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SPICE PAPRIKA VOLATILES AS AFFECTED BY DIFFERENT POSTHARVEST RIPENING TREATMENTS OF RED PEPPER (*Capsicum annuum* L.) VARIETY ALEVA NK

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The influence of post-harvest ripening conditions of pepper AlevaNK picked in red maturity stage on the composition of volatiles in spice paprika was investigated by GC-MS. The post-harvest ripening in the dark and under daylight was conducted under semi-controlled conditions for two weeks. The obtained chromatograms indicated that the aroma of investigated spice paprika consisted of a large number of volatile compounds regardless of the application and conditions of the post-harvest ripening. The main volatiles of the analyzed paprika samples were fatty acids and their esters, terpenes and terpenoides and aldehydes and ketones. The share of fatty acids and their esters decreased during the post-harvest ripening, and the ripening in the dark favored the decrease. The share of terpenes and terpenoides and the share of aldehydes and ketones in the total volatiles increased during the post-harvest ripening. The post-harvest ripening in the dark favored the increase of the share of terpenes and terpenoides, while the ripening under daylight favored the increase of the share of aldehydes and ketones.

KEY WORDS: red pepper, post-harvest ripening, volatiles

INTRODUCTION

Ground red paprika is a common spice widely appreciated in gastronomy due to its intense red color and specific aroma. The composition of the pigments and color of spice paprika have been in the focus of numerous authors studying the dependence on the variety (1, 2), maturity stage (3, 4), processing (5, 6, 7) and storage (8) parameters. On the other hand, the investigations related to the issue of spice paprika aroma, which is a result of the complex composition of paprika volatiles, mainly remained at the identification level of volatiles in paprika of different origin (9, 10, 11, 12). The influence of pepper processing on paprika volatiles was the subject of interest only for a few authors like Luning et al. (13, 14) who investigated the effect of drying on paprika volatiles and Lee et al. (15) who investigated the influence of gamma irradiation. There is no data in

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academic literature related to the influence of pepper post-harvest ripening conditions on the composition of the volatiles in spice paprika.

The aim of this study was to investigate and compare the composition of volatiles in spice paprika in dependence of the presence of light during two weeks of post-harvest ripening of pepper (Capsicum annum L.) variety AlevaNK harvested in red maturity stage.

EXPERIMENTAL

Pepper fruits (*Capsicum annuum* L.), variety "AlevaNK" dedicated to paprika powder production were picked from the field near Stara Pazova, Serbia. The fruits were picked when approximately 80% of all fruits in the field reached red stage of maturity. After picking, all fruits were divided in three lots. The first lot was dried within three days after picking. The drying process was conducted with gradual increase of temperature from 40°C to 80°C in 10 h. Two remaining fruit lots were subjected to 14 days post-harvest ripening treatment under semi-controlled conditions (temperature 25-45°C, humidity 40-60%). One lot was ripened in the glasshouse under natural daylight, while the other was ripened in the dark, without presence of any light source. After the post-harvest ripening both lots were dried in the same way as the control one. The pericarp of the dry fruits was separated from the pods and seeds and used for further analysis. The pericarp was briefly (10 s) ground in a laboratory grinder in order to avoid sample heating, and the obtained powder was used for the analysis of volatiles.

The extraction and separation of essential oils from powdered sample was performed according to the procedure described by Kevrešan et al. (16). In brief: 30 g of dry sample was mixed with 500 ml of water and 1 ml of hexane was added to the mixture before distillation. The distillation time was 90 minutes and the obtained organic phase was collected and kept at -20°C until the analysis. Separation of the volatiles from pepper fruit essential oils was performed by GC-MS (Thermo Finnigan) under the following conditions: the interface temperature 280°C, ion source temperature 230°C, injector temperature 250°C, helium flow 1.5 ml min^{-1}. The separation was performed on a 60 m × 0.25 mm, J&W DB-5MS (Agilent, USA) column with 0.25 μm film thickness by applying the following temperature program: the initial 50°C, the final 280°C, with a linear gradient 2°C $minute^{-1}$. The injection volume was 1 μl of the prepared sample. The obtained peaks were identified by comparing their mass spectra with those in the NIST database (17). The results are expressed as the percent of the area of individual peaks (compounds) in relation to the total chromatogram area.

