Aroma Profile of Strawberry Juice Cocktail Produced in Industrial Conditions

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The influence of different production methods of semi-products and final products on the aroma of juice cocktail from mixed fruits (strawberries, apples and grapes) in the conditions of industrial production was investigated in this study. Strawberry puree in frozen blocks or preserved by aseptical processing, apple puree in frozen blocks and apple and grape concentrates were used as raw materials for the production of juice cocktail. Manual headspace solid-phase microextraction (SPME) containing coated fibre (100 μm) coupled with gas chromatography (GC-FID and GC-MS) was used for the analysis of the aroma compounds. Based on the analysis of the volatile compounds and sensory evaluation of the juices, the obtained result was showed that juice cocktail made from frozen puree was of better quality than the ones produced from the aseptically preserved strawberry puree. The strawberry puree, as a dominant semi-product in these juices, had the strongest influence on the aroma profile of the examined juices. The changes of aroma compounds of the juice produced from frozen puree were less significant after 1, 3 and 6 months of storage than the one from the juice produced from aseptically preserved puree. Degradative changes (nonenzymatic browning), as well as a possible influence of the products degradation on the aroma profile, were observed during storage.

Keywords: *Aroma, industrial conditions, juice cocktail, SPME, strawberry*

Introduction

Fresh fruit is a subject to unfavourable degradative changes and therefore, after harvesting it is usually processed into different semi-products (fruit puree) or products such as fruit juices. During the production of juices, it is difficult to retain the complete natural aroma of fresh fruit. During the cutting of fruit, the aroma compounds are transferred into juice, diluted or partly degraded by enzymes so that the general impression achieved is of a weaker aroma, in comparison to the fresh fruits aroma. Also, loss of aroma compounds occurs during all thermal treatments (pasteurisation, sterilization, cooking or concentration), so they should be performed continually and in as short intervals as possible. Apart from the loss of aroma compounds, secondary compounds of unpleasant odour such as diacetyl, acetoin and acetic acid, may appear during this treatments.[1,2] Therefore, a search for new technological methods and for improvement of the old ones is constant in order to preserve the aroma of fresh fruits as much as possible. As a very important sensory characteristic of all food products, aroma is a result of a large number of volatile compounds found in small mass concentrations (up to 59 mg mL^{-1} in fruit and vegetables) in foodstuffs.[3,4] The most important among these compounds are esters, acids, alcohols, carbonyls and terpenes. According to the literature data, the composition and content of esters are most important in determining the sensory characteristics of the products investigated in this study.[5,6,7] Two types of strawberry puree were used frozen puree and aseptically preserved puree.

Frozen puree is preserved at low temperatures (–30 °C) so that the temperature in the centre of the cube is –15 °C or lower and thus frozen puree is kept at the temperature of –18 °C.

Aseptic treatment of the puree includes a brief thermic treatment (3 min at 85–95 °C) and the addition of ascorbic acid in order to prevent the process of enzymatic browning. Then follows HTST sterilization and filling in previously sterilized package material in aseptic conditions.

Gas chromatograph equipped with flame ionisation (GC-FID) and mass spectrometry (GC-MS) detectors was used for gas chromatographic analyses of the compounds in strawberry purees and in the juices produced from them. For the preparation of samples a solid phase microextraction method was used.[8]

Experimental

Samples of juices

Products presented in Table 1 were used in this study.

Juices 1 and 2 were made from strawberry puree (preserved by freezing and aseptically, respectively), frozen apple puree, apple and grape concentrates, pectin as stabiliser, sugar, and citric acid to improve the taste. They were produced under the industrial conditions in batches in the minimum volume of 10 000 L. The raw materials prescribed for this amount of final product were all added with cons-

T a b l e 1 – *Samples of the investigated juices*
T a b l i c a 1 – *Ispitivani uzorci sokova*

