Effects of storage temperature and time on metabolic and flavouromic profiles of roasted germinated sunflower seeds

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Summary
Roasted germinated sunflower seeds were stored at 20 °C and 40 °C for 1 to 4 weeks. Integrative metabolomics-flavouromics was applied to monitor dynamic changes of metabolites and volatile compounds during storage using gas chromatography-flame ionization detection and headspace solid-phase microextraction combined with gas chromatography-mass spectrometry. In total, 63 metabolites and 73 volatile compounds were identified. Relative targeted quantification showed a reduction of unsaturated fatty acids, amino acids, γ-aminobutyric acid and organic acid contents, and an increase in the contents of hexanal, heptanal and nonanal at 40 °C. Principal components analysis differentiated all samples related to their storage temperature and time. Heatmap and agglomerative hierarchical clustering analysis further revealed the differences and similarities among the samples. The storage of roasted germinated sunflower seeds clustered into three major groups. For instance, group I showed higher levels of off-flavour compounds at 40 °C, indicating a higher potential of lipid oxidation occurring at 40 °C. Group III exhibited higher levels of amino acids and pyrazines at 20 °C, indicating a low loss of nutrients and a high potential of roast aroma at 20 °C. The samples stored at 20 °C better maintained nutrients and the roast aroma.

Keywords
sunflower seed; metabolite; flavour; storage

Sunflower (*Helianthus annuus* L.) belongs to *Asteraceae* family. The seed is one of the world's leading oilseeds, second only to soybeans in terms of the total output of oleic acid [1]. The values of sunflower seeds go far beyond their pleasant taste and satisfying crunchy texture.

The germination process is an inexpensive and effective technology for improving the nutritional quality of seeds [2]. Germination can activate hydrolytic enzymes, resulting in breaking down a portion of starch, proteins, lipids and fibre [3], and lead to structural modification and synthesis of new bioactive compounds [4, 5]. The nutrients of whole seeds including dietary fibre, minerals, free amino acids, phenolic compounds and antioxidant capacity were reported to improve during germination [6–9]. To improve the nutritional quality of sunflower seeds, additionally, aroma precursors, such as reducing sugars and free amino acids, are formed during germination, from which the typical roast and nutty aroma are suggested to be generated during the roasting process. Therefore, germinated sunflower seeds were used as a material for the roasting process in this study.

Roasting is a good way to prepare the seeds for snacks or long-term storage. It is the key process for obtaining nuts with a better taste, aroma, crunchy texture and enhanced crispiness. With unique nutty flavours and pleasant aromas, roasted sunflower seeds are greatly appreciated by consumers worldwide. Furthermore, sunflower seeds supply a large number of nutrients including unsaturated fats, protein, polyphenols, vitamins and minerals. Sunflower seeds contain approximately

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20 % protein and are rich in polyunsaturated fatty acids (approximately 31 %) [10]. Thus, sunflower seeds occupy a unique position among oilseeds. However, high levels of unsaturated fatty acids make sunflower seeds susceptible to lipid oxidation during storage, which can lead to an important quality loss, with deleterious effects and the occurrence of toxic compounds [11]. The shelf life of stored sunflower seeds is impacted by many external factors, storage temperature is one of the main factors. Suitable storage temperature control is key to preserving the nutritional value and flavour of sunflower seeds [12].

Metabolomics refers to the comprehensive unbiased profiling of all metabolites in a biological sample. The combination of gas chromatography (GC) with flame ionization detection (FID) or mass spectrometry (MS) has been demonstrated to be a valuable tool for metabolite and flavour profiling in plants [13]. Detailed untargeted metabolite and flavour analysis of various plant cultivars can provide the opportunity to better understand the relevant processes, accumulation and retention of flavour-relevant compounds regerading new cultivar development, germination and roasting practices. Previous studies showed that metabolomics and flavouromics can be successfully used for monitoring of changes in the volatile profile of roasted hazelnuts and for sensitive, robust and reliable identification of chemical markers of hazelnut roasting [14], enabling chemical changes to be correlated with technological processing and sensory quality [15]. The hypothesis of the applied metabolomics and flavoroumics technology is to identify the metabolic and flavour profiles that could be adopted as quality indicators. The application of metabolomics techniques for the investigation of metabolic profiles can provide a more holistic view of the composition of sunflower seeds and generate new hypotheses as to how and why metabolites and flavour profiles of sunflower seeds differ during storage.

The study aimed at investigation of the effects of temperature and time on metabolite and flavour profiles of roasted germinated sunflower seeds during storage. Further, aimed at investigation of the relationship between the characteristic flavour of roasted germinated sunflower seeds and metabolites.

MATERIALS AND METHODS

Roasted germinated sunflower seed preparation

Raw sunflower seeds (50 g) were germinated in a constant climate chamber with controlled humidity model KBF 720 (Binder, Tuttlingen, Germany) at 25 °C, 75% relative humidity for 24 h. The germinated seeds were roasted at 125 °C in an electric forced air oven Model UF55 (Memmert, Tuttlingen, Germany) for 45 min. The roasted germinated sunflower seeds were cooled to ambient temperature, shelled and ground using an electric grinder MX-337 (Panasonic, Tokyo, Japan) and stored in a sealed plastic bag at 20 °C and 40 °C in an incubator with 50% relative humidity. Accordingly, samples were evaluated on weeks 1, 2, 3, and 4. At each sampling time, samples were removed from the incubator and subjected to metabolite and flavour evaluation by gas chromatography with flame ionization detection (GC-FID) and headspace solid-phase microextraction coupled to gas chromatography-mass spectrometry (HS-SPME-GC-MS), respectively. The experiment was repeated three times.

Chemicals

Tetracosane (1 $mg \cdot ml^{-1}$), 5α-cholestan-3β-ol, *p*-chloro-L-phenylalanine and phenyl-β-D-glucopyranoside (2 $mg \cdot ml^{-1}$) were used as internal standards for semi-quantification of metabolites in fraction I, II, IV, and III, respectively. Undecane, hexadecane, tetracosane, triacontane, and octatriacontane were used as retention-time standards. All reference standards for identification of metabolites were obtained from Sigma-Aldrich (St. Louis, Missouri, USA). An internal standard of 50 $mg \cdot kg^{-1}$ ethyl decanoate of sunflower seed (ethyl decanoate 1 $mg \cdot ml^{-1}$ in 10% methanol) was used for semi-quantification of volatile compounds. The reference standards for volatile compounds identification were isobutanol, 2-methylpropanal, 3-methylbutanal, 3-penten-2-one, 1-pentanol, 3-hydroxy-2-butanone, hexanal, *n*-hexanol, 2,5-dimethylhexane, furfural, 1-octen-3-ol, γ-butyrolactone, methional, 2-methyloctane, nonanal, L-carvone, benzaldehyde, heptanal, trans-2-heptenal, *trans*-2-octenal, camphene, phenol, 2-heptanone, *p*-cymene, 2-acetylfuran, 2-acetyl-3-methylpyrazine, 2,5-dimethylpyrazine, 2-ethyl-3-methylpyrazine, 2-methylpyrazine, 2-ethylpyrazine, 2,3-diethyl-5-methylpyrazine, pyridine, 2-ethyl-3,5-dimethylpyrazine, 2-acetylpyrrole, 2,3-dimethylpyrazine, γ-valerolactone, 2,5-dimethyltetrahydrofuran, 2-pentylfuran, phenylacetaldehyde, D-limonene, 2-ethylhexyl acrylate, β-phellandrene, α-pinene, β-pinene, ethyl nonanoate and ethyl octanoate. All the reference standards were obtained from Sigma-Aldrich.