RESULTS AND DISCUSSION

The chromatograms obtained from the distillates of volatiles from the paprika lots ripened after harvest under different conditions are presented in Figure 1. It is obvious, oppositely to the finding of Luning et al. (13, 14) who reported a decrease of the number of volatiles in bell pepper after drying, that a large number of volatile compounds was registered in all the analyzed samples (97 compounds in the paprika dried without post-harvest ripening, 96 compounds in the paprika ripened in the dark, and 89 compounds in the paprika ripened under natural daylight). Such result was probably a consequence of a gradual increase of temperature during the drying process, which is recommended for the production of high quality spice paprika with supreme aromatic properties.

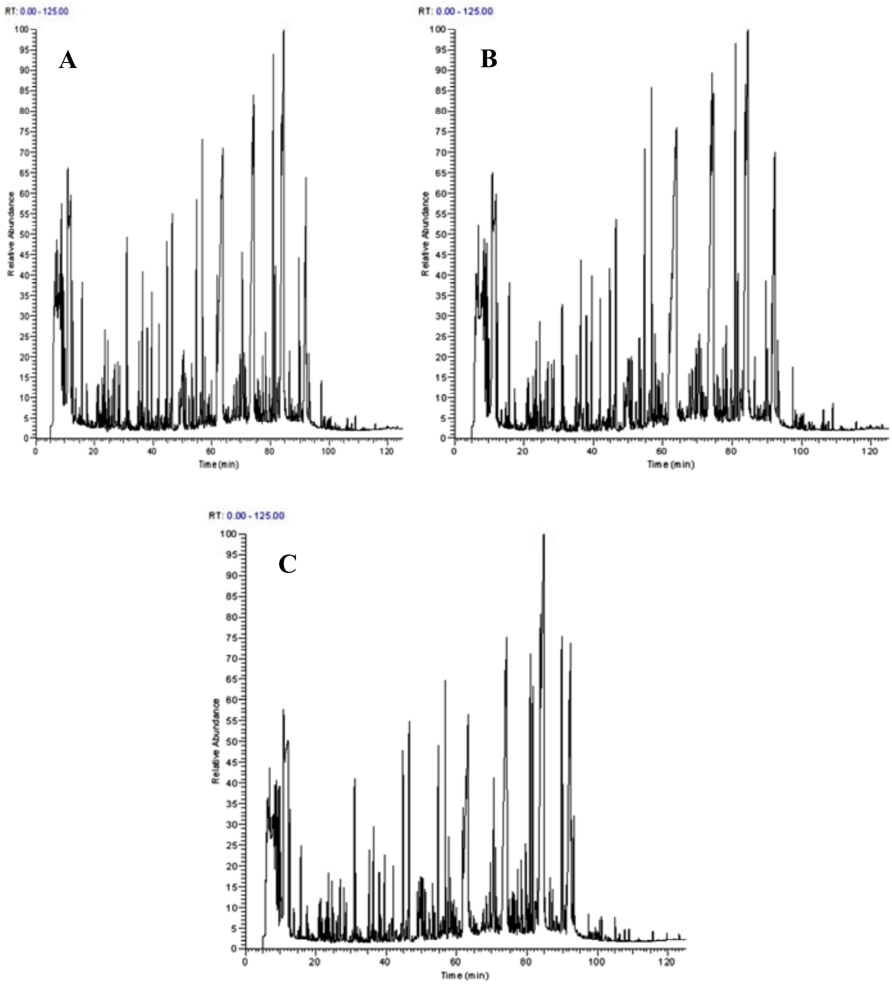

Figure 1. Chromatograms of the volatiles distilled from extract of the paprika: not ripened (A), ripened in the dark (B) ripened under natural daylight (C)

Out of the total number of registered volatile compounds, those with the peaks in the chromatograms representing more than 0.1% of the total chromatogram area were used for further analysis of the differences of volatiles composition in paprika in dependence of the applied post-harvest treatment (46 compounds in the paprika dried without post-harvest ripening, 44 compounds in the paprika ripened in the dark, and 43 compounds in the paprika ripened under natural daylight). The other identified, but not quantified compounds, are just listed without the analysis of the differences.

