

## I.2. VOLATILE COMPOUNDS IN DIFFERENT FRACTIONS OF FRUITS DISTILLATE OBTAINED BY TRADITIONAL DISTILLATION

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### Abstract

*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 and GC/FID analysis in different fractions of fruits mash distillates made from two pears varieties, one peach variety and blackberry and cranberry fruits. The purpose of this investigation was to determinate the volatile compounds distribution of higher alcohols, esters, aldehydes and terpenes in some fruits fraction of distillates. Because of the abundance in odoriferous monoterpenes, utilization of the tail fraction as a raw material for re-distillation was considered appropriate.*

**Key words:** distillation fractions, volatile compounds, traditional alembic distillation, fruits distillates.

### INTRODUCTION

In Romania, delicatessen fruit alcoholic drinks are produced from autochthonous fruit berry species such as blackberry and cranberry and also from cultivated species such as pears and peaches. Together, with plum distillate which is a national beverage called *țuică*, these fruit distillates have not been studied and there are not chemical data for many of these. An important feature of fruit distillates is the flavor of the fruit from which it originates. The most important compounds in fruit distillates that influence flavour are higher alcohols and esters along with terpenes. (Lukic et al., 2011; Stoica et al., 2015).

The final flavour of fruit distillates consists of several chemical compounds that are formed in different stages of the process. Thus, there are 4 phases: primary flavour (during fruits ripening), secondary flavour (during the fermentation process of the marc), tertiary flavour (during the distillation process of the marc) and quaternary flavour (after the maturation of the distillate) (Leaute, 1990; Spaho et al., 2013).

After the fermentation of fruits mash is finished, the distillation process follows, when alcohol and important amounts of volatile

compounds are recovered. (Lukic et al., 2011; Stoica et al., 2015).

The distillation technique can significantly influence the behaviour of the volatiles compounds during distillation and therefore the chemical composition and the sensorial properties of fruit distillates (Cabaroglu and Yilmaztekin, 2011; Colonna-Ceccaldi, 2008; Tesević et al., 2005).

The traditional distillation, in comparison with the modern distillation with rectification columns, is performed with a copper pot stills called alembics (Leaute, 1990). It is a discontinuous process involving a simple separation of three important fractions which distil consecutively: the first part of the distillate (the head fraction), the middle part of the distillate (the heart) and the last part of the distillate (tail fraction).

The first (head) and the last (tail) fraction of the distillate, are usually eliminated because it contributes negatively to the quality of the distillate by certain volatile compounds found in important quantities in these fractions. (Silva et al., 2000; Prado-Ramírez et al., 2005).

The heart fraction, which contains the ethanol and the volatile compounds that add the sensory quality of the distillate, is the only one intended for consuming.

In traditional alembic distillation without computerized control, the distillation cut is determined by measuring the alcohol concentration during the process or by tasting. Therefore, the skills and the experience of the distiller have an important role (González et al., 2010; Cichi, 2015).

In the literature there are some studies which give information on the behaviour of the main volatile chemical compounds (higher alcohols, esters and terpens) during distillation (Apostolopoulou et al., 2005; Cortés-Diéguéz et al., 2003; Cortés et al., 2009; Crowell and Guymon, 1973; Flouros et al., 2003; Geroyiannaki et al., 2007; Hernández-Gómez et al., 2005, 2008; Nykänen, 1986; Plutowska et al., 2010; Santos et al., 2013; Soufleros et al., 2004).

The quality of distillates depends decisively on the quality of fruit mash and fresh fruit quality used by default (Durr and Tanner, 1983).

The aim of this investigation was to determinate the chemical composition of a fermented pear mash and peach mash correlated on the fruit variety and distillation with a traditional alembic. Since, these fruit alcoholic drinks are delicatessen in Romania, this study proposes a research of distillates obtained from the peach, blackberry, cranberry and pears varieties used for this purpose.

