



ENGINEERING SCIENCES

Volatile Compounds of Different Fresh Wet Noodle Cultivars Evaluated by Headspace Solid-Phase Microextraction-Gas Chromatography-Mass Spectrometry

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Abstract: This study was carried out to determine the volatile compounds from four samples of fresh wet noodles and the changes in the volatile compound composition during the storage process. The volatile compounds from four samples of fresh wet noodles were characterized by headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME/GC-MS). The compositions of the volatile compounds varied among fresh and cooked wet noodles made from the raw potato/wheat flour or wheat flour. A total of 194 volatile compounds were detected in the raw potato noodles, main volatiles including aldehydes, alcohols, ketones, esters and organic acids. The total volatile compounds in the potato/wheat flour noodle samples contained mainly aldehyde compounds and were greater than those in the wheat noodles. The total volatile compounds in the cooked noodle samples were greater than those in raw noodle samples. Alcohols and ketones were the least common types of volatile substances in the samples at 0 h. During storage time, alcohols and ketones were increased in volatile substances, and the amount of acids increased dramatically. The results indicated that the aroma of fresh wet noodles was affected by the storage process.

Key words: Potato flour, fresh wet noodles, HS-SPME/GC-MS, volatile compounds.

INTRODUCTION

Asian noodles have been in existence for thousands of years. There are many different types of noodles, including chilled, fresh, dry, etc. (Fu 2008, Giannetti et al. 2014). Noodles are an important staple food in Asian countries. They are convenient, nutritious and delicious (Ahmed et al. 2015, Kaur et al. 2016).

Currently, to enrich the types of noodles, wheat noodles have been added a variety of ingredients. The researchers have tried to prepare wheat noodles by adding flour from rice, oat, barley, sweet potato, potato and buckwheat (Ahmed et al. 2015, Huayin et al. 2017, Mitra et

al. 2016, Montalbano et al. 2016, Ndayishimiye et al. 2016). Moreover, appropriate to add these ingredients to noodles is acceptable, can also provide a better nutritional value (Ahmed et al. 2015).

Wheat noodles are made from four basic ingredients: including flour, water, salt and alkali. There are two distinct types of wheat-flour noodles: alkaline noodles and common salted noodles (Fu 2008, Hou 2010). However, using these gluten-free alternatives to small wheat flour usually has a negative effect on the quality of the noodles, including colour, sensory, cooking and flavour characteristics. Nevertheless, noodles with a suitable addition

of these ingredients is acceptable and provides for better nutritional value.

Potato (*Solanum tuberosum* L.), is the fourth most important crop after rice, wheat and corn in the world. It is a tuber plant, which is mainly used for food and contains a variety of nutrients such as starch, minerals, dietary fibre, vitamins, amino acids, and phenolic compounds (Akyol et al. 2016, Bártová et al. 2015, Burlingame et al. 2009, Ezekiel et al. 2013). As the world's largest produced non-grain crop, The potato tuber as a human food crop, second only to rice and wheat in the world, has an important place in the human diet (Camire et al. 2009, Singh et al. 2012).

The three major components of flavour are taste, aroma and texture. All three components interact to produce a flavour response. Flavour precursors synthesized by the plant, found in raw potatoes, are made up of sugar, amino acids, RNA and lipid. Plant genotype, production and storage environment affect the levels of these chemicals and the enzyme that reacts to them. During cooking, the flavour precursor reacts to Maillard reaction compounds and degradation product sugars, lipids and RNA (Morris et al. 2010).

Potato tubers do not give off volatile aromatic at maturity. Conversely, aroma compounds develop when tuber tissues are sliced and heated. Cooked potatoes can generate hundreds of aromatic compounds (Coleman et al. 1981, Zhao et al. 2017), which show a wide range of concentrations and odour thresholds. The methods including solid-phase microextraction (SPME), simultaneous distillation and extraction (SDE), and solvent-assisted flavor evaporation (SAFE), are used for the analysis of aroma compounds of potato products (Majcher & Jeleń 2009). The most abundant aromatic compounds are the oxidation products of the fatty acids in potato chips (Pangloli et al. 2006). Sugars and amino acids are also important in

the development of flavour compounds in chips (Dresow & Bohm 2009). The important volatile compounds of cooked potato were reviewed (Jansky 2010).

