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VOLATILE COMPONENTS OF STRAWBERRY JAM

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ABSTRACT
Strawberry jams of such cultivars as "Ducat", "Honey" and "Polka" were studied to define the content of aromatic volatiles using the methods of highly efficient liquid chromatography. Volatiles contain a considerable amount of acids (65.6-76.8%), a small amount of furanone (8.3-14.6%) and that of aldehydes (3.4-10.8%). The share of esters in jams exceeds 0.7-3.1% of the total volatile amount. Typical compounds for strawberry jam flavor are hexanoic (caproic) acid, hexadecanoic acid, 2-ethyl hexanoic (capronic) acid, trans-cinnamic acid, linoleic acid, furil hydroxy methylketone, 2,5-dimethyl-4-methoxy-3(2H)-furanone (mesifurane), furfural, 5-hydroxymethylfurfural, vanillin. As to aroma activity furanone derivatives dominate: 2.4-dioxy-2.5-dimethyl-3(2H)-furan-3-one, 2.5-dimethyl-4-methoxy-3(2H)-furanone (mesifurane) та 2.5-dimethyl-4-hydroxy-3(2H)-furanone (furaneol); they add sweet caramel scents to jams. For strawberry jams of cultivar "Polka", ethyl 2-methylbutanoate, decanal are active components which add fruity and grassy scents, strawberry jam made of cultivar "Ducat" – 2-decenal and γ- decalactone, 'Honey' - γ- decalactone (fruity, sweet) and linalool (sweet and floral scent).

1.Introduction

Garden strawberry is the most popular and valuable berry crop due to its high flavoring characteristics, fast and early ripening, not-demanding to growing conditions, high yielding capacity and high economic efficiency of growing. Strawberries are rich in sugars, organic acids, vitamins, phenol compounds, mineral substances, they have well-expressed typical flavor; they are in constant demand among consumers due to their high gustatory properties (Markovskyi *et al.*, 2008). Strawberries are consumed both as fresh and as juices, drinks, wines, puree, stewed fruits, jams (Amaro *et al.*, 2012).

Strawberry flavor is a combination of esters, aldehydes, ketones, ethyl, lactones, terpenic compounds, furanones (Larsen *et al.*, 1992; Larsen *et al.*, 1992, Forney *et al.*, 2000, Kafkas *et al.*, 2005). Esters are the main components (25-90% of the total amount), aldehydes and furanones – 50% (Larsen *et al.*, 1992; Larsen *et al.*, 1992). The latter add fruity and floral green and sweet or caramel flavors to strawberries (Pérez *et al*, 1996; Jetti, 2005; Kim *et al.*, 2013). Strawberry taste develops during ripening and it changes during storage (Zabetakis *et al.*, 1997; Forney, 2000) and processing for canned products (Lambert, *et al.* 1999).

One of the most popular processing products made of strawberry is jam which is due to high organoleptic properties, availability for consumers and long shelf-life of the product. To get strawberry jam with well-felt strawberry flavor it is advisable to use the most fragrant berries (Lesschaeve *et al.*, 1991; Suutarinen *et al.*, 2002).

The main volatile compounds of strawberry jams belong to the classes of acids, alcohols and esters (Barron D. *et al.*, 1990; Kimura *et al.*, 1994.); they have natural origin and can be formed as a result of heat treatment (Sloan *et al.*, 1969). Thus, due to high temperatures of the treatment, changing sugar into caramel and Maiyar reaction, the product gets boiled, burned and caramel taste (Avasoo *et al.*, 2011), whereas green and fruity flavor, typical for fresh berries, becomes less expressed (Ozcan *et al.*, 2011). High furanol concentrations add typical caramel and sweet flavors to strawberry jams (Lesschaeve *et al.*, 1991; Pérez *et al.*, 1996).

However, not enough information is available in scientific literature concerning the content and composition of volatiles of jam made of strawberries of some pomological cultivars.

The purpose of our research was to identify the content and composition of volatiles of jam made of strawberry cultivars Ducat, Honey and Polka.