	Type of product Vrsta proizvoda	
	Juice cocktail 1 (strawberries, apples and grapes) made from the puree preserved by freezing Koktel-sok 1 (jagoda, jabuka i grožđe) dobiven iz kaše konzervirane smrzavanjem	Juice cocktail 2 (strawberries, apples and grapes) made from aseptically preserved puree Koktel-sok 2 (jagoda, jabuka i grožđe) dobiven iz kaše konzervirane aseptičnim postupkom
Fruit fraction in cocktail / % Udjel voća u koktel-soku / %	minimum 42	minimum 42
Dry matter fraction / % Udjel suhe tvari / %	minimum 11	minimum 11

tant mixing, first the filtered sugar solution, and then all the other ingredients. In preparation of juice 1, the frozen strawberry puree was crushed through the grinder before putting it in to the batch. The final stage of the process is performed on *Combi Block*. The in-going temperature of the product entering the filling unit was 20 to 25 °C. After that follows deaeration in vacuum (the removal of oxygen at room temperature) and then a single-step homogenisation at the pressure of 80 bar and temperature of 65 to 70 °C. Pasteurisation was done at temperature of 90 to 95 °C for 40 s. The out-going temperature was between 20 and 25 °C.

Hydrogen peroxide (35 %) was used to sterilize the packaging material before filling. The juices were analysed after 30, 90 and 180 days of storage at room temperature.

Physico-chemical analyses

Basic physico-chemical parameters of strawberry purees, preserved by freezing or aseptically and in juice cocktails produced from them, were determined: density, pH, dry matter, and total acidity of the product.[9]

Sensory analysis

Sensory evaluation of the juices produced in industrial conditions was performed using the point system by the panel of five professional judges. The system was developed on three sensory characteristics (colour, odour and taste) with the maximum of 16 points.[10]

Headspace-Solid Phase Microextraction (HS-SPME)

The SPME device used was a Supelco (Bellefonte, PA) manual SPME holder 57330-U. Fused silica fiber coated with polydimethylsiloxane (PDMS), 100 μm film thickness (Supelco) was used for extraction and concentration of aroma compounds. The fiber was preconditioned at 250 °C for 1 h in the inlet of the GC prior to sampling as instructed by the manufacturer. The samples of puree and juice (20 mL) were placed in a 50 mL glass vial in which was added NaCl p. a. (3 g) and sealed with aluminium cover and Teflon-lined septum. Following this, samples were warmed to 50 °C in water bath and gently mixed. Samples were equilibrated for 10 min prior to insertion of fiber and were maintained at 50 °C throughout the 30 min assay. The SPME fiber was exposed to headspace at 50 °C for 30 min and immediately transferred to the GC injection port at 200 °C for 3 min in splitless mode. A 0.75 mm i. d. liner (Varian Inc.) was used.

Chromatography

A Varian 3300 gas chromatograph equipped with a flame ionisation detector (FID) was used for GC analysis. Compounds were separated on a DB 624 column (6 % cyanopropylphenyl-94 % dimethyl polysiloxane stationary phase; 30 m × 0.32 mm, i. d. 1.8 μm; J&W Scientific, Folsom, CA). Nitrogen was used as a carrier gas at the flow rate of 5 mL min^{-1}. A split/splitless injector was used (ratio 1 : 5) and maintained at 200 °C. The detector was kept at 250 °C. Temperature programme was: 3 min at 40 °C, from 40 to 190 °C at 5 °C min^{-1} and 10 min at 190 °C.

The same conditions were applied for the GC-MS analysis on a Hewlett-Packard 5890 gas chromatograph with a 5970 series mass selective detector. The ionization of the samples was achieved at 70 eV under the SCAN mode. The mass range studied was from 30 to 250 *m*/*z*. Carrier gas was helium 5.0 (purity 99.999 %; Messer, Austria) at a flow rate of 5 mL min^{-1}. The aroma compounds were identified by comparing retention times and MS spectra of the pure standard substances. The MS spectra were also compared with the data from NBS75K library spectra. All the analyses were carried out in triplicate for each sample.