Gas chromatography (GC) is often chosen as the analytical technique for the determination of volatile compounds, using mass spectrometry (MS) or flame ionization detector (FID) (Cserháti 2010). Headspace solid-phase microextraction (HS-SPME) mainly consists of three steps as follows: once the equilibrium is reached, the sample is extracted from the headspace with a fiber and then directly injected into the GC normally with the MS detector (Lu & Zhang 2014, Miao et al. 2016, Ruiz et al. 2003).

Oh (1983) and Ross (2006) measured the textural characteristics of cooked noodles. However, there are few studies on the characterize the volatile compounds in fresh wet noodles. In this paper, we characterized the volatile compounds of fresh wet potato noodles with different storage time using HS-SPME coupled with GC-MS to provide a reference source for studying the quality of fresh-wet potato noodles. This paper mainly studies the noodles produced from potato flour and different flavours between potato noodles and wheat noodles.

MATERIALS AND METHODS

Noodle Materials

Four varieties of fresh wet noodles were prepared for the experiment (Table I). All of the varieties of fresh wet noodles were managed under the same conditions. Sample 1, sample 2, and sample 3 were potato noodles with different amounts of salt and alkali added. Sample 1 and sample 4 did not contain salt and alkali. The fresh wet noodle samples were immediately prepared for analysis.

Table I. Sample information of four varieties of fresh wet noodle

	Potato flour added (g)	Wheat flour added (g)	Distilled water added (g)	Salt added (g)	alkali added (g)
Sample 1	40	60	56	0	0
Sample 2	40	60	56	0.2	0
Sample 3	40	60	56	0.2	0.1
Sample 4	0	100	33	0	0

Sample Extraction

Using an SPME fiber for HS-SPME. Two centimeters of fiber had a 50/30 μm divinylbenzene/Carboxen/polydimethylsiloxane (DVB/Carboxen/PDMS) coated with a fused silica core (Agilent Inc., USA) (Zhang et al. 2014). The fiber has high sensitivity and reproducibility to a variety of volatile compounds.

Five grams of the sample was placed in a 20-mL capped vial with 1 μL of 0.816 $\mu\text{g}/\text{mL}$ 2,4-dimethyl-3-heptanone hexane solution as the internal standard (Zhao et al. 2017), as 2,4-dimethyl-3-heptanone can be well separated and dissolved resolved for GC analysis under the experimental conditions established in this study. The samples were placed in a water bath of 50 $^{\circ}\text{C}$. After balanced 20 minutes and the headspace of the capped vial for 40 minutes. The injection port of the GC and desorption was set at 260 $^{\circ}\text{C}$ 5 min in splitless mode (Zhao et al. 2017).

GC-MS Analysis

Analysis of flavour compound was implemented following the method of GC-MS using an Agilent 7890B gas chromatograph equipped with a DB-WAX capillary column (30 m \times 0.25 mm \times 0.25 μm), which is well suitable to analyze complex volatile compounds, coupled to an Agilent 5977A MSD (Agilent, USA). The oven program was as follows:

The column was set at an initial temperature of 40 $^{\circ}\text{C}$ (3 min), increased to 200 $^{\circ}\text{C}$ at 5 $^{\circ}\text{C}/\text{min}$, 200 $^{\circ}\text{C}$ to 230 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C}/\text{min}$, and held at 230 $^{\circ}\text{C}$ for 3 minutes. The electronic impact (EI) voltage was 70 eV, scanned in the range of 55-500 m/z.

Volatile compounds present were preliminarily identified and were made with the data system library (NIST 11). The values obtained were compared to known RIs (NIST 11) under the same conditions to match the compounds. The internal standard was 2-methyl-3-Heptanone, and the concentration was 0.816 $\mu\text{g}/\text{mL}$.

Statistical Analysis

Quantified GC peak areas of identified compounds of each noodle were analysed using multivariate analysis; statistical analysis was carried out using SPSS 18.0 for Windows.

