Effect of Freezing, Freeze-Drying, and Air-Drying on Odor of Chive Characterized by Headspace Gas Chromatography and Sensory Analyses

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Aroma compounds of fresh and processed chives were analyzed using headspace gas chromatography–mass spectrometry to determine the effect of freezing, freeze-drying, and air-drying on chive. The main aroma component of fresh chive was dipropyl disulfide. The relative proportions of disulfides were smaller in the frozen, freeze-dried, and air-dried chives than in the fresh. The most abundant carbonyl compounds were 2-hexenal in frozen chive and 2-methylpentenal in freeze-dried and air-dried chives. Differences between chive samples were also studied by sensory analyses to determine whether processing affects the odor characteristics of chives. Freezing and drying treatments alter the perceived intensities of the odor attributes, and significant differences between chive samples were found.

INTRODUCTION

The major aroma precursors of onion (*Allium cepa* L.) are known to be (+)-*S*-(1-propenyl)-L-cysteine sulfoxide, (+)-*S*-methyl-L-cysteine sulfoxide, and (+)-*S*-propyl-L-cysteine sulfoxide. (+)-*S*-(2-Propenyl)-L-cysteine sulfoxide is the main precursor in garlic and is also present in small amounts in other *Allium* species (Granroth, 1968; Collin and Musker, 1988). The pyridoxal phosphate requiring alliinase enzyme (alliin alkyl-sulfenate-lyase, EC 4.4.1.4) catalyzes the formation of primary reaction products, thiosulfinates, from sulfoxide amino acids. Alliinase is specific to S-substituted L-cysteine sulfoxides, and the naturally occurring substrate of onion alliinase is predominantly *trans*-(+)-*S*-(1-propenyl)-L-cysteine sulfoxide. The production of flavor is determined by the activity of alliinase enzyme and the amount of individual flavor precursors.

Primary products are unstable and decompose to secondary products, alcohols and aldehydes, disulfides containing methyl, propyl, and propenyl groups, trisulfides, thiophene derivatives, and other sulfur-containing heterocycles.

Chive (*Allium schoenoprasum* L.) has, with minor exceptions, the same precursors and flavors as onion, although usually in smaller amounts (Carson, 1987). The aroma compounds of chive were apparently first analyzed by Saghir et al. (1964), who identified six disulfides using headspace gas chromatography. The main aroma component of fresh chive is dipropyl disulfide. Other abundant sulfur-containing compounds contributing to the aroma are dimethyl disulfide, methyl propyl disulfide, methyl 2-propenyl disulfide, 1-propenyl propyl disulfide, dipropyl trisulfide, and methyl propyl trisulfide (Saghir et al., 1964; Wahlroos and Virtanen, 1965; Bernhard, 1969; Hashimoto et al., 1983; Kallio et al., 1990). Kallio et al. (1990) identified 3-ethyl-1,2-dithi-5-ene and the corresponding dithi-4-ene in onion and chive. Kameoka and Hashimoto (1983) detected methyl pentyl disulfide and pentyl hydrosulfide from chive; neither have been reported found in onion flavor.

Processing onions causes changes in the aroma compounds. The activity of alliinase enzyme has been shown to decrease during processing. Freeman and Whenham (1975) compared the activity of alliinase of fresh onion (100%) to that of freeze-dried (45%), laboratory frozen (18%), hot-air-dried (8.8–10%), commercially frozen (6.6%), boiled (5.2%), and pickled (0.01%) onion. The decrease in quantities of some aroma compounds is caused by partial, or complete, inactivation of enzymes in processing, partial nonenzymatic destruction of precursors, and partial enzymatic hydrolysis of precursors.

The decrease in enzyme activity in both frozen onion and chive has been observed by many researchers. On the other hand, Freeman and Whenham (1975) noted stable activity of alliinase after 30 days at −20 °C. Obviously conditions during freezing (time, temperature, etc.) affect the degree of loss in activity. Kallio et al. (1990) noticed that the freezing of chive decreases the amounts of some saturated disulfides, such as dimethyl disulfide, methyl propyl disulfide, dipropyl disulfide, and dipropyl trisulfide, and that −80 °C inactivates enzyme more effectively than −20 °C. The amount of later eluting isomer of 1-propenyl propyl disulfide increases at −20 °C in frozen chive. Frozen chive contains more carbonyl compounds (aldehydes, ketones) than fresh chive (Freeman and Whenham, 1974; Kallio et al., 1990).

Freeze-drying has been shown to be the processing method least destructive to the activity of alliinase, although it decreases during processing (Freeman and Whenham, 1975; Wijaya et al., 1991).

