Storage Stability of Deep-Fried Shallot Flavoring

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Shallot slices were heated with soybean oil at 150−160 °C for 3 min and stored at 60 °C. The volatiles of the deep-fried shallots and the lipid degradation during storage were studied. The amount of degraded components, which were formed from deep-fried oil, increased during storage, while the amount of some characteristic compounds of deep-fried shallot flavors decreased. However, both the peroxide and thiobarbituric acid values of the test (deep-fried shallot with oil), the control (fried oil only), and the blank (oil without frying) increased over storage time. The test showed a better storage stability than the control and the blank.

Keywords: *Shallot flavor; deep-fried; volatile components; storage stability*

INTRODUCTION

Shallot (*Allium cepa* L. var. *aggregatum*) is one of the important seasoning spices in Chinese foods. Normally, shallots were crushed and cut into pieces before cooking, and then the slices were put into the deep-frying oil at high temperatures until the shallot flavor was produced. After being deep-fried, the shallots were then taken out of the oil for the application to Chinese cuisine such as instant noodles, fried noodles, fried rice, and rice with ground pork. Since the deep-dried foods are susceptible to oxidation during storage, Wu and Wu (1982) examined the development of the oxidative off-flavor during storage using sensory evaluation. They suggested that the off-flavor was attributed to those lipid-degraded compounds. Furthermore, the volatile components from raw, baked, and deep-fried shallots had been identified by Wu *et al*. (1982) and had been reviewed by Fenwick and Hanley (1985), Ho *et al*. (1989), and Maarse and Visscher (1989). In the volatiles of deep-fried shallots, sulfides, disulfides, trisulfides, and thiophenes were found to be the major components.

In addition, Chan *et al*. (1991) used the methods of short-path distillation followed by acidic, alkaline, and neutral fractionation to separate the volatiles of deep-fried shallots. Volatiles from the acidic, alkaline, and neutral fractions were identified with different compounds, including sulfides, pyrroles, and the compounds from the degradation of lipid. The lipid-degraded compounds were identified as (*E*)-2-hexenal, (*E*)-2-heptenal, (*E*)-2-decenal, (*E*,*E*)-2,4-heptadienal, (*E*,*E*)-2,4-decadienal, pentanoic acid, and hexanoic acid.

Recently, Chinese-style foods flavored with deep-fried shallots have become more and more popular. However, the stability of deep-fried shallot flavor is of great importance due to the fact that the flavor may be susceptible to oxidation during storage. Therefore, our objective was to investigate the volatile components of deep-fried shallots at different numbers of storage days and evaluate the stability of deep-fried shallots by comparing them with their lipid-degraded components formed during storage. The lipid degradation of deep-fried shallots was also studied in terms of peroxide and thiobarbituric acid values.

MATERIALS AND METHODS

Materials. Shallots were purchased at a local market in Taichung, Taiwan. Soybean oil was a commercial product purchased from a local market and was used as the frying oil.

Sample Preparation. Peeled shallots were cut into slices approximately 2 mm thick using a slicer. Shallot slices (1.5 kg) were put into a pot containing 4.5 kg of frying oil and then deep-fried at 150−160 °C. After the shallots were deep-fried for 3 min, the color of the shallots changed to light brown. The mixture of shallot slices and oil was immediately cooled to room temperature using an ice/water bath and stored in an oven at 60 °C. On days 0, 1, 4, 8, 12, and 15, the mixture of deep-fried shallots and oil was sampled as the test for the flavor isolation and the evaluation of lipid degradation, respectively. In the control, oil was heated under the same condition above without the addition of shallots, and stored in the same oven at 60 °C. Unheated soybean oil was used as the blank and was also stored in the same oven at 60 °C. Both the control and the blank were evaluated only for the lipid degradation. All samples were stored in screw-capped brown glass bottles.