RESULTS AND DISCUSSION

Volatile compounds identified in potato/wheat flour and wheat flour noodle samples

In a preliminary sensory experiment, the overall volatile compounds of fresh noodles with wheat flour and potato flour were compared. The SPME-GC/MS experiments were carried out with raw and cooked wheat flour noodles and potato/wheat flour noodles. Extracted compounds belonged to different chemical

classes and came either from different reaction pathways during cooking or from raw noodles. A total of 194 volatile compounds were detected by GC-MS in the raw potato noodles, including 28 aldehydes, 52 alcohols, 14 ketones, 15 esters, 11 organic acids, 1 phenolic, 5 ethers, 1 furan, 34 hydrocarbons, and 33 other compounds. Of these 194 compounds, 90 of the compounds were tentatively identified in raw potato noodles, 123 of the compounds were identified in cooked potato noodles, 57 of the compounds were identified in raw wheat noodles, and 47 of the compounds were identified in cooked wheat noodles (Fig. 1). Differences in the volatile compound compositions of the produced noodles were observed when the potato flour was introduced into the wheat flour noodles and were evaluated in terms of nature and relative abundance of all the detected compounds in both raw and cooked potato/wheat flour and wheat flour noodles samples. Among the identified volatile compounds, aldehydes, alcohols and ketones were the largest groups.

The volatile compounds of the raw noodles are shown in Figure 2. Sample 1 contained 54 volatile compounds, including 16 aldehydes, 6 alcohols, 13 ketones, 1 ester, 1 organic acid, 2 phenolics, 1 ether, 1 furan, 8 hydrocarbons, and 5 other compounds. Sample 2 contained 49 volatile compounds, including 12 aldehydes, 6 alcohols, 3 ketones, 4 esters, 1 organic acid, 2 ethers, 1 furan, 12 hydrocarbons, and 8 other compounds. Sample 3 contained 69 volatile compounds, including 11 aldehydes, 16 alcohols, 7 ketones, 4 esters, 3 organic acids, 1 ether, 1 furan, 14 hydrocarbons, and 12 other compounds. Sample 4 contained 57 volatile compounds, including 7 aldehydes, 14 alcohols, 4 ketones, 5 esters, 2 organic acids, 5 ethers, 1 furan, 10 hydrocarbons, and 9 other compounds.

The volatile compounds of the cooked noodles are shown in Figure 3. Sample 1 contained 69 volatile compounds, including 17 aldehydes, 16 alcohols, 5 ketones, 3 esters, 3 organic acids, 1 ether, 1 furan, 11 hydrocarbons, and 12 other compounds. Sample 2 contained

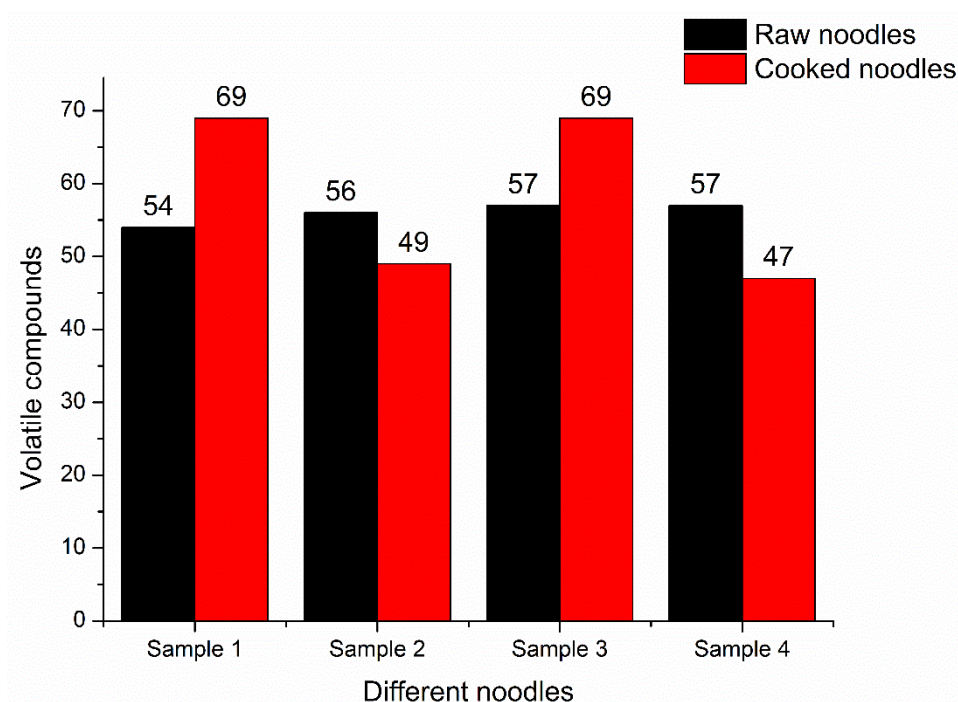


Figure 1. Sum of volatile compounds identified in four different noodle samples.

