Chemical composition and distribution of the headspace volatiles in commercial culinary herbs and spices: Chemometric approach

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Abstract: Fast determination of the volatile components of widely used culinary herbs and spices, without prior preparation of the sample, using headspace sampling (HS) and GC–MS/FID analysis, is presented. Generally, monoterpenes were the dominant class in HS volatiles (HSV): monoterpene hydrocarbons in dill, celery, parsnip and parsley, and oxygenated monoterpenes in coriander, bay laurel and rosemary. The HSV of ginger and clove samples differed from the others due to their high content of sesquiterpene hydrocarbons and phenylpropanoids, respectively. The most extreme case of limited distribution was related to the samples of garlic-HSV comprised organosulfur compounds and none of the above mentioned classes. Furthermore, the HSV data were processed *via* principal component analysis, making visible the variability and tendency between the investigated samples. Lastly, the research provides insights in many HS profiles of culinary herbs and spices, giving relative representation of volatiles as well as differences and deviation in terms of HSV content.

Keywords: chemical composition; spice; herbs; HS-GC–MS/FID; principal component analysis.

INTRODUCTION

Spices and culinary herbs are known as flavouring, colouring, preservative and healing agents.[1] The characteristic flavour of spices and herbs originate both from aroma compounds in essential oils (more volatile) and oleoresins (less volatile) compounds that impart taste.[2] Qualitative and quantitative composition of volatiles in spices and herbs are in direct relation to aroma experience and delivery. Therefore, the analysis of volatiles is a required step in quality assessment.

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Monoterpenes, sesquiterpenes, and miscellaneous compounds make up the volatile (aroma) fraction of spices and herbs.[3] There are different applicable methods for aroma compound isolation: stem distillation, Soxhlet extraction, simultaneous distillation–extraction, supercritical fluid extraction, and headspace (HS) techniques such as static-headspace sampling, purge and trap, and solid-phase microextraction.[4] Data on spice and herb volatiles mostly originate from studies that examined chemical composition and different biological activities of essential oils isolated from specific plants (organs). One procedure that is often used for the isolation of essential oil is hydrodistillation. It is a time-consuming method and the plant material is exposed to high temperatures, which often allows various (undesirable) chemical changes of essential oil volatiles.[1,5] Also, the procedure requires larger amounts of sample, posttreatment of isolated essential oil (partitioning and preparation for further analysis), and it is not suitable for screening a large number of samples.[5] On the other hand, headspace sampling of volatiles is simple, quick, requires small amount of the sample, no artefacts are formed and, if properly used, no contaminants are introduced.[6] Modern technology enables easily automated procedure, which means subsequent injection of vapour, from a vial with the sample, into GC for further analysis. Static headspace coupled with GC is a frequently applied technique for the analysis of volatiles in solid matrices, applicable for the isolation and characterization of flavour and fragrance compounds.[4,5] There are studies that consider both headspace and essential oil volatiles. In general, the chemical composition of headspace volatiles differs from that of hydrodistillated oil regarding the content of more volatile monoterpene fraction.[7–9] Finally, a recent study has demonstrated that headspace sampling is applicable in the quality assessment of sage essential oil as a complementary extraction technique for rapid screening.[5]

With the intention to contribute to and enrich scientific insights using fast analytical procedures such as automated HS-GC–MS/FID, this study aims to show and compare the chemical profiles and distribution of volatile compounds from numerous spices and culinary herbs. Therefore, the study was done on 20 different commercial spices and culinary herbs (84 samples) from various manufacturers. Aroma compounds were sampled by HS technique and analysed by GC–MS/FID. The study also includes chemometric approach *via* conducted principal component analysis (PCA) which picturized the characteristic variation of the volatiles and the aggregation of samples affected by the different chemical composition of the headspace volatiles (HSV).

EXPERIMENTAL

Plant material – purchased spices and herbs

The study includes 84 samples from 8 different plant families: Myristicaceae, Piperaceae, Lauraceae, Zingiberaceae, Amaryllidaceae, Myrtaceae, Lamiaceae and Apiaceae. They were purchased ftom the local markets in Niš (Serbia) in bags of net mass up to 30 g. A speci-

fications and details on the studied spices and herbs are given in Table I and in Supplementary material to this paper.