Flavor Isolation. The mixture of fried shallots and oil (400 g), with the ratio of shallots to oil of 1:3, was put into a modified Likens-Nickerson apparatus. After 2 mL of the internal standard [propyl propionate (Aldrich, Milwaukee, WI), 53 mg in 50 mL of 1:1 diethyl ether/*n*-pentane, v/v] was added, the mixture was immediately subjected to the flavor isolation. A mixture of 25 mL of diethyl ether (Merck, Darmstadt, Germany, glass-distilled) and 25 mL of *n*-pentane (Merck, glass-distilled) was used as an extraction solvent. The simultaneous steam distillation−solvent extraction (SDE) was allowed to proceed for 2 h, and the extract thus obtained was dried over anhydrous sodium sulfate (Merck) and filtered. The filtrate was preconcentrated at 40 °C in a distillation apparatus packed with glass beads and then carefully reconcentrated to approximately 50 μL using a 10 cm × 0.2 mm inside diameter Vigreux column at 40 °C.

Gas Chromatography−Mass Spectrometry (GC−MS). The concentrated isolate was analyzed using a Hewlett-Packard 5890A Series II gas chromatograph coupled to a Hewlett-Packard 5971A MSD mass spectrometer. A 50 m × 0.32 mm fused silica WCOT capillary column coated with CP-Wax 52 CB (0.25 μm film thickness, Chrompack, Middelburg, The Netherlands) was used for the separation of components and was interfaced directly into the ion source of the mass

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spectrometer. The operating conditions were as follows: injector temperature, 250 °C; GC−MS interface temperature, 265 °C; and helium carrier flow rate, 1.0 mL/min. The oven temperature was held at 50 °C for 5 min, then programmed from 50 to 210 °C at 1.5 °C/min, and held at 210 °C for 10 min. A split ratio of 60:1 was used. Mass spectra were obtained with an electron multiplier voltage and an electron ionization energy of 1500 V and 70 eV, respectively. Retention indices of the volatile components were calculated with *n*-paraffin (C_5−C_{25}) as references (Schomberg and Dielmann, 1973). The amount of each component was determined using an internal standard method (propyl propionate as the internal standard) and calculated by each peak area of the ion spectrum.

Determination of the Peroxide Value (POV) and the Thiobarbituric Acid (TBA) Value. The POV of each sample at different storage days was carried out according to a method of AOAC (1990). The mixture of fried shallots and oil (5 g) was dissolved in 30 mL of a glacial acetic acid/chloroform (3:2, v/v) solution. After 0.5 mL of freshly prepared saturated KI solution was added, the mixture was shaken for 1 min and then 30 mL of distilled water was added. When the mixture was titrated with 0.01 N $Na_2S_2O_3$ until the yellowish color almost disappeared, 0.5 mL of a 1% starch solution was added. The titration was continued and finished as the blue color just disappeared. The TBA value was determined according to the method of Sun *et al.* (1976) and Shahidi (1994) with a slight modification described below. After 3 g of sample was weighed into a flask, 10 mL of CCl_4 was added to dissolve the sample. To this sample solution was then added 25 mL of the TBA solution, and the upper layer of the mixture was separated after thorough mixing. The TBA value was determined by measuring the absorbance at 535 nm.

RESULTS AND DISCUSSION

According to the findings of Wu and Wu (1982), the deep-fried shallot flavor is judged to be unacceptable by sensory evaluation after 2 months of storage in an accelerated experiment at 45 °C. They suggested that the off-flavor of deep-fried shallots might have resulted from lipid degradation. Therefore, this study was conducted to further examine those volatile components formed during storage.