## MATERIALS AND METHODS

### *Fruit samples*

Two pear varieties and each one variety of peaches, blackberries and cranberry were used for this study. Each variety was used for production of the fruit distillate, in Romania, especially in Oltenia region (South-West of the country).

The raw material was as follows: Williams and Favorita lui Clapp which are summer pears varieties depending on the maturation period without sclereids. The peach variety is Babygold 7, while blackberry and cranberry are wild fruits.

### *Fermentation*

Fruits harvested from hilly areas of Oltenia (Dragasani, Valcea), were transported to the Faculty of Horticulture Craiova, Department of Horticulture and Food science laboratory. All three varieties were crushed and put into

vessels with 40 L capacity each. The vessels were filled up approximate 70% of the volume. Spontaneous fermentation with indigenous yeasts has been done at 20°C ± 1°C. Monitoring of fermentation was followed every day and it lasted until the concentration of sugar decreased to 4°Brix.

### *Distillation*

Fermented fruit mashes were distilled in a traditional copper alembic with stirrer and without dephlegmator. The alembic was hermetically sealed. This is necessary before starting distillation to prevent an escape of vapour and even an explosion.

Gradual warming of the alembic was carried out by direct fire. The distillation process was induced by strong heating, which was continued for a short time after the condensation of the distillate started.

During distillation, attained flow was maintained constant by gradually increasing the heating temperature, due to a decrease in ethanol/water ratio in the boiler and in the vapours. The flow rate obtained during the distillation process was constantly maintained by the gradual increase of the heating temperatures and the water temperature in the cooling vessel was 20-22°C throughout the distillation. A thermometer was used to measure the temperature in the pot still and in the spirits tube and the alcohol content of distillate was measured by an aerometer.

### *Analytical method*

The first 200 ml of distillate formed the head fraction. The next fraction of distillate, collected up to an alcoholic concentration of up to 30% vol, was the heart fraction. This was divided into three 100 ml single samples, marked as heart 1, heart 2 and heart 3. The last fraction of distillate collected - the tail fraction was 200 ml at a level of alcoholic concentration of 20% vol. Thus, there were obtained the 5 main fractions: one head, one tail and three heart fractions.

All distillates were stored in dark bottles at 20°C for three months and then analyzed. All 25 samples (five for each variety) were analyzed 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 consist in 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, climb to 20°C/min, up to 70 to 150°C with 27°/min, stand 2 minutes, climb 200°C, stand 2 minutes, climb to 240°C with 20°C/min and stands 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.

#### ***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 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, 2, 3, 4 and 5.

#### ***Methanol***

Methanol had the same pattern of distillation for all five varieties of fruits distillate (Tables 1, 2, 3, 4 and 5). In the head fraction, the methanol concentration has been increasing followed by a decrease to tail fraction which has the lowest value. Researchers Silava and Macata (2000), have shown that methanol can form azeotropic mixtures with ethanol and water, also being present in the other distillate fractions. Also, this makes it even more difficult to remove it in the case of discontinuous distillation with alambic. Other researchers (Geroyiannaki et al., 2007; Hernández-Gómez et al., 2008) found approximately equal concentrations of methanol throughout the distillation.

The content of the higher alcohols and esters in the 25 samples of the fruits distillates are presented in Tables 1, 2, 3, 4 and 5.

From a quantitative point of view, higher alcohols present the broadest group of aromatic compounds in the distillates (Lehtonen and Eriksson, 1983; Versini et al., 2012; Spaho et al., 2013). Due to lower boiling point (below 200°C), higher alcohols are distilled in the distilled head and heart fractions.

However, a non-uniform behaviour of distillation of higher alcohols has been observed. (Apostolopoulou et al., 2005; González et al., 2010).

In this study, 3-methyl-1-butanol (isoamyl alcohol) and 1-propanol were quantified as higher alcohols (Onishi et al., 1977; Cortés et al., 2009; Cabaroglu et al., 2011). These alcohols are produced by yeasts during alcoholic fermentation by converting the amino acids (leucine, isoleucine, valine) that are in this medium (Garcia-Llobodanin et al., 2007).