TABLE I. General information of studied spices and culinary herbs; commercial name is given in both English and Serbian language. Abbreviations: L – leaves, S – seeds, F – fruits, SB – stem bark, Bb – bulbs, Bd – buds, R – roots and Rh – rhizomes. *AM* assigns average mass of samples taken for headspace analysis (with standard deviation not higher than 0.01)

Family	No.	Species	Common name	Plant organ	Country(ies) of origin	No of samples	*AM* g
Myrist-icaceae	I	*Myristica fragrans*	Nutmeg Muskantni orah	S milled	Belgium, India, Indonesia	3	0.1167
Piperaceae	II	*Piper nigrum*	Black paper Crni biber	F milled	Vietnam, Poland	6	0.1100
Lauraceae	III	*Cinnamo-mum verum*	Cinnamon Cimet	SB powdered	Indonesia	6	0.1150
Lauraceae	IV	*Laurus nobilis*	Bay laurel Lovor	L whole	Turkey, Indonesia	6	0.1083
Zingibe-raceae	V	*Curcuma longa*	Turmeric Kurkuma	Rh milled	India	2	0.1150
Zingi_ber-aceae	VI	*Zingiber officinale*	Ginger Đumbir	Rh milled	China, India	2	0.1050
Amaryl-lidaceae	VII	*Allium sativum*	Garlic Beli luk	Bb granulated	China, India	6	0.1283
Myrtaceae	VIII	*Syzygium aromaticum*	Clove Karanfilić	Bd whole powdered	Comoro Islands, Madagascar, Brasil	4	0.1425
Lamiaceae	IX	*Ocimum basilicum*	Basil Bosiljak	L chopped	Serbia, Spain, Egypt	7	0.1114
Lamiaceae	X	*Origanum majorana*	Marjoram Majoran	L chopped	Egypt	2	0.1050
Lamiaceae	XI	*Origanum vulgare*	Oregano Origano	L chopped	Serbia, Turkey, Netherlands, Greece	6	0.1083
Lamiaceae	XII	*Rosmarinus officinalis*	Rosemary Ruzmarin	L chopped	Serbia, Spain, Egypt, Tunisia, Albania	5	0.1220
Lamiaceae	XIII	*Thymus vulgaris*	Thyme Timijan	H chopped	Serbia, Poland, Brasil	3	0.1067
Apiaceae	XIV	*Anethum graveolens*	Dill Mirođija	L chopped	Serbia	8	0.1075
Apiaceae	XV	*Apium graveolens*	Celery Celer	L chopped	China	1	0.1109
Apiaceae	XVI	*Carum carvi*	Caraway Kim	F whole	Egypt, Turkey, Lithuanian	6	0.1267
Apiaceae	XVII	*Coriandrum sativum*	Coriander Korijander	S whole milled	Ukraine	2	0.1100
Apiaceae	XVIII	*Levisticum officinale*	Lovage Selen	L chopped	Serbia	1	0.1083
Apiaceae	XIX	*Pastinaca sativa*	Parsnip Paškanat	R chopped	Serbia	1	0.1922
Apiaceae	XX	*Petroselinum crispum*	Parsley Peršun	L chopped	Serbia	7	0.1143

Headspace sampling, GC-MS/FID analysis and identification

Headspace (HS) sampling, gas chromatography–mass spectrometry (GC–MS) analysis and identification of volatiles were done in accordance with already published procedure.[9] Sample preparation for analysis was done immediately on opening the bags, by measuring the spice into 20-mL headspace vials and subsequently adding 1 mL of distilled water (*AM* (g) assigns average mass of sample taken for headspace analysis, Table I). HS volatiles were isolated by automatized procedure: the sample was heated at 80 °C for 20 min with shaking for 5 s and pausing for 2 s, after which the aliquot of volatiles was injected into the GC. Qualitative and quantitative data of HS volatiles were obtained by GC–MS and GC-flame ionization detector (GC/FID) analyses. Samples were analyzed on Agilent Technologies 7890B GC equipped with a fused silica capillary column (HP-5MS, 250 μm×25 m, film thickness 0.25 μm, Agilent Technologies, Santa Clara, CA, USA) and coupled with a 7890A FID and 7000B MS/MS spectrometer (operating in MS1 scan mode) from the same company. The GC was operated under the following conditions: injector temperature 250 °C; GC–MS interface temperature 300 °C; oven temperature programmed from 50 °C for 2.25 min, then to 290 °C at 4 °C/min (carrier gas He, 1.0 mL/min, constant flow mode). MS conditions were as follows: ionization voltage of 70 eV; acquisition mass range 40-440; scan time 0.32 s. HSV were identified from total ion chromatogram (TIC) by comparison of their linear retention indices relative to C_8–C_{40} *n*-alkanes recorded, on the same column/temperature program, with literature values – NIST Chemistry WebBook and Adams.[10,11] Their mass spectra were compared with those of standards from Wiley 6, NIST02, Adams by the application of the automated mass spectral deconvolution and identification system (AMDIS) software (ver. 2.7, distributed within software package for 7890-7000 BGC–MS/MS triple quadrupole system). The percentage composition of the oil was computed from the GC peak areas without any corrections.