The drying of onion causes great loss of aroma (Mazza, 1980). Different compounds behave differently in drying processes. The proportion of dipropyl disulfide decreases considerably, from 100 to 30%, in the 2-h drying time (Mazza and LeMaguer, 1979); moisture content and drying temperature also affect the proportion of dipropyl disulfide. Mazza et al. (1980) stated that certain disulfides act as precursors of thiophenes during processing at high temperatures. During drying the amount of 3,4-dimethylthiophene increased, whereas the amount of propyl propenyl disulfide decreased.

The objectives of the present study were to determine the effect of freezing, freeze-drying, and air-drying on aroma volatiles of chive, compared to those of fresh chive, and to evaluate the impact of processing on odor characteristics.

MATERIALS AND METHODS

Chive Samples. The chive (*Allium schoenoprasum* L.) for fresh, frozen, freeze-dried, and air-dried samples was harvested from a local garden in southwest Finland in 1990.

Fresh chive (dry matter 11%) was analyzed over 3 days by headspace gas chromatography immediately after harvesting

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(May 28, June 6, and June 19). The chive for freezing and drying processes was both collected and processed in 1 day (May 28).

Fresh chive for the frozen samples was quickly chopped into 2-mm pieces and packed in polyethylene bags. Whole chive leaves were also packed. Both types of sample bags were frozen immediately after packing and kept at −18 °C until analyzed.

The chopped (2 mm) as well as whole chive leaves were both freeze-dried and air-dried. The fresh chive was first frozen at −18 °C for 5 h and freeze-dried (Dura Top bulk tray dryer, FTS Systems Inc.). Drying took 8 h for chopped chive and 16 h for whole leaves. Dry matter of chopped samples and whole chive leaves was after freeze-drying 90 and 88%, respectively.

Air-drying was carried out in a hot-air oven (Orakas 5510, Ky. Marlemi) at 40 ± 5 °C over 2 h 30 min for the chopped chive and 7 h 30 min for whole leaves. After drying, dry matter of chopped chive and whole chive leaves was 95 and 86%, respectively.

Freeze-dried and air-dried samples were packed in polyethylene bags and stored at room temperature until analyzed.

Frozen, freeze-dried, and air-dried samples were analyzed by headspace gas chromatography, after 4, 21, 65, 108, and 220 days, over a 7-month period. Sensory evaluation of all four chopped samples (fresh, frozen, freeze-dried, air-dried) was carried out during 6 consecutive working days at the beginning of June 1990.

Gas Chromatographic (GC) Analysis. *Sample Preparation for GC Analyses.* A 4.00-g sample of fresh or frozen chopped chive or a 1.00-g sample of freeze-dried or air-dried chopped chive with 3 mL of added water was weighed (each in triplicate) in a 25-mL bottle closed with a headspace valve (J&W Scientific, Folsom, CA). Dried chives did not absorb as much water as they lost during drying, and relatively more freeze-dried and air-dried samples were weighed than fresh or frozen, measured as dry matter. The samples were kept at 37 °C for 35 min, which is close to the optimum conditions for enzymatic formation of chive volatiles, and the headspace compounds analyzed with a gas chromatograph.

Headspace GC. The chive samples were analyzed using a static headspace gas sampling previously applied by Kallio et al. (1989, 1990). A DB-1701 fused silica open tubular column (30 m × 0.254 mm i.d., d_f 0.25 μm, J&W Scientific) was connected to the on-column injector kept at room temperature. The Varian Aerograph 3700 gas chromatograph (Walnut Creek, CA) equipped with a flame ionization detector was connected to a Hewlett-Packard 3388A integrator (Palo Alto, CA). The detector temperature was 250 °C and the average carrier gas flow 27 cm/s at 20 °C, the inlet pressure of the He carrier being 90 kPa. Before injection, the He pressure was lowered to 60 kPa. Part of the front of the column was placed in liquid nitrogen. A 5-mL sample of chive headspace was injected into the column with a 10-mL gastight syringe (Hamilton, Reno, NV) at a rate of 0.5 mL/min. Pressure was adjusted to 90 kPa before the cold trap was removed. The GC program was 23–230 °C at 2 °C/min.

Gas Chromatography–Mass Spectrometry (GC–MS) Analyses. The compounds of the different chive samples (fresh, frozen, freeze-dried, air-dried) were identified by GC–MS. The electron impact mass spectral (EI-MS) analyses (ionization energy 70 eV) were carried out on a VG 7070E spectrometer employed with a VG-11-250 data system and a Dani 3800 HRch gas chromatograph (VG, Wythenshawe, Manchester, U.K.). Both column and program were the same as used in the GC analyses.

Sensory Analysis. *Judges.* Fourteen judges (10 females, 4 males) between 22 and 58 years of age took part in the sensory evaluation; 13 were nonsmokers and 1 was a smoker. Judges had no previous experience in sensory evaluation.

Evaluations were carried out in separate booths. Each judge was scheduled at a fixed time of day during 6 consecutive working days.