Detection and identification of metabolites

Extraction and fractionation procedures of

sunflower flour were described as previously procedures by NA JOM et al. [16] with some modifications. A sample of 100 g sunflower flour was obtained by adding 4 ml dichloromethane and 10 ml of methanol-deionized water (80:20 v/v). 100 μl tetracosane (1 mg·ml^{-1}) and 100 μl 5α-cholestane-3β-ol (1 mg·ml^{-1}) were added to 4 ml of the lipid extract. The lipid extract was transesterified by adding 500 μl of methyl-*tert*-butylether (MTBE), 300 μl dry methanol and 50 μl sodium methylate (5.4 mol·l^{-1} in methanol) and the mixture reacted at room temperature in the darkness for 90 min. Then, 1 ml dichloromethane and 2 ml aqueous 0.35 mol·l^{-1} hydrochloric acid solution were added for selective hydrolysis. The upper phase was discarded and the lower phase containing transmethylated lipids was evaporated to dryness in a vacuum parallel evaporator at 50 °C and then re-dissolved in 250 μl dichloromethane. The transmethylated lipid was separated by solid-phase extraction into two fractions.

Fraction I containing fatty acid methyl esters (FAME) and hydrocarbons was fractionated by elution with 6 ml of a mixture of hexane and MTBE (100:2 v/v) and evaporated to dryness. The residue of fraction I was re-dissolved in 300 μl hexane and 1 μl were analysed by GC.

Fraction II containing minor lipids, for example, free fatty acids (FFA) and sterols, was fractionated by elution with 6 ml hexane-MTBE (70:30 v/v) and then evaporated to dryness. The flask was flushed with argon and the residue of fraction II was re-dissolved in 250 μl of dry pyridine and 50 μl of *N*-methyl-*N*-(trimethylsilyl)fluoroacetamide (MSTFA). Silylation was carried out in a water bath at 70 °C for 15 min. A volume of 1 μl of the sample was analysed by GC-FID.

Volumes of 250 μl phenyl-β-D-glucopyranoside (1 mg·ml^{-1}) and 250 μl *p*-chloro-L-phenylalanine (1 mg·ml^{-1}) were added to 1 ml polar extract from methanol-deionized water solution (80:20 v/v). The polar extract was treated by a silylation procedure by adding 100 μl trimethylsilylimidazole (TMSIM) and 300 μl pyridine and reacted at 70 °C for 15 min in a water bath. The silylated samples were diluted with 300 μl hexane and 300 μl deionized water. The upper, hexane phase containing silylated sugars and sugar alcohols was fraction III and was thus ready for GC analysis.

For fraction IV, 1 ml polar extract was oximated by adding 300 μl hydroxylammonium chloride in pyridine solution (2 mg·ml^{-1}). After oximation for 30 min at 70 °C in a water bath, 100 μl of MSTFA was added and the sample was silylated in a 70 °C water bath for 15 min. The silylated sample was diluted with 500 μl hexane and 300 μl deionized water for selective hydrolysis. The lower phase containing amino acids and organic acids was re-silylated by adding 50 μl MSTFA and 200 μl acetonitrile, reacting at 70 °C for 60 min in a water bath. A volume of 1 μl of the sample was used for GC analysis.

All samples were analysed using HP 6890 Plus equipped with FID (Agilent Technologies, Santa Clara, California, USA). GC-FID data were acquired and integrated using HP-ChemStation A.06.03 (Agilent Technologies). The chromatography column used for analysis was DB-1, 60 m × 0.32 mm internal diameter fused silica capillary coated with a 0.25 μm film of polydimethylsiloxane (Agilent Technologies). Helium was uased at a constant flow rate of 1.8 ml·min^{-1}. The splitless injection was performed at 280 °C. The column temperature was programmed from 100 °C to 320 °C (15 min hold) at 4 °C·min^{-1} for the analysis of fractions I, II, III and IV. Identification of sunflower seed constituents was achieved by comparison of retention times of the chromatographic peak of the analyst to that of reference standards.

Extraction and identification of volatile compounds

For extraction of volatiles, the ground roasted germinated sunflower seeds (2 g) were placed into a headspace bottle, with 50 mg·kg^{-1} ethyl decanoate of sunflower seed (ethyl decanoate 1 mg·ml^{-1} in 10% methanol) as an internal standard. The sample was left for 20 min at 60 °C in the incubator to allow for equilibration of volatiles in the headspace. After the equilibration time, a 50/30 μm divinylbenzene/carboxen/polydimethylbenzene SPME fibre 57348-U (Supelco, Bellefonte, Pennsylvania, USA) was inserted into the vial and exposed to the headspace for 30 min at 60 °C. Polydimethylbenzene was used for nonpolar analytes and divinylbenzene was used for polar analytes, the latter being especially useful for pyrazines. When the process was completed, the fibre was inserted into the injector port of the GC-MS instrument.

The volatile compounds were analysed by Agilent 7890A gas chromatograph equipped with 5975C mass spectrometer (Agilent Technologies). DB-1ms column (60 m × 0.25 mm × 0.25 μm) was used. The volatiles absorbed on the fibre were thermally desorbed in the GC injection port at 250 °C for 20 min with a splitless mode. Helium was operated at a constant flow rate of 1.5 ml·min^{-1}. The initial oven temperature was 50 °C for 1 min, followed by a temperature program of 5 °C·min^{-1} to 100 °C (5 min), 4 °C·min^{-1} to 140 °C (5 min), 5 °C·min^{-1} to 180 °C (2 min) and 10 °C·min^{-1} to 250 °C (7 min). The mass spec-

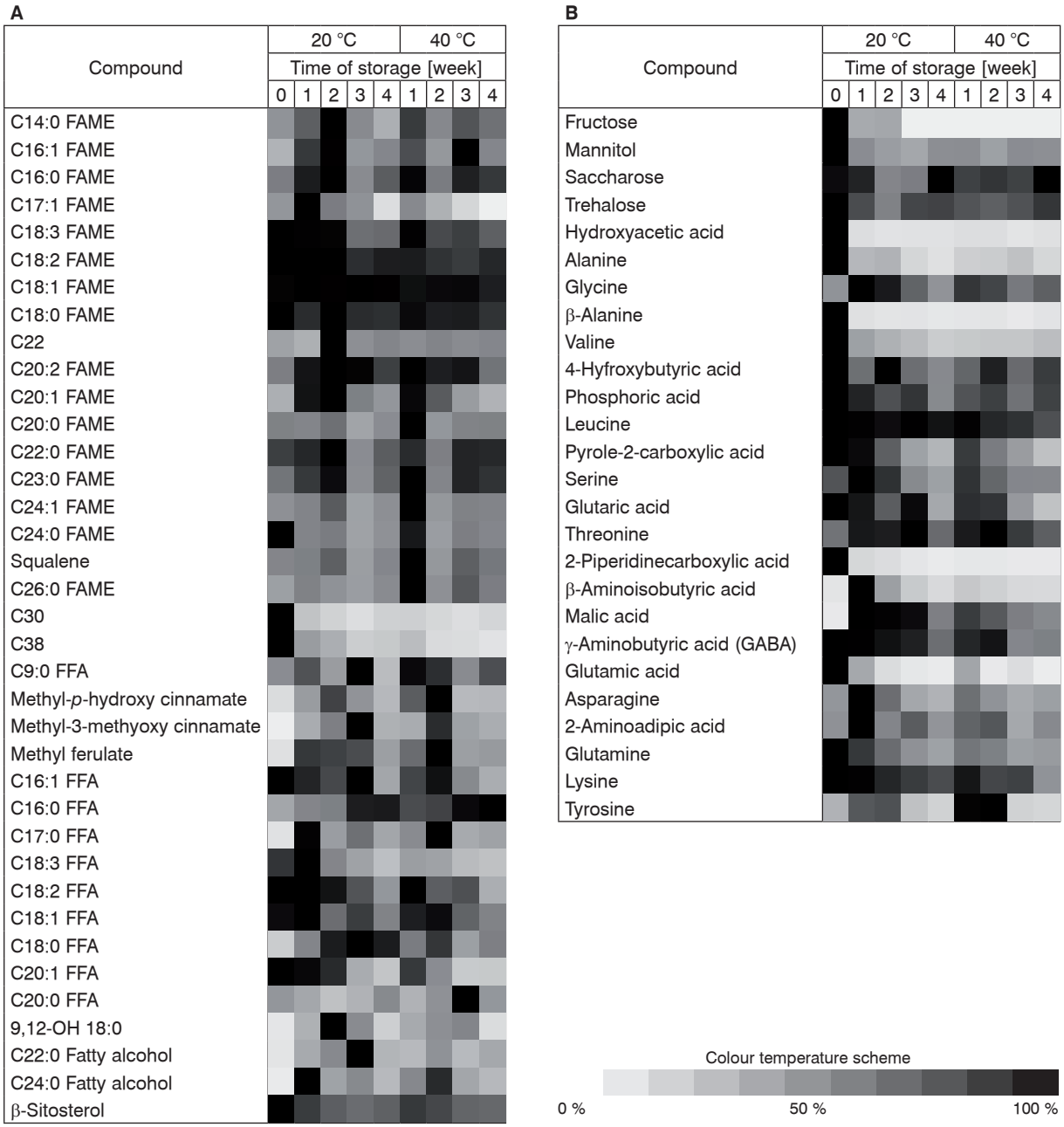

Fig. 1. Heatmap visually depicting the contents of metabolites in roasted germinated sunflower seeds.