Among the compounds present in at least one sample with the share above 0.1% of fatty acids and their esters (Table 1), terpenes and terpenoides (Table 2), aldehydes, ketones (Table 3) and other compounds (Table 4) were registered. The presented grouping of the results was based on the classification into the same compound class, but more important for understanding of the meaning of the obtained results are the differences among the groups of compounds in their odor strength, substantivity and type. Fatty acids and their esters are characterized in most cases with low strength fatty or waxy odors with long substantivity. Terpenes and their derivatives are usually the source of strong floral and fruity odors with short substantivity, while aldehydes and ketones represent the green, earthy, woody or fruity type odors with different strength and substantivity levels (http://www.thegoodscentscompany.com).

Table 1. Shares (%) of acids and esters identified in paprika in dependence of red pepper post-harvest ripening treatments

Compound	Odor type/strength/ substantivity[1]	Post-harvest ripening, %		
		none	14 days in the dark	14 day under daylight
ACID				
Palmitic	Waxy/low/400h	11.55	9.04	8.11
Lauric	Fatty/medium/no data	6.92	<0.1	9.15
Myristic	Waxy/low/400h	5.40	5.34	5.5
cis,cis-Linoleic	Fainth fatty/none/none	4.22	2.75	2.06
Pentadecanoic	Waxy//medium/no data	0.57	0.64	0.64
Capric	Fatty/medium/no data	0.46	1.09	1.08
Total acids		**29.12**	**18.86**	**26.54**
ESTER				
Methyl linolenate	None/none/none	1.66	0.67	0.55
Methyl palmitate	Waxy/low/no data	1.31	0.73	2.46
Isopropyl palmitate	Bland/none/208h	0.32	0.36	0.33
Isopropyl myristate	Bland/none/400h	0.22	<0.1	0.12
Methyl stearate	Waxy/low/no data	0.18	<0.1	0.12
Total esters		**3.69**	**1.76**	**3.58**
TOTAL ACIDS AND ESTERS		**32.81**	**20.62**	**30.12**

[1] http://www.thegoodscentscompany.com

The highest share in composition of volatiles in paprika, regardless of the applied post-harvest treatment, had volatile acids (Table 1). The dominant acid was palmitic acid with the share of around 11.5 % in the paprika produced without post-harvest ripening. Despite of slightly decreasing amount of palmitic acid after both investigated post-harvest treatment, it was the compound with the highest share. Significant shares in the paprika obtained without post-harvest ripening of red pepper were determined also in the cases of lauric, myristic and linoleic acid, but their behavior during the post-harvest ripe-

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ning differed in dependence of the applied ripening conditions. Lauric acid almost completely disappeared when ripening was conducted in the dark, but its share increased when ripening was conducted under natural daylight. Regardless of ripening conditions, myristic acid remained at the same level, while the share of linoleic acid was halved after two weeks of post-harvest ripening. Among the identified acids were also pentadecanoic and capric acid. The percent of pentadecanoic acid slightly increased, while the share of capric acid, which was also identified in Hungarian spice paprika by Kocsis et al. (11, 12) more than doubled during both applied post-harvest treatments. The presence of propionic and butiric acids reported by Mateo et al. (9) and Lee et al. (15) was not registered in samples of variety "AlevaNK".