The highest content of methanol in the head fraction is recorded in the Favorita lui Clapp distillate and the lowest in the blackberry distillate. In all 5 distillates, Heart 1 will notice a greater amount of methanol, which might be the answer to the somewhat empirical separation of the three fractions during distillation.

Compared with methanol, higher alcohols are found in much lower amounts in all fractions, including the head fraction. From the data

presented in the tables, it can be seen from the quantitative point of view that 3-methyl 1-butanol is dominated followed by 1-propanol. It is formed by hydration of the corresponding acetals during the alcoholic fermentation of the fruit jar. Benzyl alcohol which is a high aromatic alcohol has shown somewhat non-uniform behaviour but it is found in most cases in larger quantities in the last two distillate fractions. A different behaviour of aromatic alcohols during distillation was observed by Plutowska (2010), as well as by some decreases in their concentration throughout distillation (Spaho et al., 2013).

#### **Esters**

From a qualitative point of view, the esters represent the major representative class of flavour compounds in distillates. As well as the higher alcohols, they are formed during alcoholic fermentation of fruit mash. (Lehtonen and Eriksson, 1983; Stewart, 2008). Ethyl acetate is the major ester present in alcoholic beverages. Esters, generally, are associated with a pleasant, fruity and flowery aroma (Stoica et al., 2016).

The positive influence of esters on the aroma of distillates is given by their concentration in the beverage (Onishi et al., 1977; Cortés et al., 2009). According to the data in the tables, ethyl acetate is found in significantly higher amounts comparatively with isoamyl acetate. It is worth mentioning the importance of ethyl acetate in distillates, which even in small quantities can impress a pleasant aroma of these beverages. However, in too large quantities it can print a too strong flavour that it is not always appreciated by the consumer (Silva et al., 2000; Lukic et al., 2011; Spaho et al., 2013). The concentration of ethyl acetate was significantly higher in the distillates from the pears and peach varieties than in distillates from the other two fruits varieties (blackberry and cranberries).

The second most abundant ester was isoamyl acetate. The concentration of isoamyl acetate was significantly higher in the distillates from the blackberry and cranberries varieties than in distillates from the other two fruits varieties (pears and peach). It appears that these esters could be the main predictors of the sensory profile of a brandy made from the wild fruits (blackberries and cranberries).

#### **Monoterpenes**

From the data presented in the tables (Cortés-Diéguez et al., 2003; Colonna-Ceccaldi et al., 2008; Cabaroglu et al., 2011; Cichi, 2015), the concentration of most identified terpenes decreased in fractions having a lower alcoholic strength, which is distilled at higher temperatures. The explanation would be that they are extremely volatile compounds and may be lost during distillation. The main analysed monoterpenes nerol, geraniol, limonene, are found in different amounts in pears and berries distillates. In contrast, in peaches distillates they were not identified. 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, including higher alcohols, esters, acids, aldehydes, ketones, terpenes, norisoprenoids and phenols. They come from volatile chemical compounds resulting from grapes and vinification and distillation process; some are derived from oak (Lukic et al., 2011). Undoubtedly the alcoholic fermentation significantly influences the flavour and final quality of the brandy (Durr and Tanner, 1983; Nykänen, 1986).

#### **Aldehydes**

Concerning the concentration in acetic aldehyde, this it is found in all the fractions of the 5 analysed distillates in larger or smaller quantities. An explanation for this could be their complete solubility in both water and ethanol.