Odor Descriptors. The odor descriptors were selected according to a preliminary laboratory inspection of different chive samples. Fresh, frozen, freeze-dried, and air-dried chive samples were served in closed aluminum-foil-covered glass bottles labeled with three-digit codes in random order.

A panel of judges listed odors perceived using whatever descriptors they regarded appropriate to the samples evaluated. It was decided to keep the number of odor attributes low, making it easier for the judges to handle the test situation and thus avoiding misinterpretation of descriptors by the judges. Four attributes (fresh-green, haylike, onionlike, fishy) were selected after evaluation and round-table discussion due to the predominance and frequency of these attributes used by the judges. The list of odor attributes and the composition of the odor samples are given in Table I.

Table I. Odor Samples of the Attributes Used in Sensory Analyses and Their Composition

attribute	composition
fresh-green	0.3 g of shredded lettuce
onionlike	1.0 g of chopped onion
haylike	0.5 g of chopped dry hay
fishy	1.0 g of sliced fresh Baltic herring

The training of the 14 judges in the use and recognition of the four odor characteristics was carried out in two sessions. Odor samples were served in closed aluminum-foil-covered glass bottles with three-digit codes.

Method. Four odor characteristics of five chive samples ("blind control" included) were compared with those of the reference sample, always the fresh chive, and scaled in terms of degree of deviation from the reference using a nonnumerical 10-cm graphic scale, where 0 equals less than reference, 5 is the same as reference, and 10 equals more than reference.

At every session, an odor sample of each attribute was also available for the judges to ensure a similar definition for every judge.

Sample Preparation for Sensory Evaluation. One-gram fresh or frozen samples were weighed in 20-mL aluminum-foil-covered glass bottles with three-digit codes and sealed; 0.33-g freeze-dried or air-dried samples were weighed, and 1.5 mL of water was added.

All of the samples and references were kept at room temperature for 1 h and served to the judges simultaneously.

RESULTS AND DISCUSSION

Headspace GC Analyses. The volatile compounds of chive headspace (Table II) were identified according to their mass spectra and Kovats indices (I_k). The chromatograms of fresh, frozen, freeze-dried, and air-dried chives analyzed with the DB-1701 column are presented in Figure 1.

The volatile compounds identified were mostly disulfides and some carbonyl compounds. Onion and chive have the same precursors and thus the same aroma compounds, although in different ratios. Over 100 aroma compounds have been identified in fresh and processed onion [reviewed by Whitaker (1976) and Fenwick and Hanley (1985)]. Dipropyl disulfide is the main compound in both onion and chive (Saghir et al., 1964; Wahlroos and Virtanen, 1965; Bernhard, 1968, 1969; Hashimoto et al., 1983; Kallio and Salorinne, 1990; Kallio et al., 1990). In this study, the proportion of dipropyl disulfide (Figure 2) in fresh chive was 70–80% of identified compounds. The other abundant compounds were mainly disulfides, such as methyl propyl disulfide, 1-propenyl propyl disulfides, dimethyl disulfide, 2-propenyl propyl disulfide, and dipropyl trisulfide, whereas the amounts of carbonyl compounds were small. *E* and *Z* isomers of 1-propenyl propyl disulfides could not be discerned on the basis of mass spectral data alone.

Various freezing and drying treatments decrease the total amount of volatile compounds in both onion (Freeman and Whenham, 1974, 1975; Mazza, 1980) and chive (Fenwick and Hanley, 1985; Kallio et al., 1990), as was also observed in this work. The formation of aroma compounds depends on the amount of precursors and the activity of the enzyme alliinase. According to Freeman and Whenham (1975), freezing and freeze-drying do not affect the amount of precursors, whereas great loss is caused by hot-air-drying. They noticed that freeze-drying is the least destructive to the activity of alliinase, but freezing and hot-air-drying result in great losses in activity. The

Table II. Volatile Compounds of Chive Headspace Analyzed with DB-1701 Capillary Column

peak	compound	relative proportion % from identified compounds				
		fresh[a]	frozen[b]	freeze-dried[b]	air-dried[b]	
1	hexane[c]					1
2	1-propanethiol[c]					1, 2, 3, 4
3	methyl thiirane[c]					1
4	1-methylthiopropane[c]					1
5	3-(methylthio)-1-propene (*E*/*Z*)[c]					1
6	pentanal[c]					4
7	dimethyl disulfide	1.4	0.3	0.3	12.8	
8	2-methyl-2-butenal				6.6	
9	hexanal		0.1	tr[e]	6.7	
10	2-methyl-2-pentenal			4.5	37.0	
11	S-propyl thioacetate	1.0				
12	2-hexenal	tr	1.2	0.5	2.2	
13	3-hexen-1-ol	0.2	0.2			
14	methyl 2-propenyl disulfide	0.2	0.1			
15	methyl 1-propenyl disulfide	0.2	tr	0.8	4.1	
16	methyl propyl disulfide	11.2	6.9	5.8	4.4	
17	2-propenyl propyl disulfide	1.3	1.7	1.2	tr	
18	1-propenyl propyl disulfide (*E*/*Z*)			5.6	2.0	
19	dipropyl disulfide	77.6	61.7	32.1	4.2	
20	1-propenyl propyl disulfide (*E*/*Z*)	3.3	24.8	38.3	7.3	
21	3-ethyl-1,2-dithi-5-ene[d]				1.4	
22	nonanal	0.3	0.2	2.0	3.1	
23	3-ethyl-1,2-dithi-4-ene[d]		1.2	2.8	3.1	
24	methyl propyl trisulfide		0.2	1.7	1.9	
25	2-decen-1-ol	0.2				
26	decanal		0.1	1.3	2.2	
27	dipropyl trisulfide	1.3	1.1	4.5	tr	