2. Materials and methods

2.1. Materials

The work was done in 2013-2014 with the berries of varieties "Ducat", "Honey", "Polka" in the laboratory of the department of the technology of storage and processing of fruits and vegetables at Uman national university of horticulture and at the experimental center of foodstuff quality control at the National institute of grape and wine "Magarach" (Ukraine).

2.2. Technological process

Strawberries were harvested at a technical stage of ripening, sorted by quality, cleaned and washed. Jams were made of the prepared berries according to current technological instruction (1992) adding pectin in concentration of 0.3%. Jam was boiled until the content of dry soluble substances reached 62%, packed in glass jars (250 cm^3). Jam was made of each pomological cultivar in five replications. The product was kept for six months at 20 °C.

2.3. Main instruments and equipment

Agilent Technologies 6890 chromatograph with mass-spectrometric detector 5973 and chromatographic capillary column DB-5 - internal diameter 0.25 mm and length 30 м – was used to identify volatiles of finished jam.

2.4. HPLC analysis on volatile components

A sample (0.75 g) was put in a 2-ml vial, and internal standard was added. Trydekan (50 mkg per sample) was used as an internal standard. To extract volatile substances, 0.5 ml of chloride methylene was added, the exposure time was 24 hours. The vial was carefully shaken several times. The extract received was taken with a micro-syringe, put into a 2-ml vial and digested in the flow of specifically clean nitrogen to the volume of 50 mkl. The concentrate was chromatographed.

A sample was injected into a chromatographic column in splitless mode, i.e., the flow was not divided; this allowed to eliminate losses and to increase the sensitivity of chromatography method considerably (10-20 times). The speed of the sample injection was 1.2 ml/min, and it took 0.2 min.

To identify the components, a library of mass spectrums NIST05 and WILEY 2007 with the total number of spectrums more than 470000 in combination with identification programs AMDIS i NIST was used.

The method of internal standard was used for quantitative calculations.

The calculation of the component content was made using the equation where:

$$C = K_1 \times K_2$$

C – volatile component content, mg/kg,

$$K_1 = \frac{\Pi_1}{\Pi_2}$$

П1 – peak area of the substance studied, П2 – peak area of the standard;

$$K_2 = \frac{50}{M}$$

50 – mass of the internal standard (mkg), introduced into a sample, M – a sample (g).

2.5. Statistical Analysis

Statistic analysis was made using StatSoft STATISTICA 6.1.478 Russian, Enterprise Single User (2007).

3.Results and discussions

38 components were identified in volatile concentration in strawberry jams made of the varieties studied: esters, aldehydes, ketones, furanones, acids, aroma compounds, lactones, terpenic compounds. The concentration of volatiles in strawberry jams was 12.1-33.54 mg/kg depending on the variety (Table 1). The most meaningful shares are: acids – 65.6-76.8%, furanes – 8.3-14.6% and aldehydes – 3.4-10.8% (Fig.1). The share of esters in jams exceeds 0.7-3.1% of the total volatile content. It is important to mention that in strawberry jams made of Polka cultivar the share of esters and aldehydes is much higher: 3.1 and 10.8%, that of furanes and acids, on the contrary, is the lowest – 8.3 and 65.6% which proves strong expression of scents typical for fresh strawberries.

Characteristic compounds for strawberry jam flavor made of the studied cultivars are hexanoic (caproic) acid (0.84-6.89 mg/kg), which is 6.9-22.9 % of the total volatile amount depending on their quantity for each cultivar, hexadecanoic acid (2.5-12.4%), 2- ethyl hexanoic (capronic) acid (3.1-10.7%), trans-cinnamic acid (17.5-25.3%), linoleic acid (0.3-7.2%), furil hydroxy methylketone (3.1-6.0%), 2.5-dimethyl-4-methoxy-3(2H)-furanone (mesifurane) (7.4-13.7%), furfural (0.8-3.1%), 5-hydroxymethylfurfural (0.8-5.2%), vanillin (0.2-0.8%).