In this study, shallot slices were heated with soybean oil at 150−160 °C for 3 min, then cooled, and stored at 60 °C. On days 0, 1, 4, 8, 12, and 15, the mixture of deep-fried shallots and oil was sampled for the flavor isolation and the evaluation of lipid degradation, respectively. The volatiles were isolated using SDE, then concentrated, and analyzed by GC−MS. Figure 1 shows the total ion chromatogram of volatile components of freshly prepared deep-fried shallots. The results of identification and quantification of flavor volatiles are shown in Table 1. A total of 35 compounds were identified by comparing their retention indices and mass spectra data with previously published literature data (Wu and Wu, 1981, 1982; Chen and Wu, 1982; Wu *et al.*, 1982; Chou *et al.*, 1983; Chan *et al.*, 1991; Block *et al.*, 1992) and those from the Wiley computer library, in which 21 compounds were further confirmed by authentic compounds. Among these components, benzeneacetaldehyde, 2,3,5-trimethylpyrazine, 2-methylpyrazine, 2-ethylpyrazine, and 2-ethyl-3,6-dimethylpyrazine were first identified. Compared to the previous results (Chan *et al.*, 1991), this study identified more pyrazine compounds but less sulfur-containing compounds.

During storage at 60 °C, changes of the volatile contents were observed in those formed from lipid degradation, such as hexanal, 2-pentylfuran, (*E*,*E*)- and (*E*,*Z*)-2,4-decadienal, and hexanoic acid (Table 1). Contents of these components increased over storage time, and therefore, they might be responsible for the off-flavor of deep-fried shallots. However, contents of some sulfides and pyrazines, which were the characteristic flavor of deep-fried shallots, decreased during storage.

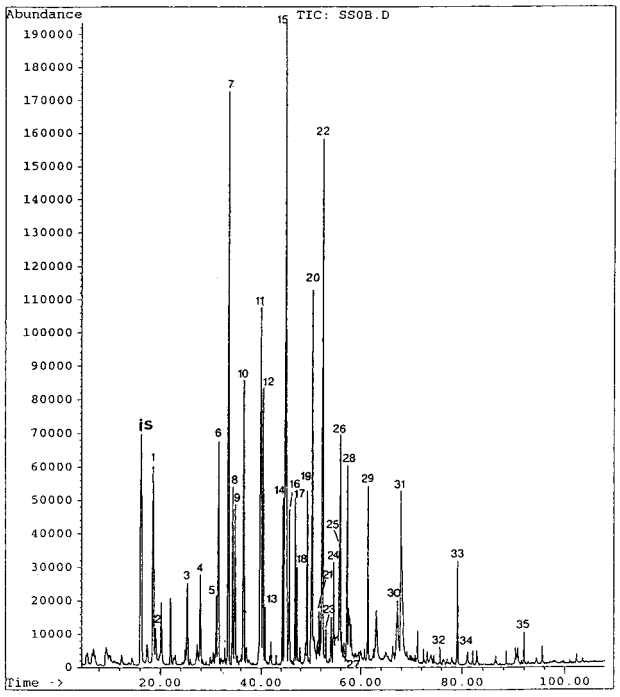

Figure 1. Total ion chromatogram of volatile components of freshly prepared deep-fried shallots.

Three classes of compounds, including sulfur-, nitrogen-, and oxygen-containing compounds, and their percentage compositions during storage are compiled in Table 2. In oxygen-containing components, the major carbonyl compounds in the volatiles of deep-fried shallots were more than those present in the raw shallot essential oil (Chen and Wu, 1983; Wu *et al.*, 1982). It is understood that these carbonyl compounds were generated from the lipid degradation of soybean oil during storage and have been found in the volatiles of deep-fried soybean oil (Wu and Chen, 1992).

Another important group of compounds in oxygen-containing components was the products from the pyrolysis of carbohydrates, such as furfural, 5-methylfurfural, and furfuryl alcohol. These components are mainly found in smoked foods (Hollenbeck, 1994), and therefore, they might contribute the smoking flavor to deep-fried shallots.