A – Fraction I and Fraction II, B – Fraction III and Fraction IV.
Dark colours indicate high values, light-grey colours indicate low values. FAME – fatty acid methyl ester, FFA – free fatty acid.

trometer source temperature was 230 °C and the transfer line temperature was 225 °C, the quadrupole temperature was 150 °C. The ionization voltage was set at 70 eV and the scan range from *m/z* 50 to 550. The reference standards were operated under the same GC-MS conditions described previously, an injection volume of 0.2 μl reference standard mixtures was employed in split mode (split ratio 100 : 1). The temperature program for the *n*-alkane mixture was 5 °C·min^{-1} to 100 °C (5 min), 4 °C·min^{-1} to 140 °C (5 min), 5 °C·min^{-1} to 180 °C (2 min), 10 °C·min^{-1} to 250 °C (7 min), 10 °C·min^{-1} to 280 °C (5 min) and 5 °C·min^{-1} to 300 °C (10 min).

Automated mass spectral deconvolution and identification system (AMDIS, version 2.66; National Institute of Standards and Technology – NIST, Gaithersburg, Maryland, USA) was applied

for automatically extracting pure component mass spectra from highly complex GC-MS data files. The purified spectra were used for a search in a mass spectral library. Identification of individual volatile compounds was accomplished either by analysing pure components under the same experimental conditions or by comparing mass spectra with reference mass spectra from the NIST 11 library (Version 2.0; NIST). All reference standards were prepared in acetone at a concentration of 5 mg·ml^{-1}. Retention indices (*RI*) were calculated based on the analysis of the C6-C26 *n*-alkane series.

Statistical analysis

Peak areas of each metabolite were acquired and integrated by the HP-ChemStation A.06.03 program. Identification was done by comparison with reference standards. Relative quantification was done on a semi-quantitative level based on peak areas of internal standards. Principal component analysis (PCA) was used for the classification of the roasted germinated sunflower seeds based on metabolites and volatile compounds composition. The output from PCA consisted of sore plots to visualize the contrast between individual samples and loading plots to explain the reason for sample separation. Agglomerative hierarchical clustering was used to construct a dendrogram on the base of the similarity between each pair of objects. The significance of the differences was evaluated by one-way ANOVA at a 95% significance level (Tukey's range test). All the statistical analyses were performed by XLSTAT version 2016.7 (Addinsoft, New York, New York, USA).

RESULTS AND DISCUSSION

Multivariable analysis

In this study, the effects of storage temperature and time on the metabolic and flavour profiles of roasted germinated sunflower seeds were studied. Altogether, 63 peaks were identified for the roasted germinated sunflower seeds. These accounted for ~ 50 % of all metabolites in the roasted germinated sunflower seeds. The metabolites were divided into four fractions (Fig. 1). Fraction I contained 16 FAME and 4 hydrocarbons, fraction II contained 17 minor lipid compounds (FFA, fatty alcohols, phytosterol), fraction III contained 4 sugar compounds (sugars and sugar alcohols);

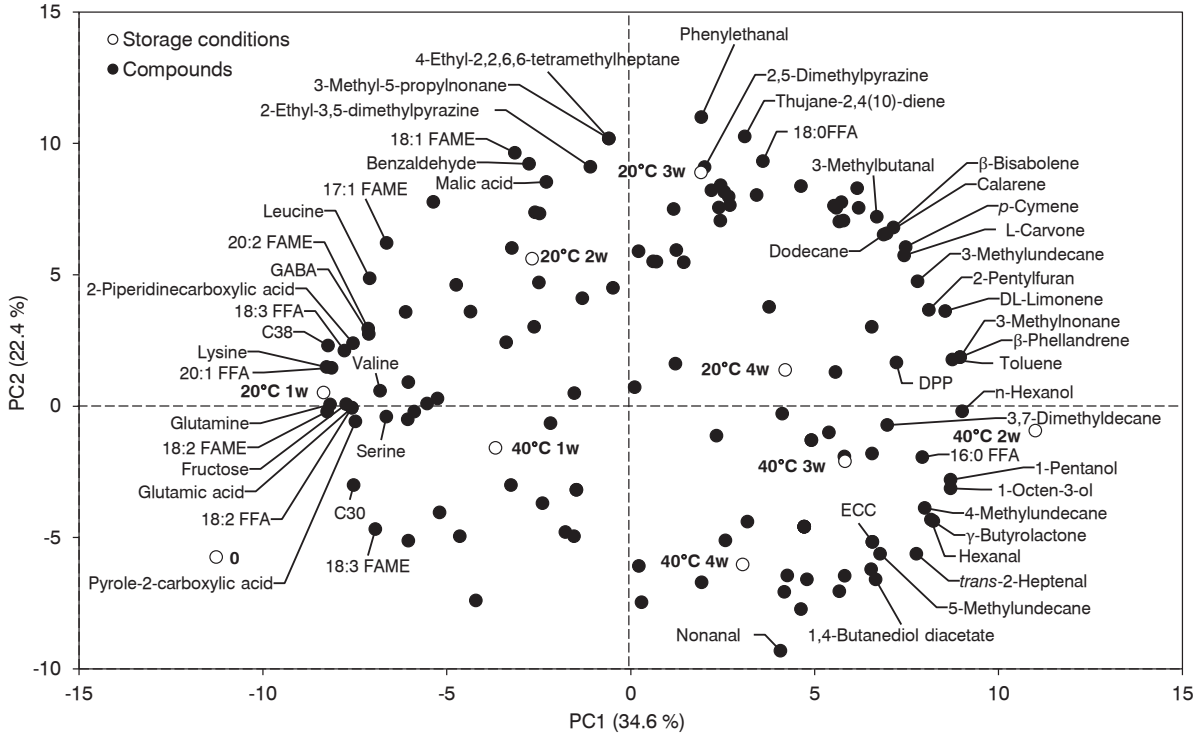

Fig. 2. Biplot of metabolic and flavour profiles of roasted germinated sunflower seeds during storage.

The compound name shown in the figure had a loading score higher than 0.7.
DPP – (*Z*)-2,5-dimethyl-3-(1-propenyl)-pyrazine, ECC – 5-ethyl-1-cyclopentene-1-carbaldehyde, FAME – fatty acid methyl ester, FFA – free fatty acid, GABA – γ-aminobutyric acid, w – week.