Results and discussion

T a b l e 2 – *Basic analytical data for the investigated juice*
T a b l i c a 2 – *Osnovni analitički parametri ispitivanih sokova*

Quality parameter Parametri kvalitete	Juice 1 Sok 1	Juice 2 Sok 2
dry matter, / % suha tvar, / %	11.13	11.15
pH	3.54	3.50
total acidity*, g L^{-1} ukupna kiselost*, g L^{-1}	4.20	4.25
density, g L^{-1} gustoća, g L^{-1}	1.044	1.048

* Total acidity was expressed as g L^{-1} of citric acid
* Ukupna kiselost izražena kao g L^{-1} limunske kiseline

Table 3 – *Results of sensory analysis of Juices 1 and 2*
Tablica 3 – *Rezultati senzorske analize sokova 1 i 2*

		Juice 1 Sok 1	Juice 2 Sok 2
immediately after production neposredno nakon proizvodnje	Average Srednja vrijednost	13.98	14.70
	Median Medijan	13.80	14.70
1 month 1 mjesec	Average Srednja vrijednost	14.02	14.24
	Median Medijan	14.00	14.40
3 months 3 mjeseca	Average Srednja vrijednost	13.22	12.60
	Median Medijan	13.20	12.40
6 months 6 mjeseci	Average Srednja vrijednost	12.48	12.36
	Median Medijan	12.60	12.30

Basic analytical data for juice cocktails are presented in Table 2 and are in agreement with the literature data.[9]

The results of sensory analysis shown in Table 3 refer to the sensory evaluation of juices immediately after the production and after1, 3 and 6 months of storage. It is interesting that the juice made from aseptically preserved strawberry puree had a better sensory evaluation immediately after the production, while the juice made from frozen puree was better evaluated after 3 and 6 months of storage. During longer periods of storage (6 months), both juices, 1 and 2 showed degradative changes (nonenzymatic browning), as well as possible influence of the products of degradation on the changes of the aroma profile of the juices, while both odour and flavour remained quite intensive.[11,12]

The results of gas chromatography analysis of the aroma compounds determined in both strawberry purees (preserved asseptically and by freezing) are presented in Table 4 and are expressed as peak area of the identified compounds. The coefficient of variation (CV) of the determined aroma compounds was between 2.3 and 10.1 %, with the mean value of 5 %.

According to literature data[5,7] the strawberry aroma is the result of the presence of different compounds such as esters, alcohols, carbonyls and sulphuric compounds, among which esters are the most important group regarding the quality and quantity of the aroma compounds of strawberry puree. Fatty acid ethyl esters: ethyl hexanoate, ethyl octanoate and ethyl decanoate, are responsible for fresh aroma of strawberry puree. Methyl butanoate, ethyl butanoate, methyl hexanoate, hexyl acetate and ethyl hexanoate contribute most to the fresh fruity aroma of strawberry, while the odour of 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone and 2,5-dimethyl-4-methoxy-3(2*H*)-furanone, important aroma compounds of strawberry, are described as caramel-like. Ethyl butanoate, another important aroma compound of strawberry, was determined in similar quantities in both aseptically preserved puree and in the frozen one, while methyl anthranilate, ethyl decanoate and

Table 4 – *Peak area of identified aroma compounds in strawberry purees preserved by freezing and aseptically*
Tablica 4 – *Površine pikova određenih aromatičnih sastojaka u kašama jagode konzerviranim smrzavanjem i aseptičnim postupkom*