49 volatile compounds, including 12 aldehydes, 6 alcohols, 3 ketones, 4 esters, 1 organic acid, 2 ethers, 1 furan, 12 hydrocarbons, and 8 other compounds. Sample 3 contained 69 volatile compounds, including 11 aldehydes, 16 alcohols, 7 ketones, 4 esters, 3 organic acids, 1 ether, 1 furan, 14 hydrocarbons, and 12 other compounds. Sample 4 contained 47 volatile compounds, including 9 aldehydes, 7 alcohols, 4 ketones, 2 esters, 1 ether, 1 furan, 14 hydrocarbons, and 9 other compounds.

Aldehydes

Aldehydes were the dominant volatile compounds in the noodles (Figs. 1 and 2). The content of total aldehydes varied with the different raw materials. The proportion of aldehydes accounted for close to 30% of the volatile components in different noodles, of which potato noodles without salt or alkali had a greater proportion of aldehydes than other

noodle samples. However, for sample 4, the proportion of aldehydes was approximately 10%. Petersen et al. (1999) found that the proportion of aldehydes was 94.40% of the total detected compounds in cooked potatoes. A greater proportion of aldehydes were detected in potato noodles than in wheat noodles.

Alcohols

Overall, the content of total alcohols varied with the different noodles. Alcohols accounted for approximately 10% of the total volatile content of the potato noodles, exception for the raw noodle sample 2, which had a content of 25%. However, alcohols accounted for more than 20% of the total volatile content of the cooked potato noodles, except sample 4, which had a content of 14% (Fig. 1).

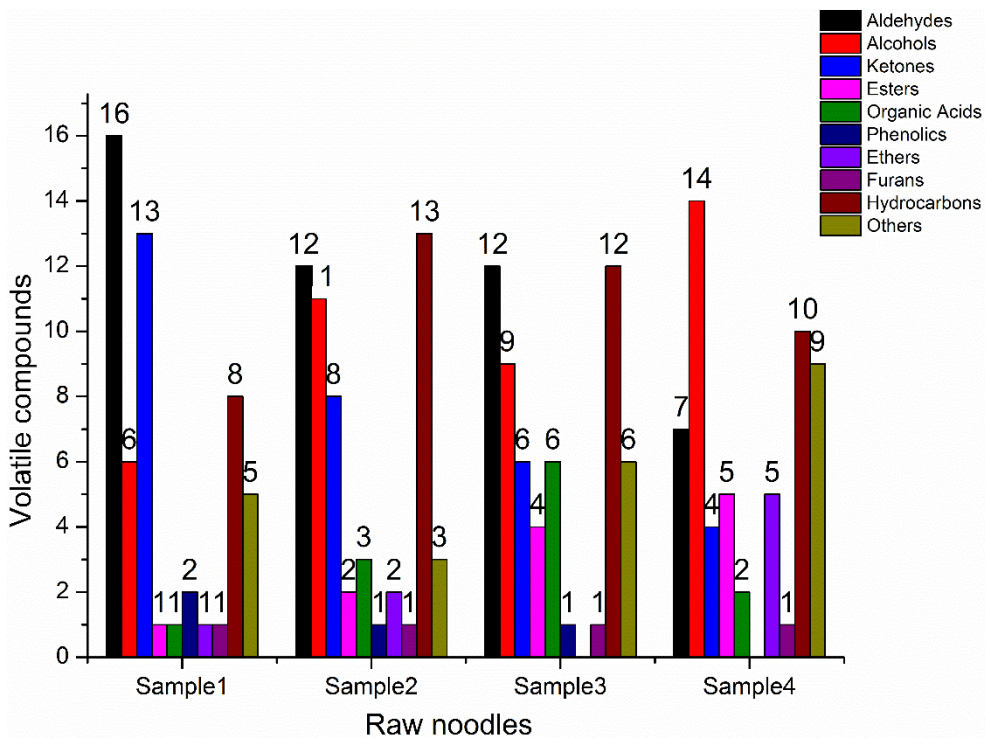


Figure 2. Qualitative descriptive analysed of volatile compounds on raw fresh wet noodle samples by HS-SPME/GC-MS analysis.