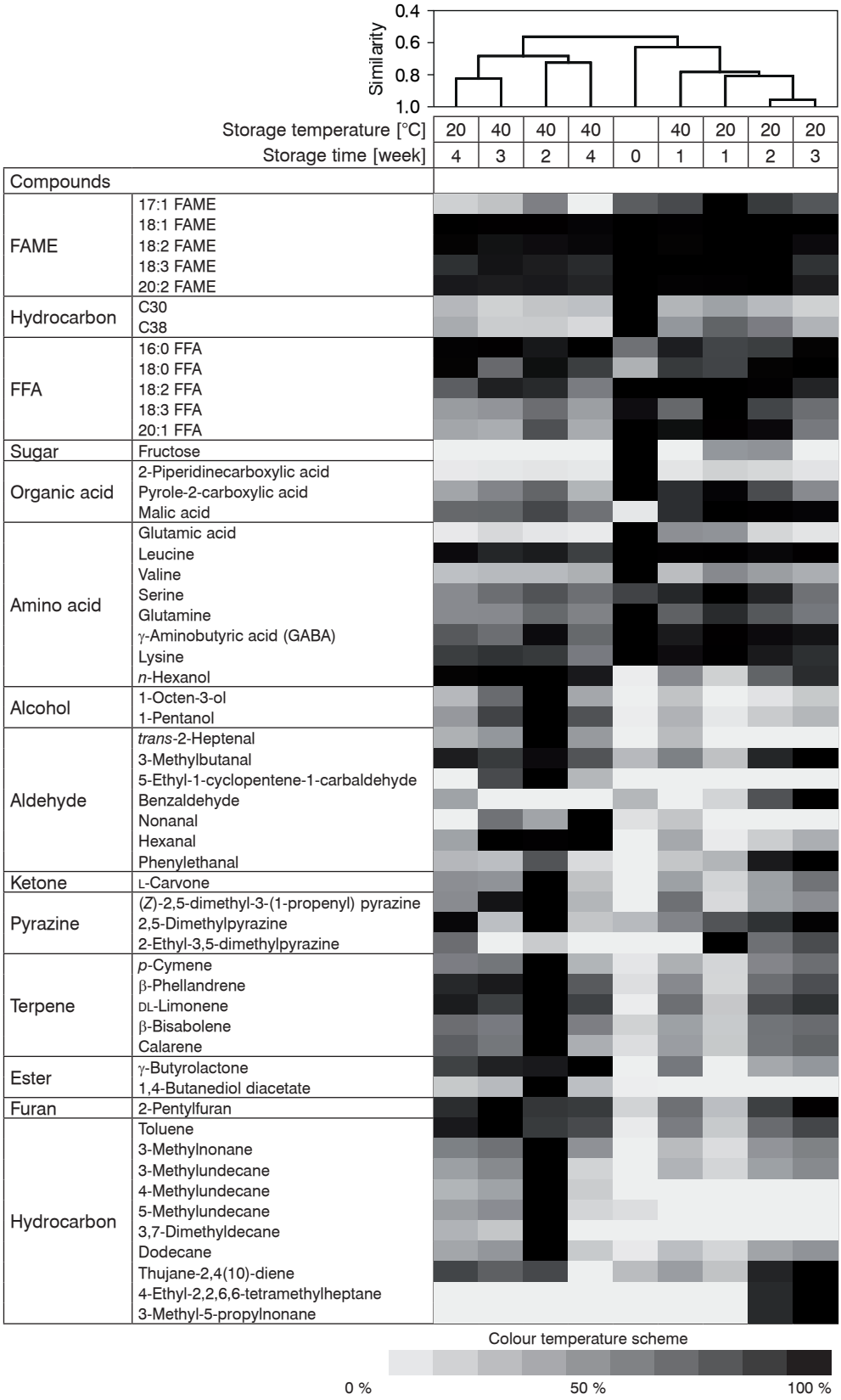

Fig. 3. Heatmap visually depicting the contents of metabolites and volatile compounds, and an agglomerative hierarchical clustering dendrogram of roasted germinated sunflower seeds at different storage conditions.

The compounds with higher loading scores (> 0.7) are presented in the heatmap. Dark colours indicate high values, light-grey colours indicate low values. FAME – fatty acid methyl ester, FFA – free fatty acid.

fraction IV contained 22 acid compounds (organic acids and amino acids). A total of 73 volatile compounds was identified, these compounds being divided into 12 groups: 15 aldehydes, 6 alcohols, 3 ketones, 8 pyrazines, 10 terpenes, 24 hydrocarbons, 3 esters, 1 furan, 2 lactones, and 1 phenol. PCA grouping was based on Spearman correlations conducted on the metabolic and flavour profiles of roasted germinated sunflower seeds. A PCA biplot (Fig. 2) showed that PC1 and PC2 axes represented 57.0 % of total variables, PC1 explaining 34.6 % and PC2 explaining 22.4 % of the total variance. PCA plots utilizing the whole data of the metabolites and volatile compounds detected in the samples showed that the overall profiles were dominated by the storage temperature and time (Fig. 2). Sunflower seed samples stored at 20 °C for 1 or 2 weeks, and at 40 °C for 1 week, were placed on the left side of PC1. The clustering was mainly explained by FAME (C18:2, C18:3), FFA, fructose, glutamine, valine, leucine, serine, γ-aminobutyric acid (GABA) and organic acids (loading value on PC1 from –0.890 to –0.714). The levels of these compounds were higher in the samples stored at 20 °C for 1 and 2 weeks and at 40 °C for 1 week. The loadings plot indicated that C18:1 FAME, C18:0 FFA, malic acid, benzaldehyde, phenylethanal, 2-ethyl-3,5-dimethylpyrazine, 2,5-dimethylpyrazine, which differentiated the samples, were located near the samples stored at 20 °C for 3 weeks, positively correlating to PC2. The positive area of PC1 listed samples stored at 20 °C for 4 weeks and 40 °C for 2, 3 and 4 weeks, which were positively associated with terpenes (β-phellandrene, DL-limonene, β-bisabolene, calarene, *p*-cymene), alcohols (*n*-hexanol, 1-pentanol, 1-octen-3-ol), aldehydes (hexanal, *trans*-2-heptenal, 3-methybutanal), L-carvone, 2-pentylfuran, (*Z*)2,5-dimethyl-3-(1-propenyl)-pyrazine and γ-butyrolactone.

The results were visualized in a heatmap diagram that was combined with agglomerative hierarchical clustering of the selected 55 compounds that had a loading value higher than 0.7 (Fig. 3). Three groups of samples were distinguished. Group I consisted of storage at 40 °C for 2, 3, 4 weeks and at 20 °C for 4 weeks, with high levels of alcohols, terpenes (*p*-cymene, calarene, DL-limonene, β-phellandrene and β-bisabolene), aldehydes (nonanal, hexanal) and hydrocarbons. Group II consisted of the sample without storage, the sample having high contents of fructose, amino acids (glutamic acid, valine, glutamine, lysine and GABA) and organic acids. Group III consisted of samples stored at 20 °C for 1, 2, 3 weeks, and at 40 °C for 1 week, which had high contents of amino acids, FFA (C16:0, C18:0), benzaldehyde and phenylethanal.

Changes of representative metabolites in fractions

The relative contents of FAME from roasted germinated sunflower seeds stored for 1 to 4 weeks at 20 °C and 40 °C are shown in Fig. 4. FAME found in fraction I, as a result of transesterification of the lipid extract, reflect fatty acid contents of the sunflower seed triglycerides. The major fatty acids in sunflower seeds were oleic (C18:1), linoleic (C18:2), linolenic (C18:3), palmitic (C16:0) and stearic acid (C18:0), while C18:1 formed a high proportion. It was previously reported that oxidative stability was improved high oleic acid, thus less painty flavour was developed during storage compared to normal seeds and this led to higher acceptability of seeds and longer

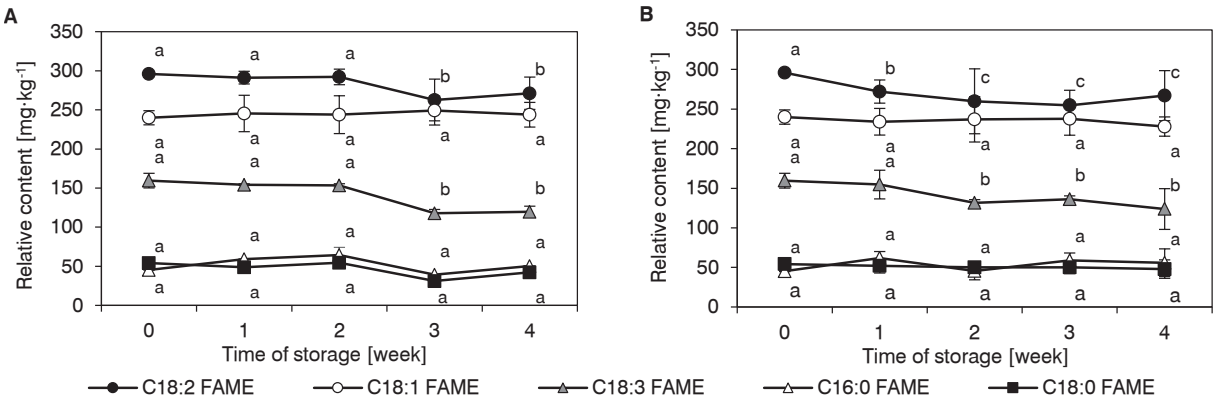

Fig. 4. Relative contents of representative metabolites in fraction I of roasted germinated sunflower seeds.