Among the volatiles of spice paprika several esters were identified: linoleic, palmtic and stearic acid methyl esters, identified also by Kocsis et al. (11, 12) in Hungarian spice paprika, along with the palmitic and myristic acid isopropyl esters. Methyl laurate was also identified but not quantified in Hungarian spice paprika by Kocsis et al. (11, 12). Oppositely to our results and the results for Hungarian spice paprika obtained by Kocsis et al. (11, 12), fatty acids esters were not identified in Spanish spice paprika (9).

The share of esters, almost as a rule, decreased during two weeks of the post-harvest ripening of red pepper. The exceptions were the double increase of the share of methyl palmitate in the paprika obtained from pepper ripened under the daylight, and almost unchanged percentage of isopropyl palmitate.

Generally, the total share of volatile acids and esters decreased after the post-harvest ripening, and this was more pronounced in the case of the post=harvest ripening in the dark. Such results indicate that the post-harvest ripening contributes to the diminution of long-lasting heavy fatty and waxy odor of paprika spice, especially if conducted in the dark. The post-harvest ripening under the daylight, which is usually applied in the production of spice paprika, resulted in our experiment in a just slight decrease in the content of fatty acids and their esters, while the post-harvest ripening in the dark resulted in a decrease of their share by more than 35%.

Terpenes and terpenoides (Table 2) were the second of compounds by its share in the total volatiles of spice paprika produced from red pepper AlevaNK. Among the compounds from this group, various terpenes and terpenoides were identified. The identified representatives of monoterpenes and their derivatives were *trans*-geranyl aceton, β-linalool, β-cyclocitral, nerol, α-terpineol, limonene and *trans*-geraniol. The share of monoterpenes and their derivatives increased for all identified compounds in the case of post-harvest ripening in the dark, while the post-harvest ripening under the daylight resulted in a decrease of monoterpenoid alcohols (β-linalool, nerol and α-terpineol).

Sesquiterpenes and their derivatives were represented by farnesyl acetone, which was at the same time the compound with the highest share among terpenes and terpenoides. The share of this compound notably increased after the post-harvest ripening in the dark, while the ripening under the daylight led to almost complete disappearance of this compound. The post-harvest ripening in the dark resulted in a slight increase of sesquiterpenes *trans, trans*-Farnesal and spathulenol, while under the daylight only *trans*-Farnesal was synthesized, while spathulenol disappeared.

Table 2. Shares (%) of terpenes and terpenoids identified in paprika in dependence of red pepper post-harvest ripening treatment

Compound	Odor type/strength/ substantivity[1]	Post-harvest ripening, %		
		none	14 days in the dark	14 day under daylight
Farnesyl acetone	Fruity/medium/no data	1.59	2.46	<0.1
trans- β -ionone	Floral/medium/no data	1.89	2.12	2.70
trans-geranyl acetone	Floral/medium/no data	1.07	1.23	1.66
β-linalool	Floral/medium/12h	0.79	0.92	0.55
trans-damascenone	Floral/high/191h	0.41	0.36	0.22
β-cyclocitral	Minty/high/85h	0.39	0.53	0.60
trans-α-ionone	Floral/medium/112h	0.29	0.36	0.47
trans-phytol	Floral/low/no data	0.27	0.13	<0.1
Nerol	Floral/medium/44h	0.25	0.27	0.16
α-terpineol	Floral/medium/20h	0.24	0.27	0.15
Isophytol	Floral/low/68h	0.19	-	0.44
Dihydro-β-ionone	Woody/medium/9h	0.15	0.14	0.24
Limonene	Citrus/medium/4h	0.11	0.18	0.16
trans, trans-farnesal	No data	<0.1	0.13	0.13
trans-geraniol	Floral/medium/60h	<0.1	0.12	<0.1
Spathulenol	Earthy/medium/no data	<0.1	0.41	-
TOTAL TERPENES		**7.64**	**9.83**	**7.48**

[1] http://www.thegoodscentscompany.com

Diterpenes were represented by a low percent of phytols (*trans*-phytol and isophytol). As a consequence of post-harvest treatments, the share of *trans*-phytol in the essential oil showed a decrease. The share of isophytol was strongly influenced by the post-harvest treatment. Namely, its content doubled when the post-harvest treatment under the daylight was applied, while after the ripening in the dark no isopytol was detected.