This result confirmed Geroyiannaki's conclusions (2007). Other authors (Crowell and Guymon, 1973; Lehtonen and Eriksson, 1983; Lukic et al., 2011) have found a decrease in the concentration of acetic aldehyde during the distillation process. Regarding the acetic aldehyde concentration in the fractions of the 5 distillates, it is found that in 4 of them (the pears, peach and blackberries distillates) is present in a larger quantity in the tail fraction. Exception makes the cranberry distillate where acetaldehyde is predominant in the head fraction. According to previous research (Garcia-Llobodanin et al., 2007; Colonna-Ceccaldi, 2008), the data in the tables confirm an increase in furfural content from head to tail, to all the analysed distillates.

This behavior is explained by the good solubility of this compound in water. Furfural is formed by Maillard reactions during the distillation process (Hernández-Gómez et al., 2008) and depends on the chemical composition of different fruits.

Distillation patterns of furfural were characterized by a constant increase in

concentration, which was in accordance with previous results (Soufleros et al., 2004; Garcia-Llobodanin et al., 2007; Colonna-Ceccaldi, 2008). High boiling points and good solubility in water certainly supported such behaviour. Furfural is formed during distillation by Maillard reactions (Hernández-Gómez et al., 2008).

Table 1. Values of volatile compounds (mg/L a.a.) in different fraction of Williams pear distillate obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	987.25	4261.86	1212.98	1196.60	739.69
Higher alcohols					
1-propanol	140.36	100.98	78.33	71.38	2.82
3-Methyl-1-butanol	997.06	862.09	573.06	396.61	119.18
1-hexanol	62.10	43.62	30.37	19.69	28.92
1-heptanol	3.22	4.02	3.33	1.30	0.52
1-octanol	2.82	2.77	18.77	3.13	3.54
Benzyl alcohol	-	22.02	0.95	1.99	1.22
Esters					
Ethyl acetate	429.11	305.06	844.47	144.37	60.90
Isoamyl acetate	1.23	1.36	5.12	3.02	4.12
Monoterpenes					
Nerol	-	8.22	2.14	2.60	1.00
Geraniol	3.66	-	-	-	-
Limonene	40.13	-	4.25	3.11	0.61
Aldehydes and ketones					
Acetaldehyde	769.15	513.25	619.03	654.12	1.155.02
Furfural	-	131.74	256.01	713.40	639.67

Table 2. Values of volatile compounds (mg/L a.a.) in different fraction of Favorita lui Clapp pear distillate obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	1364.62	8945.42	1112.54	1012.45	834.24
Higher alcohols					
1-propanol	75.99	75.22	48.95	31.43	20.83
3-Methyl-1-butanol	745.93	521.71	481.93	306.15	169.17
1-hexanol	43.80	37.39	27.96	19.47	11.53
1-heptanol	1.00	5.02	0.25	2.33	4.02
1-octanol	3.87	3.51	29.80	3.31	39.32
Benzyl alcohol	0.94	1.11	-	0.98	0.15
Esters					
Ethyl acetate	1408.98	264.95	137.69	83.90	749.22
Isoamyl acetate	5.72	-	2.58	0.89	0.25
Monoterpenes					
Nerol	1.01	-	-	1.00	0.25
Geraniol	-	-	-	-	-
Limonene	0.60	2.22	-	0.65	0.11
Aldehydes and ketones					
Acetaldehyde	714.00	1087.02	912.02	1087.02	1002.25
Furfural	117.98	1066.43	512.11	512.33	315.45

Table 3. Values of volatile compounds (mg/L a.a.) in different fraction of Babygold 7peach distillate obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	1029.56	1131.30	777.79	719.65	315.00
Higher alcohols					
1-propanol	131.98	165.66	133.33	139.87	111.82
3-Methyl-1-butanol	119.65	136.31	92.12	86.79	47.87
1-hexanol	41.97	40.08	32.98	33.17	38.48
1-heptanol	8.55	4.47	-	3.25	1.25
1-octanol	5.27	6.60	4.36	4.02	5.08
Benzyl alcohol	13.98	19.05	17.30	14.76	18.85
Esters					
Ethyl acetate	884.22	108.62	258.69	313.17	67.76
Isoamyl acetate	1.00	0.8715	0.47	6.36	2.15
Monoterpenes					
Nerol	-	-	-	-	-
Geraniol	-	-	-	-	-
Limonene	-	-	-	-	-
Aldehydes and ketones					
Acetaldehyde	959.69	487.23	1081.36	973.22	713.25
Furfural	1.90	1.90	2.15	3.88	6.41

