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COMPARATIVE BEHAVIOR OF VOLATILE AND AROMATIC COMPOUNDS OF TAMAIOASA ROMANEASCA AND MUSCAT OTTONEL GRAPE MARCS FERMENTED DURING TRADITIONAL STILL DISTILLATION

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Abstract

The demanding requirements of consumers in recent years and the desire of domestic producers to export as much as possible to European Union countries, make issues related to the quality, safety and authenticity of alcoholic beverages increasingly of concern for Romanian producers.
To investigate the behaviour of volatile compounds during a traditional alembic distillation, large numbers of important volatile compounds were identified and quantified by GC/MS analysis in different fractions (one of Head, three of Heart and one of Tail) of grape marc distillates made from two aromatic varieties Tămâioasă românească and Muscat Ottonel. Monoterpenes were confirmed to be responsible for a typical Muscat aroma, as well as for descriptors such as flowery, rose and spicy/menthol in distillates made from Muscat varieties. Due to the abundance of volatile terpene compounds, it has been considered beneficial to use the tail fraction as a raw material for re-distillation.

Key words*: grape marc, distillation fractions, traditional alembic distillation, volatile and aromatic compounds, GC/MS.*

INTRODUCTION

In Romania, the production of grape marc brandy, along with țuica plum distillate by traditional distillation with still, is a very important part of national identity.
Such traditional distilled beverages exist in many Mediterranean countries, such as Grappa in Italy, Tsipourro in Greece, Bagaceira in Portugal, Orujo in Spain, with specificity in terms of sensory properties, chemical composition and manufacturing techniques (Flouros et al., 2003)
Grape marc distillates are highly appreciated, especially after meals (Silva et al., 2000). The aroma of these distilled drinks comes primarily from the grape variety used but also from each successive stage of the manufacturing process: raw material processing, fermentation process, distillation method, and finally maturation (Mangas et al., 1996a).
Considering that grape pomace is the main by-product of the vinification process, a series of specific processes are involved in obtaining distilled pomace drinks that influence both their chemical composition and sensory attributes.
Thus, after pressing the grape pomace and obtaining the wine, the pomace obtained is stored and subjected to alcoholic fermentation. During this process, the most important volatile flavour compounds (alcohols, acids, esters and aldehydes) are synthesized together with the terpene compounds of the grapes used.
In the constitution of the varietal aroma, the terpenic compounds play a very important role being stored in the skin of the grape. Some of them end up in must and come through maceration-fermentation but some remain in pulp (Stoica et al., 2014; 2015)
After the completion of the fermentation of aromatic pomace, the ethanol produced and relatively large amounts of volatile substances as well as terpenes are recovered from the fermented pomace through the distillation process (Silva & Malacata, 1999).
The composition of distillates can be significantly affected by the distillation process (Cortes et al., 2009). Traditional alembic distillation is a discontinuous process that involves separating three major fractions that are distilled consecutively (Leaute, 1990). The first fraction is called the head fraction, the next is known as the middle fraction and the last is the tail fraction. Usually, the first fraction (head) and the last (tail) are eliminated because they contain compounds dangerous to human

health and worsen the sensory quality of the distillate (Stoica et al., 2015). However, they can be redistilled with the next batch of pomace to partially recover the flavours (Da Porto et al., 2010).

Stopping the distillation process in traditional distillation with still without computer control is done by measuring the concentration of alcohol during the process or by tasting. Therefore, the skills and experience of the distiller play an important role (Stoica & Giurgiulescu, 2016).

All these studies related to the raw material, the development of the maceration-fermentation processes but also the distillation represent the research work of several scientists over time (Di Stefano, 1986; Cortez et al., 2003; Geroyiannaki et al., 2007; Lukić et al., 2010; 2011; Stoica et al. 2019; 2020).

The aim of this study was to identify and quantitatively determine the most important volatile compounds in the different fractions of grape marc distillates. The distillation process was the traditional one with the copper still.

Two varieties of Tămâioasă românească and Muscat Ottonel aromatic grapes were used due to the abundance of aromatic compounds, but also due to the different aromatic profiles, highlighting the potential of this raw material - aromatic marc for obtaining natural distillates, of high quality.

MATERIALS AND METHODS

Grape marc samples.