The availability of furfural (0.8-3.1%), 5-hydroxymethylfurfural (0.8-5.2%) 5-methylfurfural (0.7%) in strawberry jams indicates non-fermentative darkening during thermal treatment (Barren *et al.*, 1990; Kimura *et al.*, 1994).

High content of 2-heptenal – 0.9% of the total volatile content and hexanol (1.9%) were found in strawberry jam made of Polka cultivar, and as to cultivars “Ducat” and “Honey” – high content of 2-methylbutyric acid (6.3-6.6%) γ, and 2.5-dimethyl-3(2H)-furanone (0.4-1.1%). According to Schwab (2013) 2.5-dimethyl-3 (2H)-furanones is synthesized via sets of fermentative changes in fruits.

γ-decalactone – 0.55 and 1.73 mg/kg which is 1.8-5.2% – was found in strawberry jams (cultivars “Ducat” and “Honey”). This volatile compound adds “fruity”, “sweet” and “peachy” scents (Ulrich *et al.*, 2007).

Small amounts of 2H -pyran-2.6(3H)-dion and 3.5-hydroxy-2-dimethyl-4H- pyran-4 – (0.09-0.66 mg/kg) which, depending on the cultivar, is 0.5-0.9% of the total volatile content in jams, were found; and according to (Barren *et al.*, 1990) they are the products of Maiyar reaction resulted from the reaction of glucose with glutamic acid, glycine, butylamine, lysine, hydroxypyroline and/or phenylalanine (amino acids).

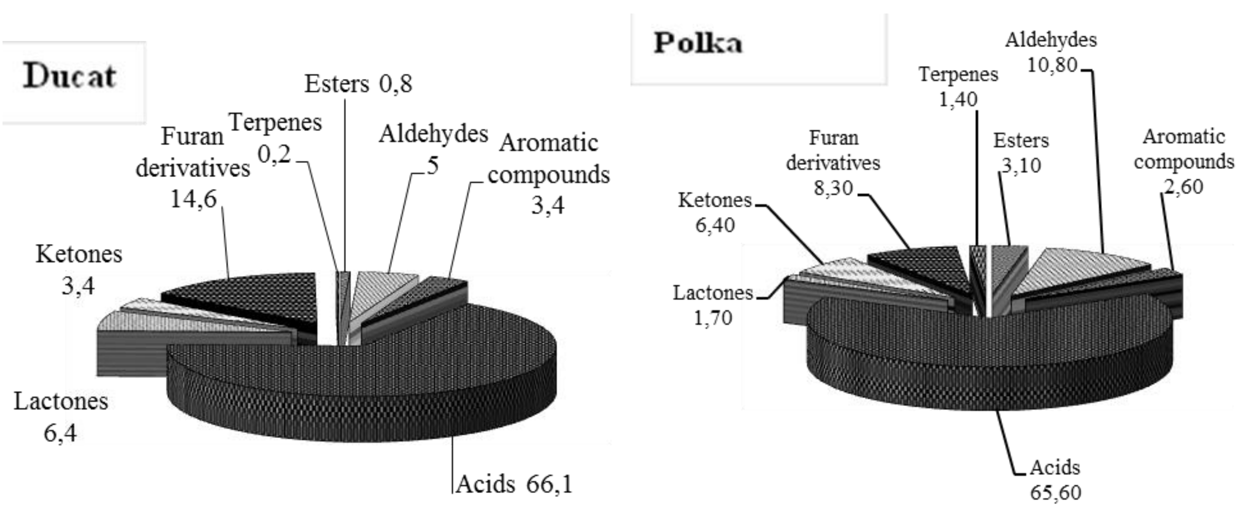
Terpenic compounds of strawberry jams are presented by small amounts of limonene (0.1 mg/kg, which is 0.8%) and α-terpineol (0.2-1.3%), they were found in fresh berries by Bianchi *et al.* (2014); these compounds add aromatic scent to fresh berries (Ulrich *et al.*, 2007, Bianchi *et al.*, 2014); oxyde bisabolol A (0.1-0.3%), trans - linalool oxyde (0.1-0.3%), cis - linalool oxyde (0.3%), however no data concerning their presence in fresh strawberries is available. Despite a large volatile amount, strawberry jam flavor is developed under the effect of their small amount.