Ketones

Overall, the ketones have a certain effect on the volatile profiles of noodles. The most relevant compounds were found in the raw noodle sample 1 and sample 3 at 23%, while other noodles contained less than 10 ketones, which corresponded to the relative contents of more than 10% of total volatile compounds (Fig. 1). The most common ketone detected in the four noodle samples was (E, E)-3,5-octadien-2-one, whereas 6-methyl-5-hepten-2-one, 4-chlorobutyrophenone, 2-octanone and 3-octen-2-one were the other main ketones detected. The well-known aromatic compound 6-methyl-5-heptene-2-one had a citrus odour and was detected in almost all studies.

Esters

It's not found that the ester is the main volatile compounds in the noodles, and the average

contents are less than 10 species in any of the noodle samples. The raw noodle sample 4 did have a relatively high content of esters in the total volatile compounds. However, the cooked noodle sample 4 had fewer esters than the raw noodle sample 4.

Organic acids

In total, 11 organic acid compounds were detected in this study. Except cooked noodle sample 4, the most common organic acids detected in the four noodle samples were acetic acid and hexanoic acid.

Ethers

Ether compounds were found in the noodles. The raw noodles of sample 4 had the greatest content of esters. However, the cooked noodle sample 4 had less ester content than the raw noodles and contained no heterocyclic compounds.

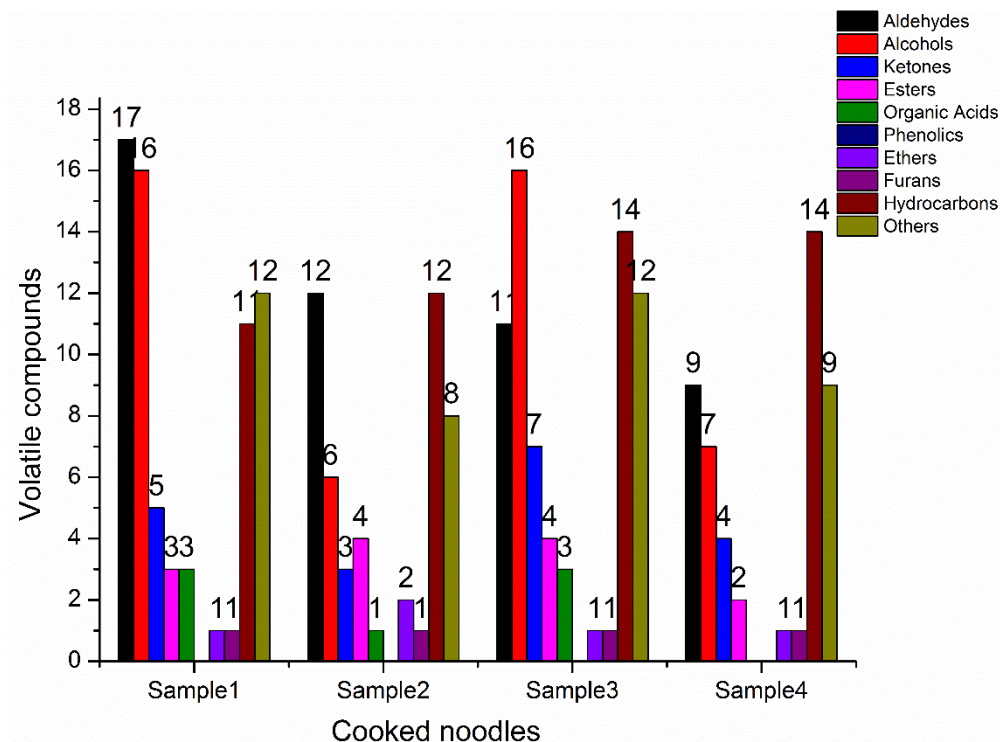


Figure 3. Qualitative descriptive analysis of volatile compounds on cooked fresh wet noodle samples by HS-SPME/GC-MS analysis.

Furan. Lactones

The only furan compound identified in this study was 2-pentyl-furan.

Hydrocarbons

Hydrocarbons are considered one of the most abundant volatile compounds in all noodles. In this study, except for the slightly lower content in the raw noodle sample 1, which contained 8 hydrocarbon compounds, the hydrocarbons in the noodle samples were contained more than 15% of the total volatile compounds, even though 34 different norisoprenoids were tentatively identified.