For this study two varieties of aromatic white grapes Tămâioasă românească and Muscat Ottonel were used.

Grape pomace samples were obtained from the 2021 harvest, from a private vineyard plantation. Both varieties were used for the production of distillates, in Romania, especially in the Oltenia region (southwest of the country).

The samples were obtained according to the usual vinification procedures and characteristic of the variety used.

The grape marc from the Tămâioasă românească, an aromatic grape variety, was obtained following standard aromatic wine production practices (grape crushing, sulfitation) and was treated with 5 g/100 kg of pectolytic enzyme preparation. Knowing that terpenes are located in the deep layers of the skin, the mixture of berry skin and pulp of Tămâioasă românească was macerated for 48 hours at 20°C and then pressed. The pH of the marc resulted was 3.8.

The Muscat Ottonel variety was vinified in a similar way with the difference that the maceration period was shorter, 18 hours, as the terpenes are located in the upper layers of the skin. The must was also treated with 2.5 g/ 100 kg of pectolytic enzyme preparation, macerated for 18 hours at 20°C, then pressed. The pH of the resulted marc was 3.4.

Alcoholic fermentation of grape marcs

Grape pomace immediately after maceration was pressed and transferred to wooden containers with a capacity of 100 L, previously cleaned. The vessels were fille up to 70%. The alcoholic fermentation was spontaneous, with indigenous yeasts, at a temperature of 20°C ± 1°C.

Due to the fact that the surface of the pomace is susceptible to oxidation, it was sulphited with a 5% aqueous solution of sulphur dioxide and covered with plastic wrap. To ensure anaerobic fermentation conditions, the plastic foil was sealed by coating with a layer of sand.

The fermentation was carried out over a period of 21 days. Monitoring of the fermentation was performed daily and the fermentation lasted until the sugar concentration dropped to 4°Brix.

Distillation

The fermented grapes marc was distilled in a traditional copper still with shaker and without dephlegmator. Marc was placed up on a copper grate placed on the bottom of the boiler, under which 20 L of water was previously added to prevent the marc from burning.

Before distillation began, the alembic was sealed to prevent any vapor leakage. Heating the alembic was made by direct fire, with natural gas as a heating source. The distillation was induced by strong heating, which was continued for a short time after the condensate distillate began to leak, in order to prevent the discontinuation of the distillation process. During the distillation, the obtained flow rate was kept constant by the gradual increase of the heating temperature, due to a decrease in the ethanol/water ratio in the boiler and in the vapours. The water from the cooling tank was maintained between 20 and 22°C throughout the process.

The alcohol content of the distillates was monitored during the distillation process by an aerometer. The first fraction - head - consisted of the first 200 ml distilled. The next fraction of distillate - heart - was collected until the alcohol content of the operating distillate fell below 30% by volume. This was divided into three 100 ml single samples, marked as heart 1, heart 2 and heart 3. After collecting the heart (middle) fraction, 100 ml portions of the tail fraction were collected, until the alcohol content of the operating distillate decreased to 20% by volume, by the end of the distillation.

Analytical method

The determinations regarding the chemical composition of the distillates were made only on the middle fraction, which is destined to be consumed. The distilled fractions were stored in dark bottles, at 20°C for three months, and then analysed.

All 10 samples (five for each variety) were analysed using gas chromatography and following the method used by the Laboratory of the Department of Horticulture and Food science and the laboratory of National Institute for Cryogenics and Isotopic Technologies (I.C.S.I. Rm. Valcea).

The volatile compounds were extracted from the distillate fractions by liquid-liquid extraction following the method proposed by Lukić et al. (2010). This method consists in the use of a 12 mL volume of a fraction sample, which was diluted with 150 mL of deionised water, and 75 g of ammonium sulphate was added in order to improve the extraction efficiency. Then, a 250 µL aliquot of the internal standard solution (3-octanol, nonanoic acid, and methyl nonanoate in ethanol) was added to control the extraction. Volatile compounds were extracted with three 5 mL portions of dichloromethane. Dichloromethane extracts were combined, dried over anhydrous sodium sulphate, and concentrated to 0.5 mL. To control injection, 10 µL of a 3-heptanol ethanolic solution was added as another internal standard (Lukić et al., 2011).