Statistical data analysis

Statistical data processing was carried out by Statistica 8 software. Statistical matrices included the content (in percentage) of HSV as original variables. From the total data set of the 120 detected original HSV, 39 compounds (present with an average concentration higher than 5 % at the level of study species) were selected for principal component analysis (PCA).

RESULTS AND DISCUSSION

Profiling of the headspace volatiles in spices and herbs

According to the results obtained, it is evident that the chemical profiles of samples which belong to the same spice or culinary herb can differ from each other, in some cases drastically. These differences are usually related to quantitative composition, while qualitative composition was not drastically different among the samples. In Supplementary material, Table S-I displays the average values of contents (mean values), except for spices/herbs which were included with one sample (lovage, celery, parsnip), while the chemical profiles of HSV of all samples, with the original percentage representation, are provided as Tables S-II–S-XXI (Supplementary material).

In general, monoterpenes (hydrocarbons and/or oxygenated derivatives) are the dominant class of HSV among the samples studied. The distribution of mono-

terpenes, sesquiterpenes and phenylpropanoids, as frequently present classes in the samples studied, is diverse. The dill, celery, parsnip and parsley samples were most abundant in monoterpene hydrocarbons, while the highest content of oxygenated monoterpenes was observed in the HSV of coriander, bay laurel and rosemary. Ginger and clove stand out from the others due to the highest content of sesquiterpene hydrocarbons and phenylpropanoids, respectively. Oxygenated sesquiterpenes and other hydrocarbons (like aliphatic alcohols, aldehydes, ketones and esters) are not present in a significant percentage. HSV of garlic consisted of sulfides, with none of the mentioned classes present (Fig. 1).
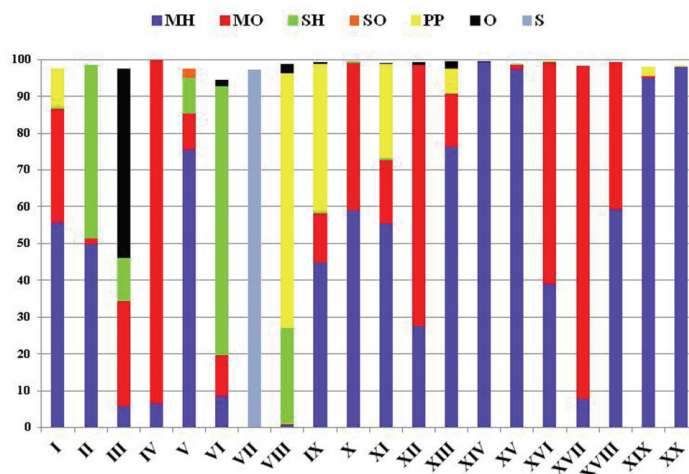

Fig. 1. Graphical representation of HSV distribution in studied spices and herbs respecting the following classification (average contents): MH – monoterpene hydrocarbons, MO – oxygenated monoterepenes, SH – sesquiterpene hydrocarbons, SO – oxygenated sesquiterpenes, PP – phenylpropanoids, S – sulfides and O – aliphatic alcohols, aldehydes, ketones, esters. I–nutmeg, II–black peper, III–cinnamon, IV–bay laurel, V–curcuma, VI–ginger, VII–garlic, VIII–clove, IX–basil, X–marjoram, XI–oregano, XII–rosemary, XIII–thyme, XIV–dill, XV–celery, XVI–caraway, XVII–coriander, XVIII–lovage, XIX–parsnip, XX–parsley.