Other compounds

Other compounds are also responsible for the flowery aroma of noodles. The most abundant terpenes were benzodiazepines and heterocyclic compounds, which were detected in all noodles.

Volatile compounds identified in cooked and raw potato/wheat flour noodle samples

The volatile compounds in cooked and raw potato/wheat flour noodle samples are shown in Figure 4. According to the GC-MS results, the sum of volatile compounds in potato/wheat flour noodle samples was higher for cooked noodles than for raw noodles; the sum of volatile compounds in the cooked noodles was 69 and the sum of volatile compounds in the raw noodles was 57. All kinds of typical flavour compounds were changed between cooked and raw noodles. The aldehydes were slightly changed between the cooked and raw forms of the potato/wheat fresh wet noodles. Alcohols increased in the cooked noodle samples more than in the raw noodle samples. The acids decreased in the cooked noodle samples more than in the raw noodle samples.

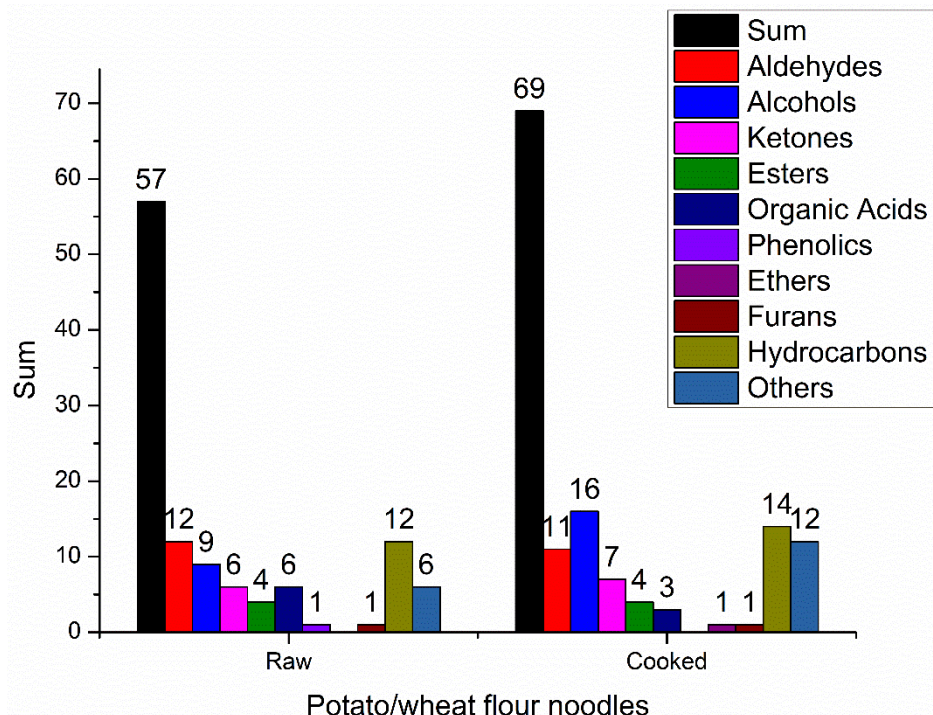


Figure 4. Comparison the contents of the volatile compounds in raw and cooked potato/wheat fresh wet noodles.

Volatile compounds in potato/wheat fresh wet noodles at different storage times

According to the GC-MS results, there were changes in volatile compounds during the storage process. The composition and volatile substances of the samples showed a change during storage. Representative GC-MS TIC trace and the relative content of volatile compounds are shown in Table II. The main odorants detected in the noodle samples were identified by mass spectra (NIST and Wiley libraries), and retention indexes (RIs). Eight kinds of typical flavour compounds were identified during the storage process, namely, aldehydes, alcohols, ketones, esters, acids, phenolics, ethers and

other aromatic compounds. These compounds would be expected to strongly affect the aroma and flavour of potato/wheat fresh wet noodles during the storage process. Alcohols and ketones were the least common types of volatile substances in the samples at 0 h. During storage time, the number of aldehydes changed slightly and the number of acid increased dramatically. Some volatile compounds decreased incessantly, while others increased primarily and then either stayed stable or declined incessantly. Different volatile compounds might be caused by the diverse rates of enzymatic synthesis and hydrolysis or chemical hydrolysis.