Quantitative determination of volatile compounds was performed using a gas chromatography system, a VARIAN 450 gas chromatograph GC-FID detector (flame ionization detection) with a set of 275°C temperature for both the column TG-WAXMS 60 m, ID 0.32 mm, film, 0.25 mm, injector temperature 150°C, column temperature 35°C, 3 min stand, temperature increase rate of 20°C/ min., up to 70 to 150°C with 27°C/min., rest 2 minutes, increase to 200°C, rest 2 minutes, increase to 240°C with 20°C/min and rest for another 6 min. The carrier gas was helium (1.2 ml/min flow rate). Injection volume is 1 µL. The identification was made by comparing the retention times of standards from the calibration curve (Stoica et al., 2020).

A professional Cartier/Gay-Lussac 0-100 alcohol meter, metrologically approved and calibrated at 20°C, was used to determine the ethanol concentration (% vol).

Chemicals and standard physicochemical analysis

Standards of volatile aroma compounds were purchased from Merck (Darmstadt, Germany) and Fluka (Buchs, Switzerland). Dichloromethane (99.8%) and sodium sulphate (99%) were supplied by Kemika (Zagreb, Croatia). Pure deionised water was obtained from an Elix 3 purification system (Millipore, Bedford, MA, USA).

RESULTS AND DISCUSSIONS

Volume fraction of ethanol (%), methanol and concentration levels (mg/L anhydrous alcohol, a.a.) of volatile compounds in different fraction of fruit distillate obtained by traditional alembic distillation are presented in Tables 1 and 2.

Ethyl alcohol

Alcohols dominate the group of volatile compounds in grape distillates and have a significant effect on quality and sensory characteristics (Plutowska et al., 2010).

Most alcohols are produced by yeasts from amino acids through specific metabolic pathways, and only small amounts are made by the yeast by reducing the corresponding one aldehyde (Li X. et al., 2012).

In both distillates, the content of ethyl alcohol in the head fraction exceeds the value of 70%, while in the tail, it reaches a much lower value. Distilling further releases vapours that contain low-alcohol substances, but are rich in impurities, thus leading to lower quality products.

Methanol

A similar pattern of methanol distillation was observed both for Tămâioasă românească and Muscat Ottonel grape marc distillates (Tables 1 and 2).

Table 1. Volume fraction of ethanol (%vol) and value of volatile compounds (mg/L anhydrous alcohol) in different fraction of distillate from Tămâioasă românească obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	3480.17	1123.01	823.72	266.91	22.76
Ethanol %vol	77.20	71.15	67.63	58.15	36.20
Higher alcohols					
1-propanol	502.99	404.67	303.21	239.41	111.51
Isoamyl alcohol	2540.46	2571.14	2250.01	1580.36	999.28
Isobutanol	740.25	670.92	558.563	350.14	201.49
1-hexanol	96.83	98.74	93.98	73.56	53.25
2 Phenylethanol	8.49	11.38	16.35	28.61	57.82
1-octanol	60.62	58.22	43.66	38.57	31.23
Benzyl alcohol	0.55	1.16	2.21	3.40	7.80
Esters					
Ethyl acetate	1334.22	1122.54	545.46	162.72	60.58
Isoamyl acetate	3.75	3.20	2.41	1.52	0.51
Isobutyl acetate	2.41	1.51	0.60	0.27	0.20
Monoterpenes					
α-terpineol	33.78.	50.45	66.15	87.15	95.35
Linalool	14.63	20.45	28.35	25.87	23.65
Nerol	6.70	10.98	12.75	15.95	19.31
Geraniol	9.89	9.52	14.32	19.42	28.54
Limonene	0.45	0.60	0.95	0.85	0.50
Aldehydes and ketones					
Acetaldehyde	553.28	458.13	390.35	370.60	439.12
Furfural	n.d.	2.15	4.68	6.65	9.15