Compound Spoj	Strawberry puree preserved by freezing Kaša jagode konzervirana smrzavanjem	Aseptically preserved strawberry puree Kaša jagode konzervirana aseptičnim postupkom
Esters / Esteri		
Methyl butanoate Metil-butanoat	425	268
Ethyl butanoate Etil-butanoat	8997	8690
Butyl butanoate Butil-butanoat	678	317
Methyl hexanoate Metil-heksanoat	2553	2274
Ethyl hexanoate Etil-heksanoat	724	917
Butyl acetate Butil-acetat	n. d.	n. d.
Hexyl acetate Heksil-acetat	563	577
Methyl anthranilate Metil-antranilat	2715	3248
Ethyl decanoate Etil-dekanoat	307	534
Ethyl octanoate Etil-oktanoat	1499	1113
Alcohols / Alkoholi		
1-hexanol 1-heksanol	910	678
1-octanol 1-oktanol	1377	790
Linalool Linalool	1759	1256
α-terpineol α-terpineol	873	441
Carbonyls and lactones/ Karbonilni spojevi i laktoni		
2-hexenal 2-heksenal	1516	620
2-heptanone 2-heptanon	1997	1236
γ-decalactone γ-dekalakton	2256	1414

n = 3

Table 5 – *Changes in peak area of aroma compounds in Juice 1 during storage*

Tablica 5 – *Promjena udjela aromatičnih sastojaka u voćnom soku 1 tijekom skladištenja*

Compound Spoj	Juice 1 Sok 1		
	1 month 1 mjesec	3 months 3 mjeseca	6 months 6 mjeseci
Esters / Esteri			
Methyl butanoate Metil-butanoat	220	n. d.	n. d.
Ethyl butanoate Etil-butanoat	10835	8905	3992
Butyl butanoate Butil-butanoat	5600	3950	2674
Methyl hexanoate Metil-heksanoat	1572	330	291
Ethyl hexanoate Etil-heksanoat	11562	8307	4551
Butyl acetate Butil-acetat	3479	2421	1572
Hexyl acetate Heksil-acetat	64549	23551	10339
Methyl anthranilate Metil-antranilat	35186	12596	10362
Ethyl decanoate Etil-dekanoat	2913	841	622
Ethyl octanoate Etil-oktanoat	2903	886	354
Alcohols / Alkoholi			
1-hexanol 1-heksanol	8606	3983	2172
1-octanol 1-oktanol	17162	2552	1891
Linalool Linalool	3507	2072	1489
α-terpineol α-terpineol	21482	3807	2856
Carbonyls and lactones / Karbinilni spojevi i laktoni			
2-hexenal 2-heksenal	3859	1550	853
2-heptanone 2-heptanon	1510	996	183
γ-decalactone γ-dekalakton	80878	25067	15881

n = 3

Table 6 – *Changes in peak area of aroma compounds in Juice 2 during storage*

Tablica 6 – *Promjena udjela aromatičnih sastojaka u voćnom soku 2 tijekom skladištenja*

Compound Spoj	Juice 2 Sok 2		
	1 month 1 mjesec	3 months 3 mjeseca	6 months 6 mjeseci
Esters / Esteri			
Methyl butanoate Metil-butanoat	227	n. d.	n. d.
Ethyl butanoate Etil-butanoat	13488	4883	2755
Butyl butanoate Butil-butanoat	3036	2194	1258
Methyl hexanoate Metil-heksanoat	1311	377	210
Ethyl hexanoate Etil-heksanoat	2021	1030	468
Butyl acetate Butil-acetat	2559	11735	577
Hexyl acetate Heksil-acetat	73013	23933	18963
Methyl anthranilate Metil-antranilat	9991	3991	1287
Ethyl decanoate Etil-dekanoat	2463	649	443
Ethyl octanoate Etil-oktanoat	2154	446	448
Alcohols / Alkoholi			
1-hexanol 1-heksanol	11187	6636	2732
1-octanol 1-oktanol	1636	464	n. d.
Linalool Linalool	1919	4274	3117
α-terpineol α-terpineol	11604	6579	3071
Carbonyls and lactones / Karbonilni spojevi i laktoni			
2-hexenal 2-heksenal	2898	878	719
2-heptanone 2-heptanon	954	906	258
γ-decalactone γ-dekalakton	76666	32855	22318

n = 3

ethyl hexanoate were detected in larger quantities in the aseptically preserved puree. Methyl anthranilate, with its strong spicy-aromatic and flowery tone, is characteristic for the aroma of wild strawberries.[13] Content of the esters largely diminishes during the storage period, which is reflected on the sensory characteristics of juices. Apart from most esters, other aroma compounds, such as alcohols (1-hexanol, 1-octanol), carbonyls (2-hexanal, 2-heptanone) and lactones (γ-decalactone), were determined largely in the frozen strawberry puree. The odour of γ-decalactone is described as peach-like.[1] The obtained results are in agreement with the literature data,[1,6] according to which the content of most aroma compounds is reduced in the aseptically preserved puree compared to the frozen puree due to the thermal treatment.