Samples listed under the Apiaceae family comprise leaves, seeds, fruits and roots. The volatile profile of dill samples includes 9 compounds among which monoterpene hydrocarbons account for more than 90 % (mostly α- and β-phellandrene: mean value±standard deviation = 72.4±11.3 and 12.6±2.4 %, respectively), while the content of dill ether in HSV is generally low. p-Cymene is the most variable compound in dill-leaf HSV (10.1±7.6 %). In the HSV of coriander seeds, oxygenated monoterpenes are significantly prevalent (linalool, 87.8±1.3 %). Although monoterpenes are the dominant class in the HSV of caraway samples, a very small amount of sesquiterpene (E)-caryophyllene (1.5 %) was detected in one sample. Among monoterpenes, carvone followed by limonene are the two most dominant volatiles of caraway seeds (56.7±18.0 and 39.0±18.3 %,

respectively). *β*-Phellandrene (42.8±7.9 %) and 1,3,8-*p*-menthatriene (15.4±15.0 %) are dominant monoterpenes in parsley-leaf HSV. According to the standard deviation value, besides 1,3,8-*p*-menthatriene, most parsley volatiles showed significant variance. Monoterpene hydrocarbons and oxygenated monoterpenes HSV of lovage are approximately represented with *β*-phellandrene (38.5 %) and *α*-terpinyl acetate (39.7 %) as dominant components of both fractions respectively. The HSV of parsnip root abound in terpinolene (70.9 %), while myristicin is present in the amount of 2.4 %. One sample of celery leaves contains limonene and myrcene dominantly (74.1 and 18.9 %, respectively).

The group of Lamiaceae family is more homogenous since all samples studied are classified as culinary herbs (basil, oregano, marjoram, rosemary and thyme). Regarding the distribution of the most abundant volatiles among the seven samples, the HSV profile of basil proved to be very inconstant: 1,8-cineole (*syn*. eucalyptol, 11.7±7.7 %), very variable content of linalool (43.8±35.2 %) and estragole (40.0±33.2 %). Despite belonging to the same plant genus and same plant organ (leaves), oregano and marjoram samples are quite different in HSV composition. The dominant compounds in oregano HSV are carvacrol (23.5±22.2 %), *p*-cymene (20.3±10.6 %), *γ*-terpinene (19.0±13.9 %) and linalool (11.7±15.7 %), while in marjoram HSV: *γ*-terpinene (15.1±1.1 %), *trans*- and *cis*-sabinene hydrate (17.3±4.9 and 6.1±0.1 %, respectively), sabinene (12.5±0.8 %), terpinene-4-ol (12.8±4.2 %) and *α*-terpinene (10.8±1.3 %). Unlike the complex HSV of oregano and marjoram, about 70 % of the HSV fraction of rosemary samples is made up of two compounds: 1,8-cineole (54.3±23.7 %) followed by *α*-pinene (14.5±11.0 %). Although the HSV profile of thyme consists of 34 volatiles, among all volatiles *p*-cymene convincingly dominates (66.2±19.0 %).

Headspace volatiles of two representatives of the Lauraceae family, cinnamon and bay laurel, showed quite different representation of HSV which is expected since the differences of examined material. HSV of bay laurel are mostly consisted of 1,8-cineole (88.6±2.6 %), which is, after (*E*)-cinnamaldehyde (50.9±4.2 %), the second most abundant volatile (16.0±3.9 %) in HSV of cinnamon samples.

According to the mean values of contents, among all the HSV of examined nutmeg samples (Myristicaceae), terpinen-4-ol (29.8±26.1 %) and *α*-pinene (11.9±8.7 %) are dominant, while sabinene and *β*-pinene are significantly present in one sample (10.3 and 11.8 %, respectively). Eugenol (68.6±19.6 %) and (*E*)-caryophyllene (24.6±19.0 %) make up about 90 % of HSV of clove buds (Myrtaceae). The rhizomes of Zingiberaceae representatives differ from each other regarding their sesquiterpenes content. While the HSV of ginger root abound in this class (*α*-zingiberene (34.0±17.1 %), *α*-curcumene (12.9±5.2 %), *β*-sesquiphellandrene (11.9±6.9 %), *β*-bisabolene (9.1±3.3 %) and 1,8-cineole (7.3±7.1 %)), the HSV of turmeric contain monoterpenes, *α*-phellandrene (56.9±

±0.4 %) and 1,8-cineole (10.0±0.8 %), mostly. Limonene (14.6±5.1 %) and (*E*)-caryophyllene (38.6±17.3 %) make up about 50 % of HSV of examined black pepper fruits (Piperaceae). Moreover, the presence of sabinene, *β*-pinene and *δ*-3-carene is significant in several samples (more than 10 %). Finally, the HSV of granulated garlic, included in the study with 7 samples, contain only organosulfur compounds where diallyl trisulfide (41.3±13.9 %) dominates followed by diallyl disulfide (18.7±6.4 %), methyl allyl trisulfide (17.9±5.8 %) and diallyl sulfide (10.6±10.0 %).