A – storage at 20 °C, B – storage at 40 °C.
FAME – fatty acid methyl ester.

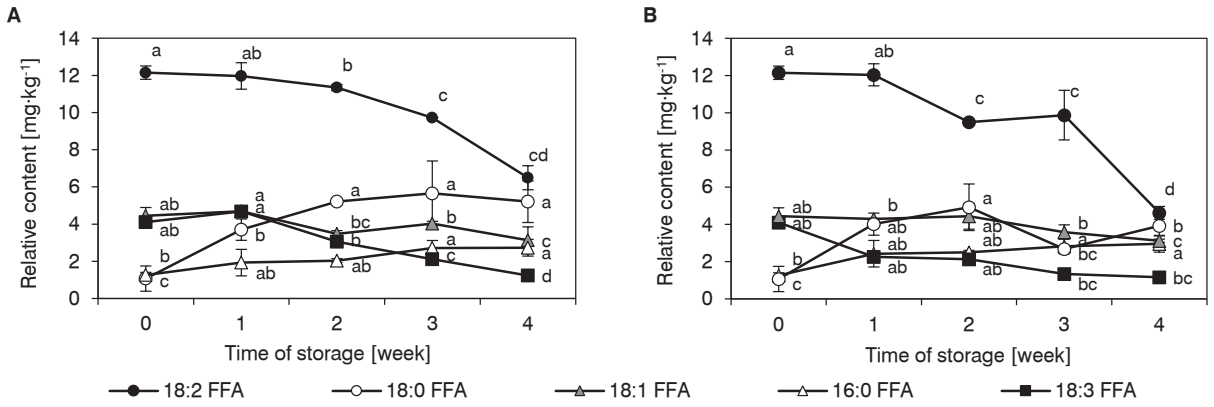

Fig. 5. Relative contents of representative metabolites in fraction II of roasted germinated sunflower seeds.

A – storage at 20 °C, B – storage at 40 °C.
FFA – free fatty acid.

shelf life [17]. In the first two weeks of storage, there were no significant changes in the contents of C18:1, C18:2, C18:3 at 20 °C, but a significant reduction in the contents of C18:2 and C18:3 was observed after 3 weeks at 20 °C. During the storage of sunflower seeds at 40 °C, reduction of C18:2 and C18:3 contents was accelerated. In the case of C16:0 and C18:0, C16:0 content showed a small, insignificant increase at both 20 °C and 40 °C during the initial period of storage. This increase could be attributed to dissociation of the triacylglycerol (TAG) fraction might from other chemical structures, e. g. proteins, thus producing an increase in C16:0 content. Nevertheless, a continuous insignificant decrease in C16:0 content was observed after two weeks of storage. Roasting led to a small, insignificant decrease in C18:0 content during the storage at both 20 °C and 40 °C. These results indicated the hydrolysis of TAG.

Lipid oxidation strongly affects the shelf life

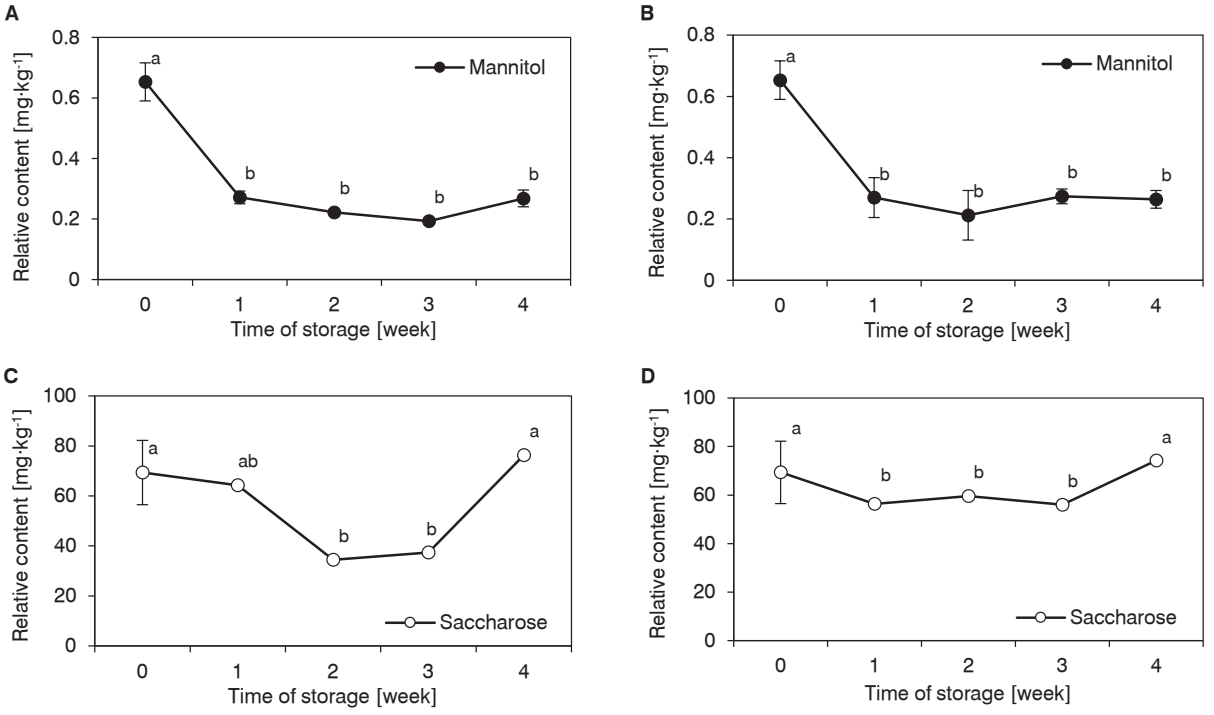

Fig. 6. Relative contents of representative metabolites in fraction III of roasted germinated sunflower seeds.

A, C – storage at 20 °C; B, D – storage at 40 °C.

