.L., and Malcata, F.J. (1998) Relationships between storage conditions of grape pomace and volatile compounds of spirits obtained therefrom. Am. J. Enol. Vitic., 49(1), 56–64.

[17] Caldeira, I., Anjos, O., Portal, V., Belchior, A.P., and Canas, S. (2010) Sensory and chemical modifications of wine-brandy aged with chestnut and oak wood fragments in comparison to wooden barrel. Anal. Chim. Acta, 660, 43–52. CrossRef,CAS,Web of Science® Times Cited: 5

[18] Canas, S., Leandro, M.C., Spranger, M.I., and Belchior, A.P. (1999) Low molecular weight organic compounds of chestnut wood (Castanea sativa L.) and corresponding aged brandies. J. Agric. Food Chem., 47, 5023–5030. CrossRef,PubMed,CAS,Web of Science® Times Cited: 46

[19] Snakkers, G., Boulesteix, J.M., Estréguil, S., Gaschet, J., Lablanquie, O., Faure, A., and Cantagrel, R. (2003) Effect of oak wood heating on cognac spirit matured in new barrel: A pilot study. J. Int. Sci. Vigne Vin, 37(4), 243–255.CAS, Web of Science® Times Cited: 3

[20] Guichard, H., Lemesle, S., Ledauphin, J., Barillier, D., and Picoche, B. (2003) Chemical and sensorial aroma characterization of freshly distilled calvados. 1. Evaluation of quality and defects on the basis of key odorants by olfactometry and sensory analysis. J. Agric. Food Chem., 51, 424–432. CrossRef, PubMed, CAS, Web of Science® Times Cited: 30