The major sulfur-containing compounds of deep-fried shallots contained propenyl group in their structures (Table 1), which was apparently different from the allyl group of garlic flavor components (Jirovetz *et al.*, 1992; Mazza *et al.*, 1992) but is in agreement with the results of investigations of onions (Boelens *et al.*, 1971). During the storage of deep-fried shallots at 60 °C, the amounts of most sulfur-containing compounds decreased. In contrast, the amount of lipid-oxidized products increased after storage. It might be one of the reasons why the deep-fried shallot flavor became less acceptable after storage. However, sulfur-containing heterocyclic compounds with antioxidative activity formed in Maillard reaction model systems have been reported previously (Eiserich and Shibamoto, 1994). Their findings suggested that volatile sulfur-containing heterocyclic compounds, including thiophenes, thiazoles, and saturated cyclic sulfides, might be in part responsible for the

Table 1. Differences of Volatile Components of Fried Shallot during Storage at 60 °C

peak no.[a]	compound	RI[b]	MW[c]	amounts (mg/kg) at storage day 0	1	4	8	12	15
1	dimethyl disulfide[d]	1083	94	3.45	2.17	1.53	0.60	0.55	0.26
2	hexanal[d]	1090	100	0.54	0.72	0.91	1.32	1.35	1.46
3	2-methyl-2-pentenal	1167	98	1.25	0.79	0.82	0.56	0.46	0.15
4	2,5-dimethylthiophene	1242	112	1.28	1.12	0.94	1.15	1.25	1.48
5	2-pentylfuran[d]	1243	138	0.86	0.25	0.51	0.81	1.14	1.39
6	methyl propyl disulfide[d]	1252	122	3.83	4.09	3.52	2.00	1.51	1.48
7	2,4-dimethylthiophene	1264	112	7.92	7.67	6.46	4.63	5.39	3.94
8	(*Z*)-propenyl methyl disulfide	1273	120	2.17	2.02	1.48	0.93	0.92	0.84
9	2-methylpyrazine[d]	1275	94	2.20	1.70	1.03	1.03	0.64	0.40
10	(*E*)-propenyl methyl disulfide	1297	120	4.12	4.21	4.03	2.64	2.82	2.05
11	2,5-dimethylpyrazine	1335	108	6.30	8.99	7.84	4.46	2.45	2.68
12	2,6-dimethylpyrazine	1341	108	3.56	4.26	2.96	2.08	1.72	1.57
13	2-ethylpyrazine	1344	106	0.51	0.56	0.31	0.22	0.27	0.45
14	dipropyl disulfide[d]	1390	150	2.03	3.21	2.17	2.42	2.86	1.80
15	dimethyl trisulfide[d]	1392	126	9.84	11.01	9.65	7.84	7.79	4.42
16	2-ethyl-6-methylpyrazine	1399	122	1.84	1.98	1.44	1.69	1.52	1.66
17	2,3,5-trimethylpyrazine[d]	1414	122	1.94	2.13	1.44	1.20	1.16	1.52
18	(*Z*)-propenyl propyl disulfide	1421	148	0.98	0.96	0.73	0.85	0.90	0.81
19	(*E*)-propenyl propyl disulfide	1447	148	2.25	2.82	2.67	1.99	1.51	2.02
20	2-ethyl-3,6-dimethylpyrazine[d]	1459	136	5.60	6.10	4.97	4.06	3.62	4.06
21	2-ethyl-3,5-dimethylpyrazine[d]	1465	136	0.57	0.69	0.64	0.91	1.14	1.36
22	furfural[d]	1471	96	7.58	8.60	6.02	3.32	2.28	2.02
23	2-methyl-3-propylpyrazine	1489	136	0.36	0.42	0.29	0.15	0.30	0.61
24	(*E,E*)-2,4-heptadienal[d]	1508	110	1.20	1.45	1.88	0.68	0.68	0.96
25	2-acetyl furan[d]	1524	110	1.17	1.35	1.01	1.03	0.95	1.20
26	2,5-dimethyl-3-propylpyrazine	1525	150	2.38	2.81	2.35	1.56	1.75	2.83
27	benzaldehyde[d]	1531	106	0.13	0.17	0.35	0.13	0.38	0.45
28	methyl propyl trisulfide[d]	1539	154	2.50	2.63	3.58	2.81	2.64	2.71
29	5-methyl furfural[d]	1577	110	1.89	2.02	1.35	1.08	1.10	1.35
30	benzeneacetaldehyde[d]	1605	120	1.63	1.76	1.31	1.01	0.83	0.89
31	furfuryl alcohol[d]	1666	98	2.56	2.11	2.47	1.14	1.15	1.40
32	(*E,Z*)-2,4-decadienal[d]	1767	152	0.15	0.32	0.25	0.30	0.23	0.58
33	(*E,E*)-2,4-decadienal[d]	1815	152	1.09	2.11	1.54	1.56	1.64	2.61
34	hexanoic acid[d]	1848	116	0.20	0.29	0.21	0.40	0.67	0.65
35	2-hexyl-5-methyl-(2*H*)-furan-3-one	1999	182	0.23	0.94	0.66	0.44	0.42	0.76
total				86.11	94.43	79.32	59.00	55.99	53.82