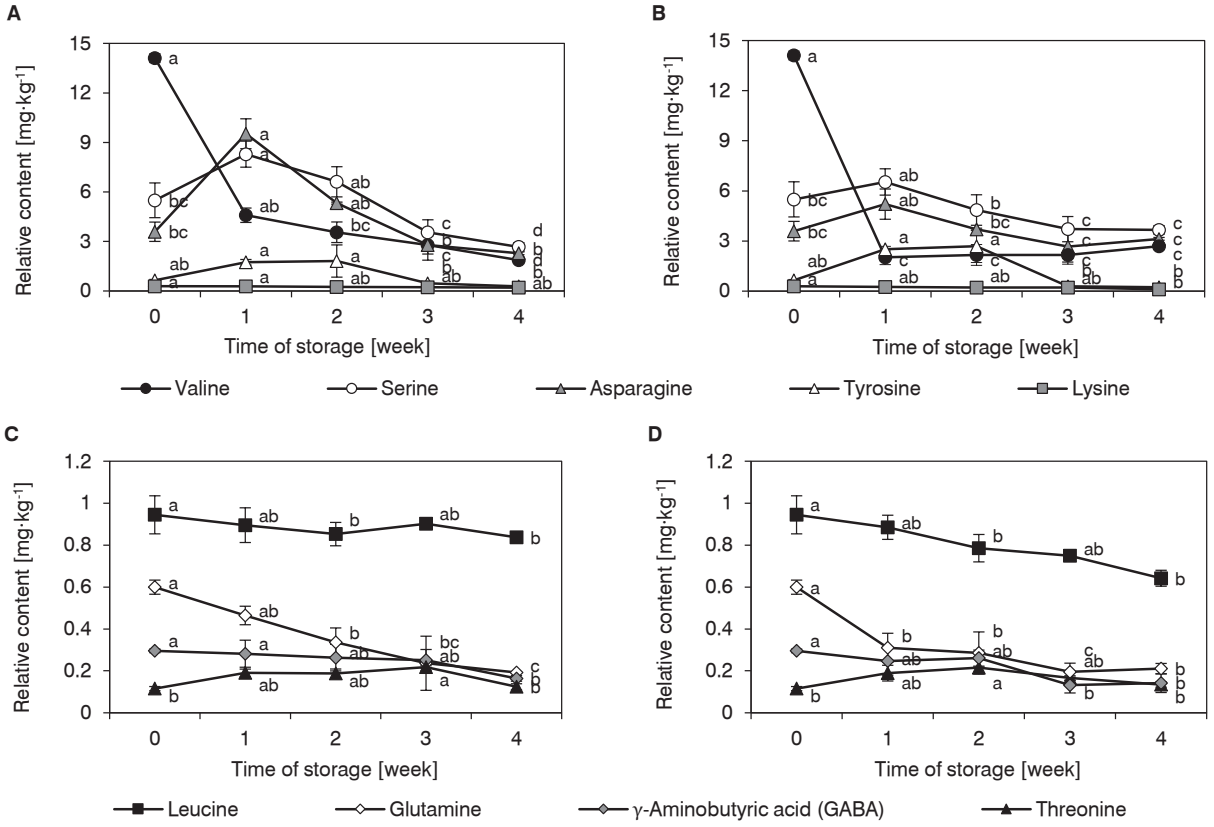

Fig. 7. Relative contents of representative metabolites in fraction IV of roasted germinated sunflower seeds.
A, C – storage at 20 °C; B, D – storage at 40 °C.

and sensory characteristics of oilseeds, depending on many factors such as the content of unsaturated fatty acids, enzymatic activity, mineral composition and the presence of natural antioxidants. FFA are formed due to the hydrolysis of TAG and may get promoted by the reaction of the oil with moisture [18]. Saturated FFA content increased during the storage period. The results showed that the content of FFA (C16:0, C18:0) in sunflower seeds stored at 20 °C was higher than in those stored at 40 °C. The lower storage temperature would promote the activities of hydrolytic enzymes [19], suggesting that regulation of the storage temperature would be one of the most important factors to control FFA accumulation. The contents of C18:1, C18:2 and C18:3 in sunflower seeds decreased during the storage. Furthermore, the storage temperature affected the progress of oxidative degradation of unsaturated fatty acids, as shown in Fig. 5. The decrease in the contents of C18:1, C18:2 and C18:3 in sunflower seeds stored at 40 °C was statistically more pronounced than in those stored at 20 °C. The heat treatment destroys lipase and lipoxygenase activities, but the decrease in unsaturated FFA value during subsequent storage may be due to lipid oxidation [19]. Previous studies reported that the oxidation of unsaturated fatty acids in rice was dependent on the storage temperature [20].

As shown in Fig. 6, during 4 weeks of storage, the contents of mannitol and saccharose decreased significantly ($P < 0.05$). Saccharose content was higher at 40 °C than that at 20 °C. Storage time had a significant influence ($P < 0.05$) on amino acids, the contents of most of them diminished over storage (Fig. 7). The greatest reduction was observed in the contents of valine, glutamine, lysine and leucine. During the storage of foods with sugars or amino acids, the Maillard reaction takes place. In its initial step, Amadori's compounds are produced followed by the formation of melanoidin in subsequent steps [21]. The contents of amino acids serine, tyrosine and asparagine increased during the first week of storage, reaching a maximum value. After two weeks, the contents of these amino acids diminished. A large decrease in the contents of amino acids was observed at 40 °C.

Tab. 1. Contents of volatile compounds of roasted germinated sunflower seeds stored at 20 °C and 40 °C for 4 weeks.