The data presented in Table 5 shows that during the storage of juice 1 the fraction of aroma compounds was reduced. According to literature data[5] during longer periods of storage the content of ethyl hexanoate is significantly reduced due to the degrading changes. Coefficient of variation (CV) was between 3.5 and 12.8 %, with the mean value of 4.2 %.

From the comparison of the results in Tables 5 and 6, as well as the Figures 1 and 2, it can be concluded that juice 1 (made from frozen strawberry puree) was richer in aroma compounds compared to juice 2 (made from aseptically preserved strawberry puree), and that the most significant reduction of the content of most aroma compounds occurred between the 1st and 3rd month of storage.

Conclusions

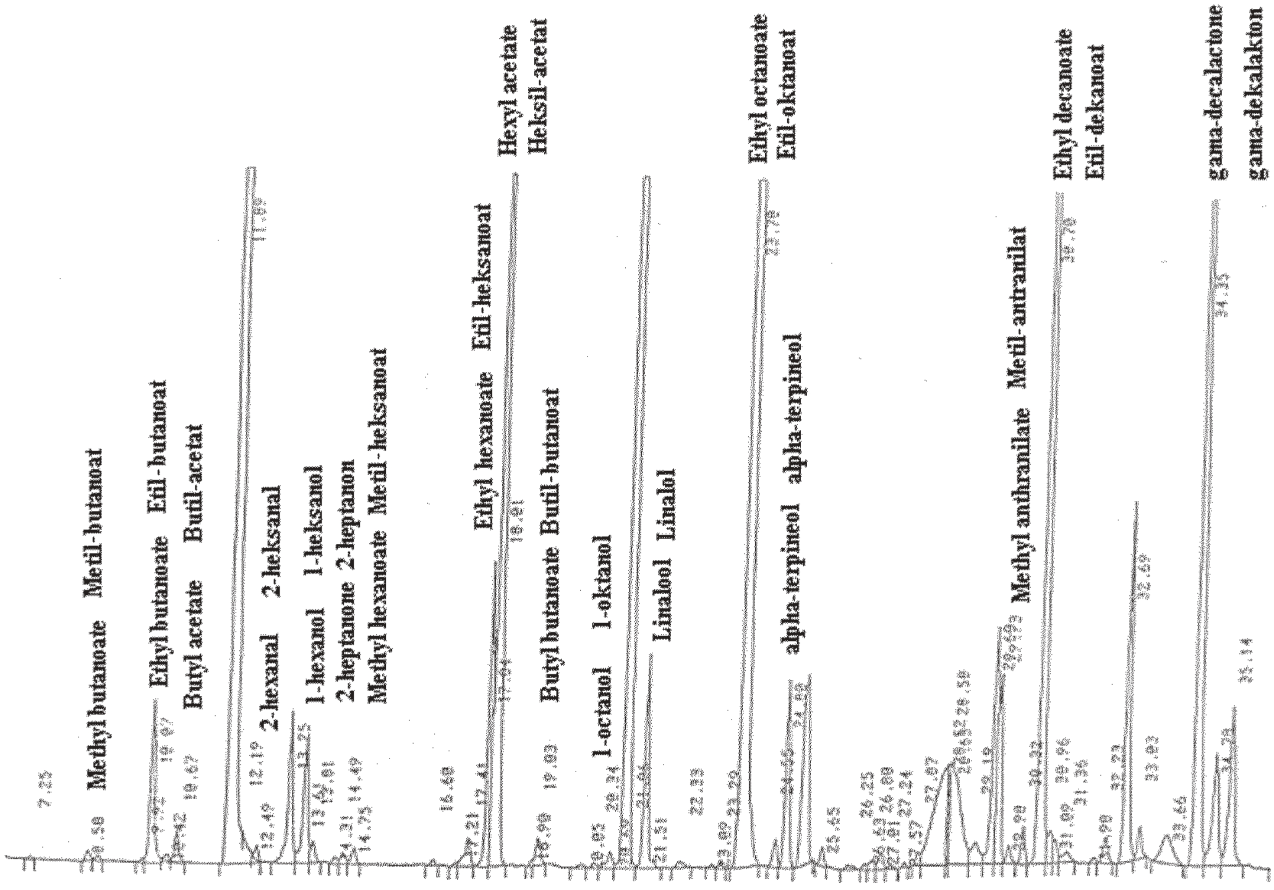
The raw material has an important influence on the quality and intensitiy of aroma. In this case, the raw material is the semi-product, i. e. strawberry puree, of the dominant fruit in the investigated cocktail (strawberries, apples and grapes).