Table II. Relative contents of volatile compounds in potato/wheat fresh wet noodles at different storage times (the concentration: ng/g).

volatiles compounds		relative odor concentration ^a										identification	
		0 h	3 h	6 h	9 h	12 h	15 h	18 h	21 h	24 h	27 h		30 h
Aldehydes	Hexanal	—	0.0467	0.1116	0.0372	0.0653	0.0686	0.0440	0.0298	0.0214	—	0.0686	MS, RI
	Octanal	0.0304	—	0.0509	0.1145	0.0112	0.0172	—	—	—	—	0.0172	MS, RI
	Nonanal	0.3925	0.3076	0.3274	0.1672	0.0770	0.0799	0.6434	—	—	0.1149	—	MS, RI
	2-Octenal, (E)-	0.0262	0.0459	0.0612	0.0185	—	—	—	—	0.0169	0.0122	—	MS, RI
	Benzeneacetaldehyde	—	0.0488	—	—	0.0169	—	0.1882	0.2325	0.0114	—	0.0789	MS, RI
	Decanal	—	0.0808	0.0962	0.0541	0.0325	0.0187	0.1906	0.0459	0.0388	0.0305	—	MS, RI
	Dodecanal	0.0781	—	0.0213	0.0073	0.0037	0.0010	—	0.0043	0.0035	0.0030	0.0010	MS, RI
	2-Nonenal, (E)-	0.0921	—	—	—	—	—	0.0932	0.0156	—	—	0.0064	MS, RI
	2-Octenal, 2-butyl-	0.1305	—	0.6506	0.0205	0.0402	0.0364	0.1827	0.0472	0.0471	0.0401	0.0364	MS, RI
Alcohols	1-Pentanol	0.0488	0.0650	0.0710	0.0249	0.0260	0.0218	—	0.0318	0.0266	0.0239	0.0218	MS, RI
	2-Hexadecanol	0.0209	0.0234	0.0098	0.0019	0.0080	0.0035	0.0238	0.0054	0.0087	0.0023	0.0038	MS, RI
	1-Hexanol	—	0.1509	0.1738	0.0607	0.0647	0.0589	0.1134	—	0.0742	0.0656	—	MS, RI
	1-Octen-3-ol	0.2008	0.2053	0.2219	0.0879	0.0727	0.0659	0.2413	0.0960	0.0923	0.0792	0.0659	MS, RI
	1-Hexanol, 2-ethyl-	—	0.0857	0.0835	0.357	0.0446	0.0044	0.0473	0.0385	0.0342	—	—	MS, RI
	2-Octen-1-ol, (E)-	—	0.0322	0.0393	0.0056	0.0123	—	0.0650	0.0186	0.0173	0.0139	—	MS, RI
	Levomenthol	—	0.0455	0.0653	0.0245	0.0142	—	—	—	—	—	—	MS, RI
	1-Propanol	—	—	0.0140	—	0.0062	0.0029	—	—	—	0.0046	0.0029	MS, RI
	Cyclohexanol, 5-methyl-2-(1-methylethyl)-	—	—	—	—	—	0.0129	—	—	—	0.0042	0.0129	MS, RI
	1-Hexanol, 2-ethyl-	—	—	—	—	—	—	—	—	—	0.0298	0.0444	MS, RI

Table II. (Continuation).