To determine the share of each compound in the flavor, its activity is defined by dividing substance concentration on its threshold concentration (OAV = concentration (ppbv)/threshold value (ppbv) (Table 2) (Rothe *et al.*, 1963, Kim *et al.*, 2013). If the result of OAV exceeds 1, it proves the contribution of a component to the flavor.

Table 1. Content of volatile components in strawberry jam, mg/kg

Volatiles	Variety		
	Polka	Ducat	Honey
Esters			
Methyl butanoate	0.14	0.03	-
Ethyl butanoate	0.06	0.15	0.09
Ethyl crotonate	0.02	-	-
Ethyl 2-methyl butanoate	0.02	-	-
Ethyl capronate	0.01	-	-
3,4-dihydropyran	0.13	0.10	0.12
Total esters	0.38	0.28	0.21
Aldehydes			
Benzaldehyde	0.04	0.10	0.14
Trans-2-heptenal	-	-	0.14
Hexanal	0.06	0.04	0.03
2-heptenal	0.11	-	-
Decanal	0.01	-	-
Furfural	0.38	0.38	0.23
5- hydroxymethylfurfural	0.63	0.66	0.24
Undecenal	0.05	-	-
2-decenal	-	0.07	-
Vanillin	0.03	0.20	0.23
5- methylfurfural	-	0.24	-
Total aldehydes	1.31	1.69	1.01
Aromatic compounds			
Hexanol	0.23	-	-
2H -pyran-2,6(3H)-dion	0.09	0.50	0.27
3,5-hydroxy-2-dimethyl-4H- pyran-4-on	-	0.66	-
Total aromatic compounds	0.32	1.16	0.27
Acids			
2-Methylbutyric acid	-	2.22	1.89
Octanoic acid	0.10	0.66	0.40
Nonanoic acid	-	0.09	-
Hexanoic (caproic) acid	0.84	4.85	6.89
Tetradecanoic acid	0.32	0.24	0.36
Palmitoleic acid	0.38	0.21	0.48
Hexadecanoic acid	1.50	0.85	1.37
2- ethyl hexanoic (capronic) acid	1.29	2.11	0.95
Trans-Cinnamic Acid	2.11	7.74	7.64
Dodecanoic acid	0.07	0.07	0.12
Pentadecanoic acid	0.21	0.10	0.26
Linoleic acid	0.87	0.85	0.10
Octadecanoic acid	0.24	0.11	0.21
Cis-Cinnamic acid	-	1.73	2.10
Oleic acid	-	0.33	0.38
Total acids	7.93	22.16	23.15

Lactones			
γ- Decalactone	-	1.73	0.55
Butyrolactone	0.12	0.38	-
γ-Caprolactone	0.08	-	-
δ- Caprolactone	-	0,04	-
Total lactones	0.20	2.15	0.55
Ketones			
2-acetylfuran	0.04	-	-
Furil hydroxy methylketone	0.73	1.13	0.95
Total ketones	0.77	1.13	0.95
Furan derivatives			
2,4-dioxy-2,5-dimethyl-3(2*H*)-furan-3-one	0.12	0.10	0.10
2,5-dimethyl-4-methoxy-3(2*H*)-furanone (mesifurane)	0.89	4.61	2.36
2,5-dimethyl-4-oxy-3(2*H*)-furanone	-	0.03	0.56
2,5-dimethyl-3(2*H*)-furanone	-	0.15	0.34
Total furan derivatives	1.01	4.89	3.36
Terpenes			
Linalool	-	-	0.08
α-Terpineol	0.03	-	0.40
Limonene	0,10	-	-
Oxyde bisabolol A	0.04	0.05	-
Trans - linalool oxyde	-	0.03	0.08
Cis - linalool oxyde	-	-	0.08
Total terpenes	0.17	0.08	0.64
Total amount	12.09	33.54	30.14
LSD_{05}	*0.06*		