The aroma profile of the juice produced from the strawberry puree preserved by freezing proved to be much better than of that obtained from the aseptically preserved puree.

The changes of aroma compounds of the juice produced from frozen puree were also less significant after 1, 3 and 6 months of storage than the one from the juice produced from aseptically preserved puree.

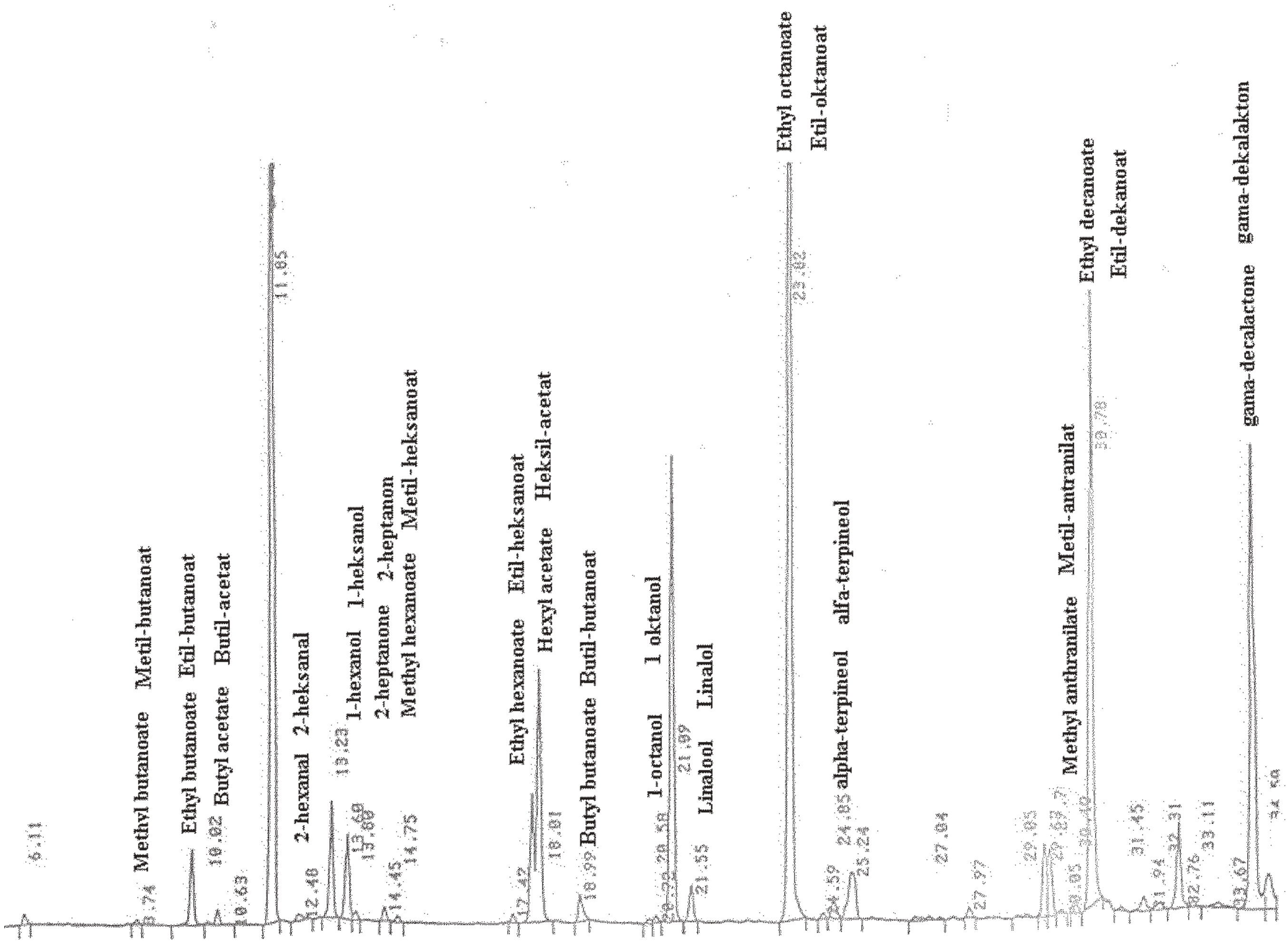
The results of the analysis of total aroma and individual aroma compounds are in agreement with the results of sensory evaluation, which also showed that the produce obtained from the frozen puree was of a much better quality and much more stable. During the storage, degrading changes were noticed not only of the aroma but also of colour. The products of degradation might have influenced the changes of the aroma profile (nonenzymatic browning).