volatiles compounds		relative odor concentration ^a											identification
		0 h	3 h	6 h	9 h	12 h	15 h	18 h	21 h	24 h	27 h	30 h	
Ketones	5-Hepten-2-one, 6-methyl-	0.0192	0.0372	0.0387	0.0249	0.0121	0.0157	—	0.0103	0.0218	0.0248	0.157	MS, RI
	3,5-Octadien-2-one, (E,E)-	—	0.1486	0.1878	0.0055	0.0443	0.0319	0.4433	0.0507	0.0485	0.0420	0.0319	MS, RI
	γ-Chlorobutyrophenone	—	0.0523	0.0503	0.4323	0.0153	0.0152	—	—	—	—	0.0152	MS, RI
	5,9-Undecadien-2-one, 6,10-dimethyl-, (E)-	—	0.0151	0.0207	0.0176	0.0157	0.0138	0.0321	0.0109	0.0142	0.0126	0.0138	MS, RI
	Acetophenone	—	—	—	—	—	—	0.2258	—	0.0218	0.0164	—	MS, RI
Esters	Pentanoic acid, 1-methylethyl ester	0.1004	0.0483	0.0886	0.0531	0.0671	0.0740	0.0533	0.0641	0.0355	0.0617	0.0740	MS, RI
	Butanoic acid, butyl ester	—	—	0.0213	—	0.0106	—	—	—	0.0142	0.0106	—	MS, RI
	2(3H)-Furanone, dihydro-5-pentyl-	—	—	—	0.0028	0.0055	0.0052	—	0.0088	0.0075	0.0071	—	MS, RI
	Methyl formate	—	—	—	—	—	0.0013	—	—	0.0118	0.0073	—	MS, RI
acids	trans-13-Octadecenoic acid	0.0451	—	—	—	0.0105	—	0.0470	—	—	0.0115	—	MS, RI
	17-Octadecenoic acid	—	—	—	—	—	0.0024	—	—	—	—	0.0024	MS, RI
	Hexanoic acid	—	—	—	—	—	—	—	—	—	0.0137	0.0093	MS, RI
	Acetic acid	—	—	—	—	—	—	—	—	—	—	0.0013	MS, RI
	6-Nonynoic acid	—	—	—	—	—	—	—	—	—	—	0.0032	MS, RI
	cis-11-Eicosenoic acid	—	—	—	—	—	—	—	—	—	—	0.0011	MS, RI
Phenolics	Butylated Hydroxytoluene	0.4626	0.0533	0.0020	0.0040	0.0091	0.1648	0.0537	0.0321	0.0242	—	0.1648	MS, RI
	Phenol	—	—	—	—	0.0025	0.0031	—	0.0036	0.0029	—	0.0031	MS, RI
Ethers	Ethanol, 2-(2-ethoxyethoxy)-	—	—	—	—	0.0049	—	0.0566	0.0640	0.0053	—	0.0589	MS, RI
Others	Tetradecane	0.0805	0.0621	0.0730	0.0232	0.0146	0.0088	0.1514	0.0241	0.0232	0.0177	0.0088	MS, RI
	Nonadecane	0.1246	—	—	—	0.0096	—	0.0690	—	—	0.0080	0.0176	MS, RI
	Naphthalene	—	—	0.0056	—	0.0096	—	0.1370	—	—	0.0010	—	MS, RI
	Dodecane	—	—	0.0145	0.0217	—	0.0145	—	0.0229	0.0163	—	—	MS, RI
	Benzene, 1,4-dichloro-	0.0455	—	0.1349	0.0558	0.0025	0.0260	0.5109	0.0457	0.0389	0.0354	0.0260	MS, RI
	Benzene, 2,4-dichloro-1-methyl-	0.0234	0.0116	0.0095	0.0092	0.0029	—	0.0409	0.0056	0.0060	—	—	MS, RI
	Benzothiazole	—	—	0.0079	0.1069	0.0031	0.0051	—	0.0050	—	0.0035	0.0051	MS, RI

a: Relative odour concentrations of compounds that were detected by the internal standard substance 2-methyl-3-heptanone. --: not found.

CONCLUSIONS

The changes in aldehydes, alcohols, ketones, esters, acids, phenolics, ethers, and other aromatic compounds in four different noodles

were evaluated and analysed using the SPME-GC-MS technique. The number of total volatile compounds in cooked noodles were higher than that in raw noodles. The total volatile compounds in the potato/wheat flour noodle samples

contained mainly aldehyde compounds and were greater than those in the wheat noodles. The total volatile compounds in the cooked noodle samples were greater than those in raw noodle samples. The volatile compounds from the potato/wheat noodles had a greater number of alcohol compounds but a lower number of ketones in cooked noodle samples than in raw noodle samples. The aldehydes might be valuable for enhancing the aroma of potato/wheat noodles. During storage time, these compounds were expected to strongly affect the aroma and flavour of potato/wheat fresh noodles. The number of aldehydes changed slightly, and the number of acids increased dramatically. These results provided a better understanding of the differences in volatile compounds between the potato/wheat flour fresh noodle samples and the wheat noodle samples. This study will contribute to further studies related to the volatile compounds of noodles.

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