Among terpenoides, the compounds with ionone structure were also present (α- and β-ionon and *dihydro*-β-ionone). A significant increase of these compounds after the post-harvest ripening was partly attributed to the degradation of β-carotene in the processes of post-harvest ripening but also in the processes of thermal degradation of β-carotene, described by Bonnie and Choo (18). The increase in the share of compounds with ionone structure was more expressed in the case of the post-harvest ripening under the daylight.

Generally, the total share of terpenes and terpenoides in the paprika volatiles increased by almost 30% after post-harvest ripening in the dark, while post-harvest ripening under the daylight resulted only in a slight decrease of the share of this group of compounds. Such observation indicate that post-harvest ripening in the dark might result in more expressed floral and fruity aroma of spice paprika in comparison to the paprika produced from non-ripened pepper or pepper ripened under the daylight.

The volatiles of spice paprika obtained from red pepper AlevaNK, regardless of applied post-harvest ripening treatment, included various aldehydes and ketones (Table 3). The main representative of this group of compounds was *trans,trans-2,4*-decadienal. The share of this aldehyde, characterized with fatty odor, decreased after both applied post-harvest ripening processes. The other identified aldehydes and ketones were registered with notably lower shares, but their shares after both applied post-harvest ripening treatments, almost as a by rule, showed an increase.

The composition of aldehydes of the investigated spice paprika obtained from red pepper AlevaNK was similar to the spice paprika from Hungary (11, 12) which contained heptadienal, benzaldehid and 2,4-heptadienal. Some of the determined aldehydes, like 2,6-nonadienala and 2,4-dekadienala, were also registered in spice paprika from Morocco (10), while octanal, 2-octenal, nonanal and 2,6-nonadienal, registered in spice paprika from Hungary (11, 12), were not identified in spice paprika produced from red pepper AlevaNK.

Generally, both applied post-harvest treatments contributed to increase of the total share of aldehydes and ketones in spice paprika with more expressed increase (above 25%) in the case of post-harvest ripening under the daylight. Such results indicate that spice paprika ripened under the daylight might have more expressed green, woody, earthy and nutty aroma in comparison to the paprika produced after post-harvest ripening in the dark.

Table 3. Shares (%) of aldehydes and ketones identified in paprika in dependence of red pepper post-harvest ripening treatment

Compound	**Odor type/strength/ substantivity**[1]	**Post-harvest ripening, %**		
		none	**14 days in the dark**	**14 day under daylight**
trans,trans-2,4-decadienal	Fatty/high/94h	2.02	1.76	1.5
3,5-dimethyl-benzaldehyde	No data	0.59	0.24	1.31
(Z)-9,17-octadecadienal	No data	0.55	0.4	0.47
benzeneacetaldehyde	Green/high/no data	0.34	0.33	0.33
Nonanal	Aldehydic/high/no data	0.33	0.46	0.41
trans-2-trans-4-heptadienal	Fatty/high/no data	0.3	0.57	0.4
Safranal	Herbal/high/no data	0.25	0.37	0.4
benzaldehyde	Fruity/high/4h	0.24	0.28	0.25
p-methylbenzaldehyde	Fruity/high/120h	0.21	0.36	0.35
Decanal	Aldehydic/high/36h	0.13	0.12	0.13
Heptanal	Green/high/12h	0.11	0.15	<0.1
(Z)-2-heptenal	Green/high/no data	0.11	0.13	0.13
pentadecanal	Waxy/medium/no data	-	<0.1	0.27
Hexanal	Green/high/no data	-	0.43	<0.1
Total aldehides		**5.18**	**5.60**	**5.95**
2-tridecanone	Waxy/medium/no data	0.54	0.38	0.54
2,2,6-trimethylcyclohexanone	Thujonic/high/no data	0.13	0.20	0.19
methyl heptenone	Citrus/medium/no data	<0.1	0.16	0.16
p-methylacetophenone	Floral/medium/20h	-	0.12	0.11
Total ketones		**0.67**	**0.86**	**1.00**
TOTAL ALDEHIDES AND KETONES		**5.85**	**6.46**	**6.95**