Table 2. Volume fraction of ethanol (%vol) and value of volatile compounds (mg/L anhydrous alcohol, a.a.) in different fraction of distillate from Muscat Ottonel obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	2126.17	1204.15	823.71	291.77	29.58
Ethanol (%vol)	72.10	70.50	65.65	55.29	34.80
Higher alcohols					
1-propanol	478.95	450.76	410.25	360.12	282.15
Isoamyl alcohol	1477.33	1505.80	1315.56	1025.78	985.63
1-hexanol	83.32	87.96	66.42	51.39	35.78
2 Phenylethanol	3.99	7.89	12.01	20.12	55.36
1-octanol	55.52	53.23	41.65	36.53	33.69
Benzyl alcohol	0.36	0.98	2.05	3.10	6.25
Esters					
Ethyl acetate	1789.80	1050.15	570.32	188.10	69.75
Isoamyl acetate	5.90	3.89	2.52	1.01	0.44
Isobutyl acetate	1.28	0.82	0.35	0.30	0.26
Monoterpenes					
α-terpineol	5.45	11.27	19.36	32.96	52.63
Linalool	33.86	50.25	69.10	67.40	62.51
Nerol	3.61	8.12	10.25	13.86	15.98
Geraniol	6.68	7.67	8.90	10.30	16.69
Limonene	0.17	0.19	0.38	0.47	0.24
Aldehydes and ketones					
Acetaldehyde	425.15	336.20	360.45	341.03	401.95
Furfural	n.d.	1.82	2.89	5.65	9.68

By distilling a mixture of completely soluble liquids having different boiling points and aiming at a gradual increase in the boiling temperature of the remaining liquid, the mixture can be divided into several fractions. Although methanol is of no importance to the flavour, its occurrence has been widely investigated. Methanol is a characteristic constituent of the head fraction. Initially, the concentration was fairly constant, followed by a significant decrease in the second half of the distillation. This result is in agreement with several previous research observations (Apostolopoulou et al., 2005; Geroyiannaki et al., 2007; Stoica et al., 2020). Other researchers (Silva & Malcata cited by Stoica, 2020) pointed out that methanol may form azeotropic mixtures with ethanol and water. This makes its distillation uneven. This also makes it difficult to remove them by traditional still distillation.
Compared to the Muscat Ottonel distillate, the Tămâioasă românească distillate registers much higher values of methanol, especially in the head fraction.

Higher alcohols

Higher alcohols, compared to methanol, are found in much smaller amounts in all fractions. From the data presented in Tables 1 and 2, it can be seen from a quantitative point of view isoamyl alcohol dominates, being followed by 1-propanol, with higher values in Tămâioasă românească. They are formed by the hydration of the corresponding acetals during the alcoholic fermentation of grape pomace. Benzyl alcohol, which is a rich aromatic alcohol, is present in both distillates, but is found in larger quantities only in the last fraction-tail. A different behaviour of aromatic alcohols was observed during distillation, as some decreases in their concentration during distillation was observed (Plutowska, 2010; Spaho et al, 2013 cited by Stoica et all., 2020).

Esters

Esters are qualitatively the most important class of aromatic compounds in distillates. Esters are formed, like higher alcohols, during the alcoholic fermentation of grape pomace. (Stewart, 2008). Esters are associated with a pleasant, fruity and flower aroma (Stoica et al, 2016).

The positive influence of esters on the aroma of distillates is given by their concentration in the beverage (Cortés et al., 2009; Stoica et al., 2020). Ethyl acetate is the main ester present in distilled alcoholic beverages. The importance of ethyl acetate in distillates, which even in small quantities can confer a pleasant aroma to these drinks, should be noted. However, in too large quantities, it can impart too strong of a flavour, that is not always appreciated by the consumer (Silva et al., 2000; Lukic et al., 2011; Spaho et al., 2013). According to the data in Tables 1 and 2, it can be seen that the values of the analysed esters are higher in the Muscat Ottonel distillate than in the Tămâioasă românească distillate. Also, they present the highest values in the head fraction, after which they start to progressively decrease. The second most abundant ester was isoamyl acetate, in both varieties distillates. Isoamyl ester has also been characterized by a steady decline in concentration with the progration of distillation (Tables 1 and 2). The decrease the most evident in the last fractions. Previous studies (Léauté, 1990; Hernández-Gómez et al.,2005; Lukic et al., 2011) showed that these esters belong to the group of compounds that distillate mainly in the head fraction and in the first fraction of the heart due to low boiling points.