[a] The peak numbers correspond to Figure 1. [b] RI, retention indices, using paraffin (C_5−C_{25}) as references. [c] MW, molecular weight. [d] Mass spectrum and retention index are consistent with those of authentic compounds.

Table 2. Percent Composition of Chemical Classes of Deep-Fried Shallot Volatiles during Storage at 60 °C

class	% composition at storage day 0	1	4	8	12	15
sulfur-containing compounds	46.88	44.38	46.28	47.22	50.26	39.78
nitrogen-containing compounds	29.34	31.39	29.37	29.42	26.02	31.27
oxygen-containing compounds	23.78	24.23	24.35	23.36	27.72	28.95

antioxidative phenomena observed with Maillard reaction products. In comparison with to their findings, the identified alkyl-substituted thiophenes present in deep-fried shallots might have the antioxidative activities.

Alkyl pyrazines have been recognized as the important flavor contributed to a large number of cooked, roasted, toasted, and deep-fat-fried foods (Maga, 1982). According to the findings of Shibamoto (1980), alkyl pyrazines in deep-fried shallots were thought to be formed from a reaction of the degraded nitrogenous substances with amine compounds from proteins, peptides, amino acids, and the α-dicarbonyl compounds in shallots. 2-Ethyl-3,6-dimethylpyrazine, a newly identified compound in the volatiles of deep-fried shallots, has a flavor threshold of 0.4 part in 10^9 parts water (Guadagni *et al*., 1972). Nevertheless, due to its unique flavor characteristics, this compound may contribute significantly to the flavor of deep-fried shallots.

As shown in Table 1, hexanal and (*E,E*)- and (*E,Z*)-decadienal were the major lipid-degraded components formed in the volatiles of deep-fried shallots as compared to raw shallot essential oil (Chen and Wu, 1983; Wu *et al*., 1982). Hexanal and 2,4-decadienals have been reported as the primary oxidation products of linoleic acid (Ho *et al*., 1989). In view of the extremely low odor threshold of 2,4-decadienals (0.07 μg/kg) as compared to that of hexanal (4.5 μg/kg) (van Germert and Nettenbrijer, 1977), the 2,4-decadienals should be the more significant oily odorant for deep-fried shallots. In fact, 2,4-decadienal is known to be one of the most important flavor characteristics of deep-fat-fried foods (Ho *et al*., 1987). In addition, some of the oxidation products, including hexanal, hexanoic acid, and 2-pentylfuran, might be responsible for the off-flavor of deep-fried shallots as well. Hexanoic acid is thought to be an oxidation product of hexanal (Shahidi, 1994). 2-Pentylfuran is a well-known autoxidation product of linoleic acid and is also one of the compounds responsible for the reversion flavor of soybean oil (Ho *et al*., 1978). Compared to the contents of lipid-degraded compounds, the major components (>1.0 mg/kg) in the volatiles of deep-fried shallots included (*E,E*)-2,4-decadienal, (*E,E*)-heptadienal, and 2-methyl-3-pentenal. These results were different from those of Chan *et al*. (1991). In their report, the first three major compounds are found to be 1-hepten-3-ol, (*E,E*)-2,4-decadienal, and (*E*)-2-heptenal.