Compounds	*RI*	Relative content [mg·kg^{-1}]								
		20 °C					40 °C			
		0*	1 week	2 weeks	3 weeks	4 weeks	1 week	2 weeks	3 weeks	4 weeks
Isobutanal	629	0.40 ± 0.11	0.62 ± 0.04	2.94 ± 0.20	4.56 ± 0.23	2.32 ± 0.28	1.11 ± 0.05	3.77 ± 0.28	2.19 ± 0.09	1.85 ± 0.33
3-Methylbutanal	646	1.22 ± 0.17	1.01 ± 0.10	6.15 ± 0.30	7.74 ± 0.51	6.38 ± 0.29	2.77 ± 0.20	6.86 ± 0.19	5.46 ± 0.13	4.46 ± 0.14
2-Methylbutanal	662	1.07 ± 0.15	1.20 ± 0.20	6.89 ± 1.17	9.11 ± 1.55	7.56 ± 1.28	3.34 ± 0.28	4.57 ± 0.78	6.80 ± 1.15	4.82 ± 0.41
3-Penten-2-one	711	0.11 ± 0.02	0.19 ± 0.06	0.90 ± 0.20	1.15 ± 0.20	2.40 ± 0.23	0.75 ± 0.16	0.61 ± 0.25	1.72 ± 0.21	1.08 ± 0.21
Pyridine	717	–	0.22 ± 0.09	0.49 ± 0.08	0.63 ± 0.20	0.62 ± 0.11	0.10 ± 0.01	–	0.64 ± 0.10	–
1-Pentanol	744	0.03 ± 0.01	0.07 ± 0.01	0.36 ± 0.06	0.58 ± 0.10	0.95 ± 0.27	0.63 ± 0.05	3.50 ± 0.57	2.31 ± 0.39	2.07 ± 0.18
Toluene	751	0.06 ± 0.01	0.54 ± 0.09	2.45 ± 0.42	3.42 ± 0.58	4.45 ± 1.19	1.99 ± 0.17	3.73 ± 1.03	5.28 ± 0.90	3.32 ± 0.28
Hexanal	757	–	0.53 ± 0.09	4.13 ± 0.70	8.62 ± 1.46	10.10 ± 1.71	9.49 ± 0.81	40.80 ± 6.92	43.37 ± 7.36	43.48 ± 3.69
2-Methylpyrazine	795	0.40 ± 0.07	0.35 ± 0.06	2.88 ± 0.49	3.96 ± 0.67	6.22 ± 1.06	1.90 ± 0.16	4.30 ± 0.73	0.15 ± 0.03	0.12 ± 0.01
Furfural	800	0.36 ± 0.05	0.61 ± 0.10	1.92 ± 0.33	2.64 ± 0.45	–	0.76 ± 0.06	2.86 ± 0.49	–	0.72 ± 0.06
n-Hexanol	849	0.03 ± 0.01	0.27 ± 0.05	1.67 ± 0.28	2.55 ± 0.43	3.05 ± 0.52	1.08 ± 0.09	3.30 ± 0.56	3.32 ± 0.56	2.78 ± 0.24
γ-Butyrolactone	850	–	–	0.48 ± 0.08	0.62 ± 0.10	1.49 ± 0.25	0.87 ± 0.07	1.87 ± 0.32	1.80 ± 0.31	2.22 ± 0.19
Methional	854	0.35 ± 0.09	0.27 ± 0.05	1.57 ± 0.27	1.90 ± 0.32	1.70 ± 0.29	0.39 ± 0.03	0.95 ± 0.16	0.52 ± 0.09	0.60 ± 0.05
2-Heptanone	866	0.07 ± 0.02	–	–	–	–	0.41 ± 0.03	1.01 ± 0.17	0.94 ± 0.16	0.73 ± 0.06
Heptanal	875	–	–	–	–	–	0.27 ± 0.02	0.81 ± 0.14	1.05 ± 0.18	1.04 ± 0.09
2,5-Dimethylpyrazine	883	2.13 ± 0.30	8.03 ± 1.36	10.30 ± 1.75	12.74 ± 2.16	12.48 ± 2.12	4.78 ± 0.41	13.79 ± 2.34	1.91 ± 0.32	1.60 ± 0.14
Ethylpyrazine	885	0.49 ± 0.07	–	–	–	–	0.80 ± 0.07	3.14 ± 0.53	–	0.45 ± 0.04
Benzaldehyde	926	0.20 ± 0.03	0.090	0.720	1.220	0.285	–	–	–	–
trans-2-Heptenal	929	–	–	–	–	1.91 ± 0.32	1.54 ± 0.13	10.19 ± 1.73	2.82 ± 0.48	2.75 ± 0.23
α-Pinene	930	3.58 ± 0.60	4.80 ± 0.82	28.26 ± 4.80	41.83 ± 7.10	43.29 ± 7.35	12.95 ± 1.10	24.70 ± 4.19	33.96 ± 5.19	2.27 ± 0.19
Camphene	943	0.17 ± 0.02	0.12 ± 0.02	0.92 ± 0.16	1.09 ± 0.18	1.08 ± 0.18	0.32 ± 0.03	0.85 ± 0.14	0.86 ± 0.15	0.60 ± 0.05
Thujane-2,4(10)-diene	946	0.11 ± 0.02	0.10 ± 0.02	0.62 ± 0.11	0.78 ± 0.13	0.51 ± 0.09	0.20 ± 0.02	0.50 ± 0.08	0.41 ± 0.07	–
Heptanol	949	–	–	–	–	–	–	0.69 ± 0.12	0.41 ± 0.07	–
Phenol	951	–	–	–	–	–	0.21 ± 0.02	1.41 ± 0.24	–	0.05 ± 0.00
1-Octen-3-ol	959	–	–	0.24 ± 0.04	0.86 ± 0.15	1.28 ± 0.22	0.97 ± 0.08	7.94 ± 1.35	3.69 ± 0.63	1.76 ± 0.15
β-Phellandrene	965	0.08 ± 0.02	0.13 ± 0.02	0.88 ± 0.15	1.19 ± 0.20	1.55 ± 0.26	0.65 ± 0.05	1.97 ± 0.33	1.64 ± 0.28	1.09 ± 0.15
3-Methylnonane	968	–	0.06 ± 0.01	0.36 ± 0.06	0.45 ± 0.08	0.46 ± 0.08	0.19 ± 0.02	1.28 ± 0.22	0.55 ± 0.09	0.38 ± 0.03
β-Pinene	970	0.62 ± 0.11	0.50 ± 0.08	3.02 ± 0.51	3.82 ± 0.65	3.78 ± 0.64	1.51 ± 0.13	4.70 ± 0.80	3.39 ± 0.58	–
2-Ethyl-3-methylpyrazine	974	0.99 ± 0.18	0.50 ± 0.09	2.75 ± 0.47	3.31 ± 0.56	3.06 ± 0.52	1.61 ± 0.14	5.62 ± 0.95	2.64 ± 0.45	0.61 ± 0.05
2-Pentylfuran	976	0.36 ± 0.07	0.66 ± 0.11	3.25 ± 0.55	4.55 ± 0.77	3.66 ± 0.62	2.06 ± 0.17	3.47 ± 0.59	4.77 ± 0.81	3.36 ± 0.28
3(*E*)-3-Methyl-3-nonene	981	0.14 ± 0.03	0.21 ± 0.03	0.97 ± 0.16	1.30 ± 0.22	1.25 ± 0.21	0.57 ± 0.05	1.66 ± 0.28	1.23 ± 0.21	–
Pantoic lactone	987	–	–	–	–	0.62 ± 0.11	0.40 ± 0.03	2.32 ± 0.39	0.79 ± 0.13	–

Tab. 1. *continued*

Compounds	*RI*	Relative content [mg·kg^{-1}]								
		20 °C					40 °C			
		0*	1 week	2 weeks	3 weeks	4 weeks	1 week	2 weeks	3 weeks	4 weeks
4-Decene	989	–	–	–	–	2.61 ± 0.44	0.99 ± 0.08	2.83 ± 0.48	–	–
Phenylacetaldehyde	1007	0.78 ± 0.11	0.71 ± 0.12	3.44 ± 0.58	4.12 ± 0.70	0.68 ± 0.11	0.47 ± 0.04	2.37 ± 0.40	0.57 ± 0.10	0.21 ± 0.08
p-Cymene	1011	0.12 ± 0.02	0.36 ± 0.06	1.79 ± 0.30	2.37 ± 0.40	1.93 ± 0.33	1.01 ± 0.09	5.27 ± 0.89	2.12 ± 0.36	0.92 ± 0.08
D-Limonene	1020	0.13 ± 0.02	2.06 ± 0.35	11.34 ± 1.92	13.67 ± 2.32	15.18 ± 2.58	8.13 ± 0.69	18.30 ± 3.11	12.54 ± 2.13	12.40 ± 1.0
2,2,4,6,6-Pentamethylheptane	1037	–	0.32 ± 0.05	1.87 ± 0.32	2.43 ± 0.41	–	0.42 ± 0.04	4.30 ± 0.73	1.04 ± 0.18	0.15 ± 0.01
2-Ethyl-3,5-dimethylpyrazine	1053	0.73 ± 0.10	5.86 ± 0.99	2.55 ± 0.43	3.62 ± 0.61	2.67 ± 0.45	–	0.60 ± 0.10	–	–
2,6,7-trimethyldecane	1058	–	–	1.15 ± 0.19	1.61 ± 0.27	–	–	–	–	–
5-Ethyl-2,2,3-trimethylheptane	1058	–	0.23 ± 0.04	–	–	1.27 ± 0.22	–	3.39 ± 0.57	1.59 ± 0.27	–
2,4-Dimethyl-1-decene	1065	–	0.18 ± 0.03	0.93 ± 0.16	–	–	–	–	–	–
Nonanal	1081	0.15 ± 0.02	–	–	–	–	0.47 ± 0.04	0.92 ± 0.16	1.68 ± 0.29	4.00 ± 0.34
Verbenol	1128	0.47 ± 0.12	0.44 ± 0.08	1.72 ± 0.29	2.40 ± 0.41	–	0.83 ± 0.07	–	–	–
2,3,5-Trimethyl-6-ethylpyrazine	1148	–	0.18 ± 0.03	0.55 ± 0.09	1.52 ± 0.26	0.75 ± 0.13	–	–	1.45 ± 0.25	–
Pinocarvone	1152	–	0.11 ± 0.02	0.28 ± 0.05	1.06 ± 0.18	0.51 ± 0.09	0.28 ± 0.26	–	0.51 ± 0.25	–
5-Ethyldecane	1155	–	0.14 ± 0.02	0.47 ± 0.08	0.73 ± 0.12	0.63 ± 0.11	0.29 ± 0.02	–	0.62 ± 0.11	0.12 ± 0.01
5-Methylundecane	1157	0.10 ± 0.02	–	–	–	0.54 ± 0.09	–	2.14 ± 0.36	0.67 ± 0.11	0.15 ± 0.01
4-Methylundecane	1161	–	–	–	–	0.44 ± 0.07	–	2.49 ± 0.42	0.58 ± 0.10	0.24 ± 0.02
3-Methylundecane	1177	–	0.32 ± 0.05	1.15 ± 0.19	1.52 ± 0.26	1.16 ± 0.20	0.84 ± 0.07	4.72 ± 0.80	1.52 ± 0.26	0.36 ± 0.03
Myrtenol	1178	0.21 ± 0.02	0.16 ± 0.03	0.54 ± 0.09	0.63 ± 0.11	–	0.61 ± 0.05	–	1.12 ± 0.19	–
2,5-Dimethyl-3-[(1*E*)-1-propenyl] pyrazine	1181	–	0.07 ± 0.01	0.28 ± 0.05	0.36 ± 0.06	0.37 ± 0.06	0.51 ± 0.04	1.17 ± 0.20	1.00 ± 0.17	0.20 ± 0.02
1-Dodecene	1189	0.11 ± 0.03	–	–	–	–	0.32 ± 0.03	2.23 ± 0.38	0.41 ± 0.07	0.24 ± 0.02
Dodecane	1201	0.09 ± 0.02	0.44 ± 0.07	1.42 ± 0.24	1.81 ± 0.31	1.40 ± 0.24	0.88 ± 0.07	6.22 ± 1.06	1.76 ± 0.30	0.66 ± 0.06
3-Methyl-2(*E*)-undecene	1203	–	–	–	–	–	–	–	1.07 ± 0.18	0.10 ± 0.01
L-Carvone	1227	–	0.24 ± 0.04	0.81 ± 0.14	1.40 ± 0.24	1.02 ± 0.17	0.81 ± 0.07	3.34 ± 0.57	0.98 ± 0.17	0.43 ± 0.04
3-Methyltridecane	1368	–	–	–	–	–	–	0.96 ± 0.16	–	0.19 ± 0.02
1-Tetradecene	1388	0.10 ± 0.01	–	–	–	–	–	0.64 ± 0.11	–	0.15 ± 0.01
Tetradecane	1399	–	0.12 ± 0.02	–	–	0.26 ± 0.04	0.26 ± 0.02	1.71 ± 0.29	0.52 ± 0.09	0.39 ± 0.03
Calarene	1433	0.09 ± 0.01	0.23 ± 0.04	0.85 ± 0.14	1.05 ± 0.18	1.11 ± 0.19	0.53 ± 0.05	2.08 ± 0.35	0.83 ± 0.14	0.41 ± 0.04
Chamigrene	1456	–	–	–	–	–	–	0.42 ± 0.07	–	0.17 ± 0.01
β-Bisabolene	1499	0.09 ± 0.02	0.12 ± 0.02	0.50 ± 0.08	0.56 ± 0.09	0.54 ± 0.09	0.29 ± 0.02	1.20 ± 0.20	0.46 ± 0.08	0.44 ± 0.04