[1] http://www.thegoodscentscompany.com

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Compounds from the other groups (Table 4) were identified with the shares of up to 0.5% (Table 4), and their share did not change much in dependence of the applied post-harvest treatment.

Table 4. Shares (%) of other compounds identified in paprika in dependence of red pepper post-harvest ripening treatment

Compound	Odor type/strength/ substantivity[1]	Post-harvest ripening, %		
		none	14 days in the dark	14 day under daylight
delta-dodecanolactone	Tropical/medium/400h	0.46	0.52	0.58
decahydro-2,2-dimethyl-naphthalene	No data	0.36	0.21	-
2,6,6-trimethyl-1-cyclohexene-1-acetaldehyde	Camphoreous/medium/no data	0.29	0.36	0.45
1,2-dihydro-1,5,8-trimethyl naphthalene	Licorice/medium	0.25	0.18	0.21
2-*n*-pentylfuran	Fruity/medium/no data	0.25	0.33	0.23
falcarinol	No data	0.24	0.13	<0.1
p-cresol	Phenolic/high/400h	0.19	<0.1	<0.1

[1] http://www.thegoodscentscompany.com

The identified phenols were not found in spice paprika from Hungary (11, 12), but *p*-cresol was identified also in spice paprika from Spain (9). Dibuthylphenol, which was present among the volatiles of spice paprika from Korea (15) and Hungary (11, 12), was not identified in our samples.

The compounds registered with shares under 0,1% (Table 5) present in paprika produced from red pepper without post-harvest ripening were identified also in paprika produced after post-harvest ripening regardless of the applied ripening treatment. Exceptions to this statement were α-(3-methylbutylidene)-benzeneacetaldehyde and phytol, which disappeared only in the case of the post-harvest ripening in the dark, hexadecane and retinol-acetate which disappeared only in the case of post-harvest ripening under the daylight and 3-hexanol, 3-*ethyl*-1,5-octadiene and 2-*ethyl*-1-hexanol which disappeared during both post-harvest ripening treatment. The only compounds which were not identified in paprika produced without post-harvest ripening were manoyl oxide which was identified in paprika after both postharvest ripening treatments and 3-methylundecane, which was identified only in paprika after post-harvest ripening in the dark.

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Table 5. Compounds identified in paprika in dependence of red pepper post-harvest ripening treatment with shares under 0.1%