Principal component analysis: variability and tendency

The conducted PCA visualizes the variability and tendency between the 20 investigated species of spices and culinary herbs. The scatter plot in the projection of the first two principal component axes shows a tendency towards the formation of three sample groups, see Fig. 2. (*a*1). The first group, composed only of *Allium sativum* samples, had negative values for both axes. As shown in Fig. 2 (*b*1), their clear separation was influenced by high contents of five sulfide metabolites: diallyl sulfide, methyl allyl disulfide, diallyl disulfide, methyl allyl trisulfide and diallyl trisulfide. This result was expected considering the limited distribution of sulfides, as well as the fact that in garlic bulbs none of the other compound classes were detected. Two samples of *Zingiber officinale* formed the second group at the positive part of axis 2. The diversity of ginger was caused by high contents of four sesquiterpenes (Fig. 2 (*b*1)): *α*-curcumene, *α*-zingiberene, *β*-bisabolene and *β*-sesquiphellandrene. These four compounds represent 67.6 % of total HSV in ginger rhizomes, and it is interesting that they were additionally found only in the rhizomes of *Curcuma longa*. However, their contents in turmeric rhizomes were very low (8.6 % for all four compounds collectively). Finally, the third and largest group, consisting of samples of all the other investigated species, showed positive values for axis 1. This group was characterrized by an abundance of all the other tested characters (mono-, sesquiterpenes and/or phenylpropanoids), which had a more even distribution between species.

The grouping obtained implicated further data processing by performing new PCA without the garlic and ginger samples. The PCA showed an overlap of most samples (Fig. 2 (a2)). However, along the first axis clear separation of two samples of *Origanum majorana* was evident. Their separation was influenced by the highest contents of four monoterpenes (Fig. 2 (b2)): *trans*-sabinene hydrate, *cis*-sabinene hydrate, *α*-terpinene and sabinene. The most represented between them, *trans*-sabinene hydrate, was detected only in marjoram leaves, while its isomer, *cis*-sabinene hydrate, was found in another four species, but with an average concentration of less than 1 %. Moreover, if we compare the HS profile of marjoram with the other species, it is evident that no other species has so many compounds (five) present in the concentration above 10 %. Besides marjoram,

two additional species showed a certain degree of separation from the remaining samples. Seven samples of *Petroselinum crispum* formed a group at the negative part of axis 2. The diversity of parsley was caused by the highest contents of three monoterpenes: *β*-phellandrene, 1,3,8-*p*-menthatriene and *p*-cymenene. As somewhat expected, 1,3,8-*p*-menthatriene and *p*-cymenene were compounds exclusive to the HS profile of parsley.
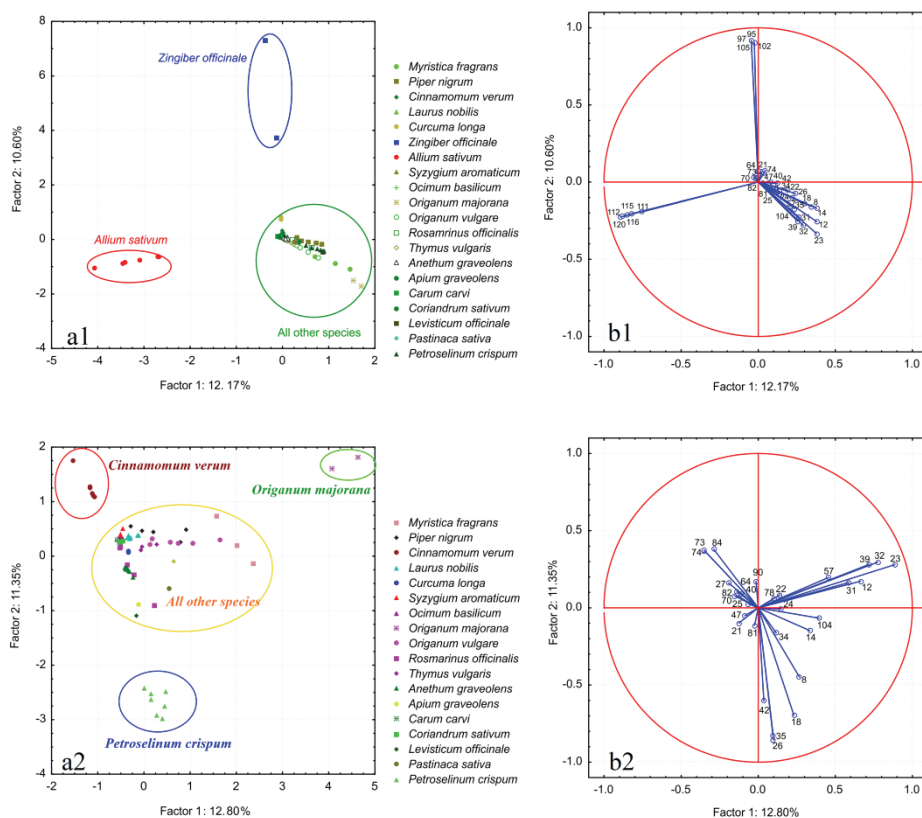