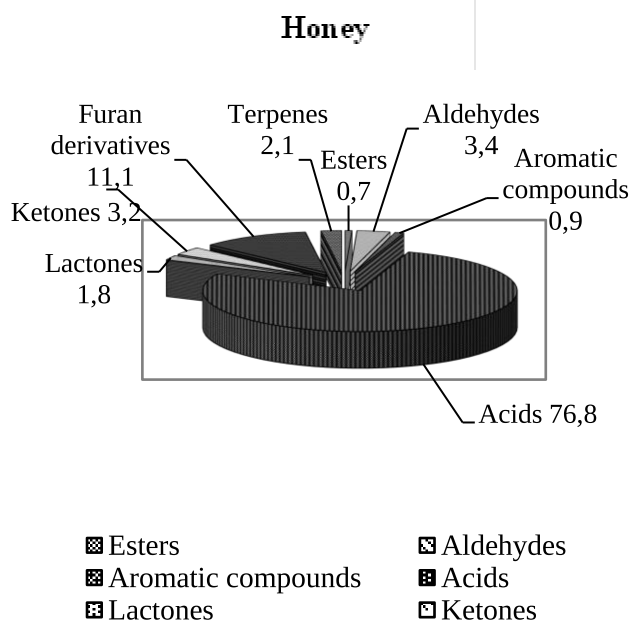


Figure 1. Volatile components of strawberry jams made of various cultivars, % of the total content

The calculation of volatile OAV of strawberry jams showed that furanone derivatives dominated: 2.4-dioxy-2.5-dimethyl-3(2H)-furan-3-one, 2.5-dimethyl-4-methoxy-3(2H)-furanone (mesifurane) and 2.5-dimethyl-4-hydroxy-3(2H)-furanone (furaneol); they add sweet, caramel scents to strawberry jams. Vanilla and hexanal are also active compounds, they are typical for vanilla and fresh grassy scents (Barren et al., 1990); 2-methylbutanoic acid, which adds sour-sweet taste, is active in strawberry jams made of "Ducat" and "Honey" cultivars.

For strawberry jams, ethyl 2-methylbutanoate, decanal along with furanes are active components which add fruity and grassy scents to strawberry jams made of cultivar "Polka". 2-decenal and γ- decalactone, 'Honey' – γ-decalactone (fruity, sweet) and linalool (sweet and floral scent) make an important contribution to the flavor of strawberry jam made of cultivar "Ducat".

Table 2. Activity of volatile components of strawberry jam flavor (OAV)

Volatiles	**Theshold, mg/kg**	**Activity of flavor volatile components (OAV)**		
		Polka	Ducat	Honey
Methyl butanoate	0.06	2.3	0.5	-
Ethyl butanoate	0.018	0.3	8.3	5.0
Ethyl crotonate	NA[1]	-	-	-
Ethyl 2-methylbutanoate	0.0001	200	-	-
Ethyl capronate	NA	-	-	-
Benzaldehyde	0.35	0.1	0.3	0.4
3,4-dihydropyran	NA	-	-	-
Trans-2-Heptenal	0.013	-	-	10.8
Hexanal	0.0045	13.3	8.9	6.7
2-Heptenal	NA	-	-	-