Differences in the dynamics of degradation of each determined aroma compound were observed. It is not possible to explain them appropriately based on this research, therefore, further investigation of this topic is needed.



F i g . 1 – *Chromatogram of the juice cocktail 1*

S l i k a 1 – *Kromatogram koktel-soka 1*



F i g . 2 – *Chromatogram of the juice cocktail 2*
S l i k a 2 – *Kromatogram koktel-soka 2*

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SAŽETAK

Profil arome koktel-sokova jagode u uvjetima industrijske proizvodnje

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U radu je ispitivan utjecaj različitih načina pripreme poluproizvoda i gotovih proizvoda na aromatski profil kašastih sokova od miješanog voća (jagode, jabuke i grožđa) u uvjetima industrijske proizvodnje.

Sirovine za proizvodnju koktel-soka bile su kaša jagode u obliku smrznutih blokova i kaša jagode dobivena aseptičnim postupkom, kaša jabuke u obliku smrznutih blokova te koncentrati jabuke i grožđa.

Za određivanje aromatičnih sastojaka primijenjena je tehnika mikroekstrakcije na čvrstoj fazi (HS-SPME) s punilom polidimetilsiloksanom (100 μm PDMS) u kombinaciji s plinskom kromatografijom s plameno-ionizacijskim (GC-FID) i maseno-selektivnim detektorom (GC-MS). Identificirani aromatični sastojci u uzorcima voćnih sokova uključuju estere, karbonilne spojeve, terpene, alkohole i kiseline.

Na temelju rezultata plinsko-kromatografske analize i organoleptičkog ocjenjivanja sokova pokazalo se da je aromatski profil soka proizvedenog iz smrznute kaše bio znatno bolji nego aromatski profil soka dobivenog iz kaše jagode konzervirane aseptičnim (termičkim) postupkom.

Promjene aromatičnih sastojaka tijekom šest mjeseci bile su manje u soku dobivenom iz smrznute kaše. Utvrđeno je da na aromatski profil proučavanog soka bitno utječe uporabljeni poluproizvod, kaša jagode koja je najzastupljenija u tom soku. Tijekom skladištenja uočene su degradativne promjene (neenzimsko posmeđivanje), kao i mogući utjecaj produkata degradacije na promjenu profila arome.

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