Fig. 2. a1) Principal component analysis (PCA) of 84 samples of 20 investigated spices and culinary herbs. b1) Representation of 39 selected variables (HSV) on the plane of the first two axes. a2) PCA of 80 samples of 18 spices and culinary herbs (without garlic and ginger samples). b2) Representation of 30 selected variables (HSV) on the plane of the first two axes. The numbers represent the entries of the components, cf. Table S-I.

Finally, *Cinnamomum verum* samples, which had the most negative values for axis 1 and positive values for axis 2, were characterized by a unique presence and high abundance of phenylpropanoid (*E*)-cinnamaldehyde as well as the highest content of two monoterpenes: isobornyl acetate and *α*-copaene. The analysis was done under the same principle for culinary herbs, and the results lead to an

identical observation as for the previous set of data; parsley and marjoram were also separated from the other herbs examined.

CONCLUSION

The research provides insights in headspace profiles of aromatic culinary plants and spices, giving relative representation of volatiles, differences and deviation in terms of HSV content as well as the distribution of HSV depending on manufacturers. The described isolation process of the aromatic compounds (HSV) is partly comparable to the average cooking procedure. In this sense, the results make a connection between the chemical composition/content of volatile compounds in the vapour (HSV) with the first sensation in the process of preparing and consuming food. PCA analysis enables visualization of the tendency of relationships, grouping and extraction in the mass of data. Thus, the analysis, conducted, visualizes the variability of a large number of spices tested with respect to the content of volatile compounds that define the aroma of a commercial product. From the raw data, the observer senses the range of variation in the quality of the spices, while looking at the resulting graphical representation of the PCA has a broader insight into variability. Considering that many samples from different spices and herbs are included, the contribution of this study is reflected in the comprehensive data on the chemical profiles/composition of the most volatile part of the flavour and the variation pattern at the level of a very diverse group. In this sense, fast automated HS-GC–MS method of isolation and analysis is applicable as complementary in assessing the quality of spices and herbs regarding volatile fraction.

SUPPLEMENTARY MATERIAL

Additional data are available electronically at the pages of journal website: http:////www.shd.org.rs/JSCS/, or from the corresponding author on request.

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ХЕМИЈСКИ САСТАВ И ДИСТРИБУЦИЈА ХЕДСПЕЈС ИСПАРЉИВИХ ЈЕДИЊЕЊА У КОМЕРЦИЈАЛНИМ ЗАЧИНИМА И ЗАЧИНСКОМ БИЉУ: ХЕМОМЕТРИЈСКИ ПРИСТУП

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Спроведен је брз и аутоматизован поступак тзв. хедспејс (headspace, HS) изоловања и GC–MS/FID анализе испарљивих једињења (HSV) из великог броја комерцијално доступних зачина и зачинског биља без претходне припреме узорка. Генерално, монотерпени су најзаступљенија класа једињења у HSV: монотерпенски угљоводоници у

мирођији, целеру, першуну и пашканату; оксигеновани угљоводоници у коријандеру, ловору и рузмарину. Састав хедспејс испарљивих једињења ђумбира и каранфилића се веома разликује у односу на остале узорке према садржају сесквитерпенских угљоводоника и фенилпропаноида. Најупадљивије одступање у погледу састава HSV примећен је код узорака белог лука обзиром да испарљиву фракцију чине органосумпорна једињења и ниједно од терпенских или фенилпропаноидних једињења. Како је у истраживање укључен велики број узорака, спроведена је мултиваријантна статистичка анализа главних компонената како би се уочила и представила тенденција испитиваних узорака ка груписању у складу са варијабилношћу у саставу HSV. Истраживање даје HS профиле зачина и зачинског биља: релативна заступљеност једињења у HS фракцији, разлике у саставу и одступања на нивоу различитих извора и произвођача.

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