Decanal	0.0001	100	-	-
Furfural	3.0	0.13	0.13	0.08
Undecenal	0.005	10	-	-
2-decenal	0.0003	-	233	-
Vanillin	0.02	1.5	10	11.5
5- hydroxymethylfurfural	NA	-	-	-
5- methylfurfural	NA	-	-	-
Hexanol	2.5	0.09	-	-
2H -pyran-2,6(3H)-dion	NA	-	-	-
3,5-hydroxy-2-dimethyl-4H- pyran-4-on	NA	-	-	-
2-Methylbutanoic acid	0.25	-	8.9	7.6
Octanoic acid	0.910	0.1	0.7	0.4
Nonanoic acid	3	-	0,03	-
Hexanoic (caproic) acid	1.0	0.8	4.9	6.9
2- ethyl hexanoic (capronic) acid	NA	-	-	-
Trans-Cinnamic Acid	NA	-	-	-
Dodecanoic acid	10	-	-	-
Pentadecanoic acid	NA	-	-	-
Linoleic acid	NA	-	-	-
Octadecanoic acid	20	0.01	0.006	0.01
Cis-Cinnamic acid	NA	-	-	-
Oleic acid	NA	-	-	-
Tetradecanoic acid	10	0.03	0.02	0.04
Palmitoleic acid	NA	-	-	-
Hexadecanoic acid	NA	-	-	-
γ- Decalactone	0,01	-	173	55
Butyrolactone	NA	-	-	-
γ-Caprolactone	NA	-	-	-
δ- Caprolactone	NA	-	-	-
2-acetylfuran	10	0.004	-	-
Furil hydroxy methylketone	NA	-	-	-
2,4-dioxy-2,5-dimethyl-3(2*H*)-furan-3-one	0.00004[2]	3000	2500	2500
2,5-dimethyl-4-methoxy-3(2*H*)-furanone (mesifurane)	0.00003[2]	29667	153667	78667
2,5-dimethyl-4-hydroxy-3(2H)-furanone (furaneol)	0.00004[2]	-	750	14000
2,5-dimethyl-3(2*H*)-furanone	NA	-	-	-
Linalool	0.006	-	-	13.3
α-Terpineol	0.330	0.09	-	1.2
Limonene	0.01	10	-	-
Oxyde bisabolol A	NA	-	-	-
Trans - linalool oxyde	NA	-	-	-
Cis - linalool oxyde	NA	-	-	-

[1]NA – not available. Threshold levels of compounds (in water) were obtained from the flavor base of Leffingwell & Associates.

[2]Siegmund B., Bagdonaite K., Leitner E. (2010)

Fruity and floral, green and sweet or caramel scents are emphasized in the aroma of fresh strawberries. Esters, ethyl acetate, butyl acetate, methyl butanoate, ethyl butanaote, ethylisovalerate, methyl hexanoate and ethyl hexanaote add fruity and floral scents to strawberry flavor. Hexanal, trans-2-hexenal, 2-hexenal, hexanol, cis-3-hexen-1-ol, hexyl acetate add green scents, and furaneols – sweet, caramel ones (Pérez *et al.*, 1996; Jetti, 2005; Kim *et al.*, 2013). Having analyzed volatile activity data of strawberry jams, it has been established that typical scents for them are sweet and caramel ones due to high furaneol activity, also there are vanilla, fruity and fresh grassy scents. The flavor of strawberry jams made of "Polka" cultivar is characterized with fruity and grassy scents, that of "Ducat" cultivar – fruity and sweet scents, and for "Honey" cultivar – sweet and floral scents.

4. Conclusions

The flavor of strawberry jams made of "Polka", "Ducat" and "Honey" cultivars consists of a complex mixture of compounds, the most active among them are furanone ones: 2.4-dioxy-2.5-dimethyl-3(2H)-furan-3-one, 2.5-dimethyl-4-methoxy-3(2H)-furanone (mesifurane) and 2.5-dimethyl-4-hydroxy-3(2H) furanone(furaneol). Ethyl 2-methylbutanoate, hexanal, decanal, 2-decenal, vanillin and γ- decalactone make a great contribution to the flavor.

Sweet, caramel scents with vanilla and fresh grassy scents are very typical for strawberry jams: the flavor of strawberry jams made of "Polka" cultivar is characterized with fruity and grassy scents, that of "Ducat" cultivar – fruity and sweet scents, and for "Honey" cultivar – sweet and floral scents.

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