Monoterpenes

According to the data presented in Tables 1 and 2, the behaviour of most monoterpenes during distillation was characterized by an increase in concentration. Final fractions (Heart 3 and Tail), with lower volume fraction of ethanol, which distilled at higher temperatures, contained higher total monoterpenes concentrations. Although due to the good solubility in ethanol and poor water solubility, higher concentrations were expected in the initial fractions, but their relatively high boiling the points were crucial in determining the distillation behaviour (Lukic et al., 2011).
The observed increase was expressed especially in the case of monoterpenes alcohols for which the concentration was almost constant, probably due to their higher polarity, hydrophilicity and boiling point relative to monoterpenes hydrocarbons. This hypothesis was corroborated, on one hand, by the fact that the last fractions of the heart and tail contained

notable amounts of acetates and polyols, which contain several polar parts in their molecular structure. One possible explanation for the increase in concentrations at the end of distillation is that the physical release of terpene compounds from grape seed cells and especially from skins was stimulated by the higher temperatures towards the end of the distillation process. This high content of terpenes compounds in the tail fraction leaves the possibility of its use for further distillation with a new batch of aromatic marc.

Another interesting aspect regarding the aromatic profile of the two distillates refers to the values of two monoterpenes, namely linalool and α-terpineol.

Both monoterpenes are found in distillates. While at the Tămâioasă românească distillate α-terpineol predominates, at Muscat Ottonel the major monoterpene is the linalool. This fact is reflected in the aromatic profile of distillates, which are similar as those of the grapes from which the beverage is prepared.

Other researchers (Ohta et al.1990; Lukic et al., 2011) have also shown that under slightly acidic conditions, such as pH 3.8 at 100° C, similar to the conditions in our study, geraniol and nerol were converted to linalool and α-terpineol in 30 min. It is possible that the conversion of nerol and geraniol into α-terpineol was the reason for which a much higher concentration accumulation rate was observed towards the end of distillation, despite the lower boiling points of the terpenes.

Aroma is one of the main characteristics that determine a brandy's organoleptic quality and style. This is the result of the contribution of hundreds of volatile compounds. They come from volatile chemical compounds resulting from grapes and vinification and distillation process (Stoica, 2008; Stoica et al, 2019).

A certain level of acetic aldehyde was found in all fractions of both aromatic distillates (Tables 1 and 2). The explanation is probably due to the complete solubility of acetaldehyde in both ethanol and water.

This result confirmed the findings of Lukic et al., 2011, but it was not in agreement with other researchers, who observed a significant decrease in concentration during distillation (Apostolopoulou et al, 2005; Leaute, 1990; Prado-Ramírez et al, 2005; Lukic et al, 2011). Slightly higher concentrations observed in the heads and the tails were probably the result of an increase in ethanol oxidation rate under higher temperature conditions, applied in our distillation phases.

The presence of furfural has been characterized by a steady increase in concentration, which has been in line with previous results (Soufleros et al., 2004; Colonna-Ceccaldi, 2008; Stoica et al, 2020). High boiling points and good water solubility have certainly supported such behaviour.

CONCLUSIONS

Based on the results obtained from the compositional and qualitative researches of the two distillates obtained from the aroma marc of Tămâioasă românească and Muscat Ottonel, it can be stated that both are quality drinks with a high degree of naturalness.

This study also showed the possibility of significantly affecting the concentrations of many classes of volatile aromatic compounds in grape marc distillates by choosing the appropriate points of fraction separations during the distillation by traditional alembic. An example would be the higher alcohols and esters that are distilled in the first head and heart fractions.

The use of traditional equipment (alembic) and the process of discontinuous distillation have proven to be quite inefficient in removing significant amounts of major toxic constituents such as methanol and acetaldehyde. Therefore, the removal of the first fraction of distillate, the head is fully justified.

The most important result of this research is the experimental evidence regarding the behaviour of varietal aroma compounds during traditional distillation.

This study showed that the concentration of terpene compounds increased during the distillation process, reaching a maximum in the tail fraction. This shows that this last fraction of distillate is a valuable raw material for re-distillation and enrichment with varietal aroma.

In conclusion, it can be argued that by using aromatic marc a natural and quality distilled beverage is obtained.

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