During storage at 60 °C, the widely used methods of POV and TBA were used to evaluate the oxidative state

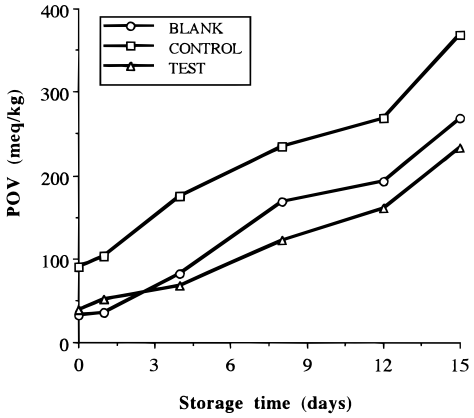

Figure 2. Peroxide values of deep-fried shallots during storage at 60 °C: blank, oil without frying; control, fried oil without shallots added; and test, fried shallots with oil.

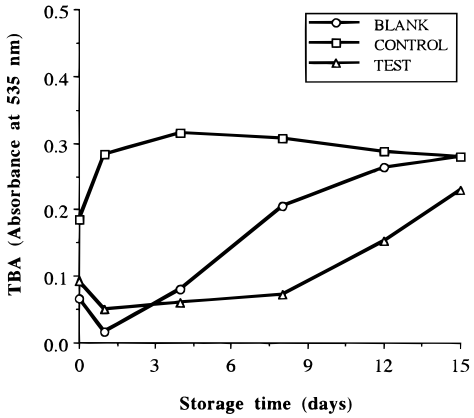

Figure 3. Thiobarbituric acid indices of deep-fried shallots during storage at 60 °C: blank, oil without frying; control, fried oil without shallots added; and test, fried shallots with oil.

of deep-fried shallots. The POV method has been generally recognized as a common method for monitoring oxidative changes in foods during storage. However, the fact that the breakdown of hydroperoxides to secondary oxidation products may result in a decrease in the POV during the storage period must be a concern (Shahidi, 1994). In this study, the blank, control, and test experiments showed a similar increasing tendency of the POV during storage (Figure 2). In deep-fried shallots with oil (the test), the POV increased from 39 at day 0 to 256 (milliequivalents/kg) at day 15, while in the control (fried oil only) and the blank (oil without frying), the POV increased from 91 and 32 at day 0 to 368 and 268 (milliequivalent/kg) at day 15. Apparently, the test had a lower POV than the control throughout the storage period, and from day 4 on, it had a lower POV than the blank. These interesting results strongly suggested that the antioxidant components might be present in shallots.

After frying, the control had a TBA value of up to 0.18 (Figure 3). During storage at 60 °C, the TBA value of the control reached a peak of 0.32 at day 4 and then declined steadily to 0.28 at day 15. It was thought that the TBA value might reach a maximum over the storage period. One of the limitations of the TBA test was the fact that it cannot be used in the detection of products containing a high concentration of peroxides (Shahidi, 1994). In the test, the TBA value increased from 0.09 at day 0 to 0.23 at day 15, while in the control, the TBA value increased from 0.07 at day 0 to 0.28 at day 15. In TBA values, the test showed lower values than those of the control and the blank. As shown in Figures 2 and 3, deep-fried shallots with oil (the test) showed better storage stability than the fried soybean oil and the oil without frying. However, the results from POV and TBA values during storage at 60 °C correlated well with the quantitative analysis of deep-fried shallot volatiles during storage.

It was concluded that the off-flavor of deep-fried shallots formed during storage resulted from the lipid oxidation but was not from the shallot itself. This could be due to the effect of antioxidation derived from the shallot itself. Nevertheless, the current study on the volatiles of deep-fried shallots could not indicate which compound has the antioxidant activity. Therefore, further research is needed to separate the compounds which are responsible for the antioxidation in shallots.

ABBREVIATIONS USED

GC−MS, gas chromatography−mass spectrometry; POV, peroxide value; TBA, thiobarbituric acid.

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