Table 4. Values of volatile compounds (mg/L a.a.) in different fraction of blackberry distillate obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	736.89	5229.18	987.56	733.96	346.08
Higher alcohols					
1-propanol	239.18	253.39	207.96	184.78	112.91
3-Methyl-1-butanol	1213.13	1421.01	1204.17	749.34	576.95
1-hexanol	15.79	17.43	14.28	13.42	10.33
1-heptanol	1.02	11.05	1.25	2.36	5.11
1-octanol	6.13	6.38	4.50	4.17	2.95
Benzyl alcohol	1.24	-	0.61	0.97	-
Esters					
Ethyl acetate	274.91	306.33	1456.54	125.45	1236.16
Isoamyl acetate	3.25	6.13	-	1.22	0.15
Monoterpenes					
Nerol	495.04	-	-	-	-
Geraniol	15.59	14.16	7.26	7.65	1.31
Limonene	1.01	1.54	-	1.21	-
Aldehydes and ketones					
Acetaldehyde	519.22	858.22	617.22	1235.13	881.25
Furfural	-	449.51	417.60	200.65	215.02

Table 5. Values of volatile compounds (mg/L a.a.) in different fraction of cranberry distillate obtained by traditional alembic distillation

Volatile aroma compound	Distillate fraction				
	Head	Heart 1	Heart 2	Heart 3	Tail
Methanol	1.07348	1295.32	673.73	445.42	52.34
Higher alcohols					
1-propanol	66.05	79.76	641.83	614.19	197.88
3-Methyl-1-butanol	745.80	344.58	987.25	456.12	262.78
1-hexanol	64.67	80.84	63.57	61.39	34.27
1-heptanol	5.44	3.02	4.25	4.25	12.15
1-octanol	3.11	2.01	2.74	2.33	3.89
Benzyl alcohol	1.29	1.37	1.18	1.77	2.09
Esters					
Ethyl acetate	479.97	553.41	565.22	348.37	110.42
Isoamyl acetate	4.55	12.25	7.52	1.25	-
Monoterpenes					
Nerol	1.73	1.51	1.64	1.41	1.23
Geraniol	4.44	5.29	3.74	3.00	-
Limonene	9.58	8.91	7.28	5.96	-
Aldehydes and ketones					
Acetaldehyde	1027.22	615.45	835.14	1008.44	875.00
Furfural	23.93	32.57	31.01	30.68	37.61

## CONCLUSIONS

The results obtained from this study show that:

- choosing fractions separating moments during a discontinuous distillation with alembic can significantly influence the concentrations of volatile compounds in fruit distillates;

- higher alcohols and esters are volatile substances with a positive impact on the sensory quality of distillates that are influenced by fraction separation;

- traditional equipment and discontinuous distillation techniques proved to be insufficient to remove as efficiently toxic compounds as methanol and acetic aldehyde;

- in this case, it is necessary to store the fruit mash more carefully, but also to control its alcoholic fermentation.

The most important novelty of this research may be the experimental evidence for the behavior of aromatic varieties during traditional distillation. It has been shown that monoterpenes concentrations increased during distillation and reached the maximum point in the tail fraction. This makes possible the re-distillation of this fraction as a precious raw material and the enrichment with a variety of flavours.

In conclusion, it should be noted that certain deviations from the observed distillation process may be influenced by the conditions of

production (type and variety of fruits, consistency and composition of the fruit mass).

## ACKNOWLEDGEMENTS

This work was partially supported by the grant number 14C/2014, awarded in the internal grant competition of the University of Craiova.

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