Content is expressed as mean ± standard deviation (n = 3). *RI* – retention index, * – without storage, (–) – not detected.

Changes in flavour profiles during storage

Data on the relative contents of volatile compounds of roasted germinated sunflower seeds during storage are shown in Tab. 1. Oxidation of polyunsaturated fatty acids produces monohydroperoxides that become precursors for volatile aldehydes such as hexanal, octanal, nonanal, or decanal [22–26]. Oxidation of fats and the rate of rancidity development are highly dependent on the storage temperature [23]. With prolonged storage, aliphatic aldehydes contents increased dramatically, in particular at temperatures exceeding 43 °C when aging reaction rates are higher [23]. The contents of hexanal, heptanal and nonanal increased significantly during storage. Hexanal is normally regarded as the oxidation products of linoleic acid from the precursor 3-hydroperoxides [24] Sterol oxidation in ready-to-eat infant foods during storage. The content of hexanal increased with increasing temperature and time, ranging from 0.53 mg·kg^{-1} to 10.10 mg·kg^{-1} at 20 °C and from 9.49 mg·kg^{-1} to 43.48 mg·kg^{-1} at 40 °C. Heptanal is formed through oxidation of both oleic and linoleic acids [25]. Heptanal was not detected at 20 °C, but its content increased and reached the maximum at 3 weeks of storage (1.05 mg·kg^{-1}) at 40 °C. Nonanal originates from 9- and 10-hydroperoxides during autoxidation of oleic acid [25]. Nonanal showed the same behaviour as heptanal, being undetected at 20 °C but starting to appear at 40 °C after 1-week storage, and reaching the highest content of 4.00 mg·kg^{-1} at 4 weeks. Fritsch et al. [26] studied the shelf life of roasted sunflower kernels and reported that using peroxide values to predict the shelf life was considerably less accurate than and not as reliable as hexanal content. Therefore, those authors concluded that hexanal content was a better indicator of the shelf life of roasted sunflower kernels than the peroxide value. The authors concluded that a hexanal content of 6 mg·kg^{-1} was the endpoint of the shelf life of roasted sunflower seed kernels. The content of 1-octen-3-ol increased significantly between 3 and 4 weeks of storage at 20 °C, while increased more between 2 and 3 weeks at 40 °C. A similar trend was observed for 1-pentanol and hexanol.

Ketones showed a low relative proportion of the volatile compounds in roasted sunflower seeds. Only 3-penten-2-one and L-carvone were detected during the storage period. The content of 3-penten-2-one increased to 2.40 mg·kg^{-1} at 20 °C and to 7.49 mg·kg^{-1} at 40 °C of storage. The content of γ-butyrolactone increased during the storage, a higher increase being observed at 40 °C.

Terpenes including α-pinene, β-pinene, β-phellandrene, D-limonene, and β-bisabolene are present in the oils of a wide variety of plants [27]. These compounds were the major terpenes determined in the roasted germinated sunflower seeds. Their contents increased during storage and reached a maximum in 2 to 3 weeks at both temperatures of storage (Tab. 1).

The volatile carbonyl compounds detected in roasted sunflower seed were 2-methylbutanal, 3-methylbutanal and furfural (Tab. 1). The contents of these compounds increased during storage. 2-Methylbutanal is produced through a Strecker reaction. The content of 2-methylbutanal increased during up to 3 weeks both at 20 °C and 40°C and decreased thereafter, however, the content remained higher compared to the first week. Furfural content showed an increase during up to 3 weeks at 20 °C, but after that, it remarkably decreased and could not be detected at 4 weeks. At 40 °C, the content of furfural increased at 2 weeks and decreased thereafter. α-Pinene and β-pinene contents increased during storage (Tab. 1).

The contents of 2-methylpyrazine and 2-ethyl-3-methylpyrazine, compounds known as contributing to nutty and roast aroma [28, 29], increased during storage at 20 °C. However, their contents increased in the initial period of storage but then decreased at 40 °C. 2,5-Dimethylpyrazine content increased significantly ($P < 0.05$) at 20 °C, but decreased after 3 weeks of storage at 40 °C. 2-Ethyl-3,5-dimethylpyrazine content increased significantly ($P < 0.05$) at 20 °C, but was only detected at 2 weeks of storage at 40 °C. Similar to our results, other researchers observed a reduction of pyrazine contents, which was possibly caused by free radicals and hydroperoxides from lipid oxidation [30, 31]. However, another work showed that pyrazine contents did not decrease with storage [32]. These differences may result from the different model systems and methods for quantification of volatiles that were used [30].

CONCLUSIONS

The study demonstrated that storage temperature and time significantly affected the metabolites and flavour profiles of roasted germinated sunflower seeds. A broad spectrum of lipophiles (mainly FAME, hydrocarbons and fatty alcohols) and hydrophiles (mainly sugars, organic acids and amino acids) detected by GC-FID included 63 identified metabolites. Additionally, 73 volatile compounds were identified by HS-SPME-GC-MS. The samples were well distinguished based on PCA and cluster analysis. Compared with 20 °C,

the storage temperature of 40 °C caused a decrease in FFA, amino acids, GABA and organic acids contents during storage. The contents of pyrazines increased greatly at 20 °C, but decreased with the storage time. On the contrary, hexanal, heptanal and nonanal, formed as a result of lipid oxidation, showed higher contents at a storage temperature of 40 °C, which was considered as a potential indicator of the quality of roasted germinated sunflower seeds. Our results suggest that the roasted germinated sunflower seeds stored at 20 °C better maintained the nutrients and roast aroma.

Acknowledgments

We acknowledge the financial support of AI Scholarship 2016 (Kasetsart University, Bangkok, Thailand). Authors thank Prof. Utai Klinkesorn (Bangkok) and Prof. Yaowapa Lorjaroenphon (Bangkok) for their valuable comments and suggestions.

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Received 22 April 2020; 1st revised 17 June 2020; accepted 29 July 2020; published online 11 August 2020.