Compound	**Post-harvest ripening, %**		
	none	**14 days in the dark**	**14 days under daylight**
Furfural	<0.1	<0.1	<0.1
2-hexenal	<0.1	<0.1	<0.1
2-heptanone	<0.1	<0.1	<0.1
acetyl furan	<0.1	<0.1	<0.1
3,5-dimethylphenol	<0.1	<0.1	<0.1
3-*hepten*-2-one	<0.1	<0.1	<0.1
propyl benzene	<0.1	<0.1	<0.1
5-*methyl* furfural	<0.1	<0.1	<0.1
1-*octen*-3-one	<0.1	<0.1	<0.1
decane	<0.1	<0.1	<0.1
Octanal	<0.1	<0.1	<0.1
1,2,3-trimethylbenzene	<0.1	<0.1	<0.1
benzyl-alcohol	<0.1	<0.1	<0.1
α-*trans*-ocimene	<0.1	<0.1	<0.1
butylbenzene	<0.1	<0.1	<0.1
trans-2-octenal	<0.1	<0.1	<0.1
trans-linalool	<0.1	<0.1	<0.1
Terpinolene	<0.1	<0.1	<0.1
α-cyclocitral	<0.1	<0.1	<0.1
2-pentylfuran	<0.1	<0.1	<0.1
2-bornene	<0.1	<0.1	<0.1
trans-2-decenal	<0.1	<0.1	<0.1
2,4-dimethylbenzene	<0.1	<0.1	<0.1
β-ionone	<0.1	<0.1	<0.1
α-ionone	<0.1	<0.1	<0.1
α-copaene	<0.1	<0.1	<0.1
Tetradecane	<0.1	<0.1	<0.1
6-*methyl*-6-(methyl- furan-2-yl)	<0.1	<0.1	<0.1
heptan-2-one	<0.1	<0.1	<0.1
β-ionone-epoxide	<0.1	<0.1	<0.1
methyl - dodecanoate	<0.1	<0.1	<0.1
trans-nerolidol	<0.1	<0.1	<0.1
Tetradecanal	<0.1	<0.1	<0.1
methyl - linoleate	<0.1	<0.1	<0.1
methyl palmitate	<0.1	<0.1	<0.1
3-hexanol	<0.1	-	-
3-*ethyl*-1,5-octadiene	<0.1	-	-
2-*ethyl*-1-hexanol	<0.1	-	-
manoyl oxide	-	<0.1	<0.1
3-*methyl*-undecanoate	-	<0.1	-
α-(3-*methylbutylidene*)-benzeneacetaldehyde	<0.1	-	<0.1
phytol	<0.1	-	<0.1
Hexadecane	<0.1	<0.1	-
retinyl-acetate	<0.1	<0.1	-

CONCLUSION

It can be concluded that the volatiles of spice paprika produced from pepper variety AlevaNK consist of a large number of compounds regardless of the application and conditions of post-harvest ripening. Among the identified compounds predominant were fatty acids and their esters, terpenes, terpenoides, aldehydes and ketones. The total share of fatty acids and their esters in the total volatiles decreased after post-harvest ripening. The ripening in the dark favored the decrease of the share of fatty acids and their esters and thus contributed to the less expressed long-lasting, heavy fatty and waxy aroma of paprika spice. The post-harvest ripening resulted in the increase of total shares of terpenes and terpenoides and total share of aldehydes and ketones. The ripening in the dark favored the increase of the share of terpenes and terpenoides, while ripening under daylight favored increase of the total share of aldehydes and ketones.

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УТИЦАЈ РАЗЛИЧИТИХ ТРЕТМАНА ДОЗРЕВАЊА ЦРВЕНЕ ПАПРИКЕ (*Capsicum annuum* L.) СОРТА АЛЕВА НК ПОСЛЕ БЕРБЕ НА ИСПАРЉИВА ЈЕДИЊЕЊА ЗАЧИНСКЕ ПАПРИКЕ

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Утицај услова послежетвеног дозревања паприке Алева НК убране у црвеној фази зрелости на састав испарљивих једињења је испитиван гасном хроматографијом - масеном спектрометријом. Послежетвено дозревање је спроведено у мраку и под дневним светлом у полу контролисаним условима у току две недеље. Добијени хроматограми указивали су на то да се арома испитиваних узорака зачинске паприке састојала од великог броја једињења без обзира на то да ли је дозревање после бербе примењени и под којим је условима спроведено. Највећи удео у структури испарљивих једињења зачинске паприке имају масне киселине и њихови естри, терпени и терпеноиди и алдехиди и кетони. Удео масних киселина и њиховхи естара у укупним испарљивим једињењима се смањује током дозревања после бербе уз фаворизовање пада удела дозревањем у мраку. Удели терпена и терпеноида као и удели алдехида и кетона се током дозревања после бербе повећавају. Дозревање у мраку фаворизује раст удела терпена и терпеноида, док дозревањем на дневном светлу фаворизује раст удела алдехида и кетона.

Кључне речи: паприка, постжетвено дозревање, испарљива једињења

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