[a] Average (n = 8). [b] Samples stored for 65 days. [c] No quantitation of compounds 1–6. 1, fresh; 2, frozen; 3, freeze-dried; 4, air-dried. [d] Tentative identification. [e] tr, trace.

main compounds of frozen and freeze-dried chives were dipropyl disulfide, 1-propenyl propyl disulfide, and methyl propyl disulfide. The relative proportions of these compounds were lower in the frozen and freeze-dried samples than in the fresh, since the former consisted of more carbonyl compounds, the most abundant being 2-hexenal in frozen samples and 2-methyl-2-pentenal in freeze-dried samples.

There were no remarkable changes in the proportions of the other abundant sulfur compounds, dimethyl disulfide and dipropyl trisulfide, in fresh and frozen chives. This was in contrast to the results previously published by Kallio et al. (1990). An increase in amounts of 1-propenyl propyl disulfide (later eluting) stored at −20 °C was also noticed in this work, although its proportion decreased after 100 days of storage. Similar reduction in amounts of several sulfur compounds during storage has been noticed in leek oil stored at room temperature (Stephani and Baltes, 1991).

The main compound of air-dried chive was 2-methyl-2-pentenal, the proportion of which, however, decreased during storage. The amount of 2-methyl-2-pentenal, the aldol condensation product of propionaldehyde (Spåre and Virtanen, 1961), has been shown to be higher in the frozen, freeze-dried, and air-dried onion samples than in the fresh (Freeman and Whenham, 1974). Air-dried samples consisted of more dimethyl disulfide than other chive samples. Bernhard (1968) reported that the content of dimethyl disulfide is higher in dried onion than in fresh. Characteristic of air-dried samples were the relatively large amounts of carbonyl compounds, such as hexanal, 2-methyl-2-butenal, nonanal, and 2-hexenal. In good accordance with our results with chive, Mazza et al. (1980) reported that dehydrated onion contains almost all of the volatile compounds of fresh onion, only the concentrations are smaller.

The identifications of 3-ethyl-1,2-dithi-5-ene and 3-ethyl-1,2-dithi-4-ene were tentative and based on the results of Kallio and Salorinne (1990).

Kallio et al. (1990) noticed that freezing decreases the amounts of saturated disulfides in chive. The relative share of dipropyl disulfide, the main compound of fresh and frozen chives in this study, is highest in fresh and lowest in air-dried samples, as can be seen in Figure 2. Freeman and Whenham (1974) observed a significant decrease in the content of dipropyl disulfide in hot-air-dried, freeze-dried, and frozen onion samples. The relatively high evaporation causes great losses in some important aroma compounds when drying (Mazza, 1980). The amount of dipropyl disulfide decreases significantly during drying (Bernhard, 1968; Mazza and LeMaguer, 1979).

The formation of methyl 1-propenyl disulfide (Figure 3) evidently follows some mechanism other than enzyme-catalyzed reaction, while the proportion of this compound was low in fresh and frozen chive samples and highest in air-dried samples. The content of methyl propyl trisulfide remained stable in frozen samples (<0.5%) but increased during storage in freeze-dried and air-dried chives.

The main difference between chopped and unchopped processed chives was in the total amount of aroma compounds. Freeze-dried and air-dried whole leaf samples in particular consisted of low amounts of aroma compounds, probably due to the long freeze-drying and air-drying period.

Sensory Analyses. Results of the evaluation of fresh, frozen, freeze-dried, and air-dried samples are illustrated in Figure 4. Significant differences designated are based on LSD values obtained in analysis of variance (data not shown). The F values from analysis variance of the odor intensity data for each attribute are given in Table III.

In chive the means of four odor attributes ranged from 4.8 to 5.1 (corresponding standard deviations 0.8–1.5) (Figure 4). Comparing the means and corresponding deviations with one another (blind control), as was the case in fresh chive, reflects the reliability of the panel, which was fairly good in this case.

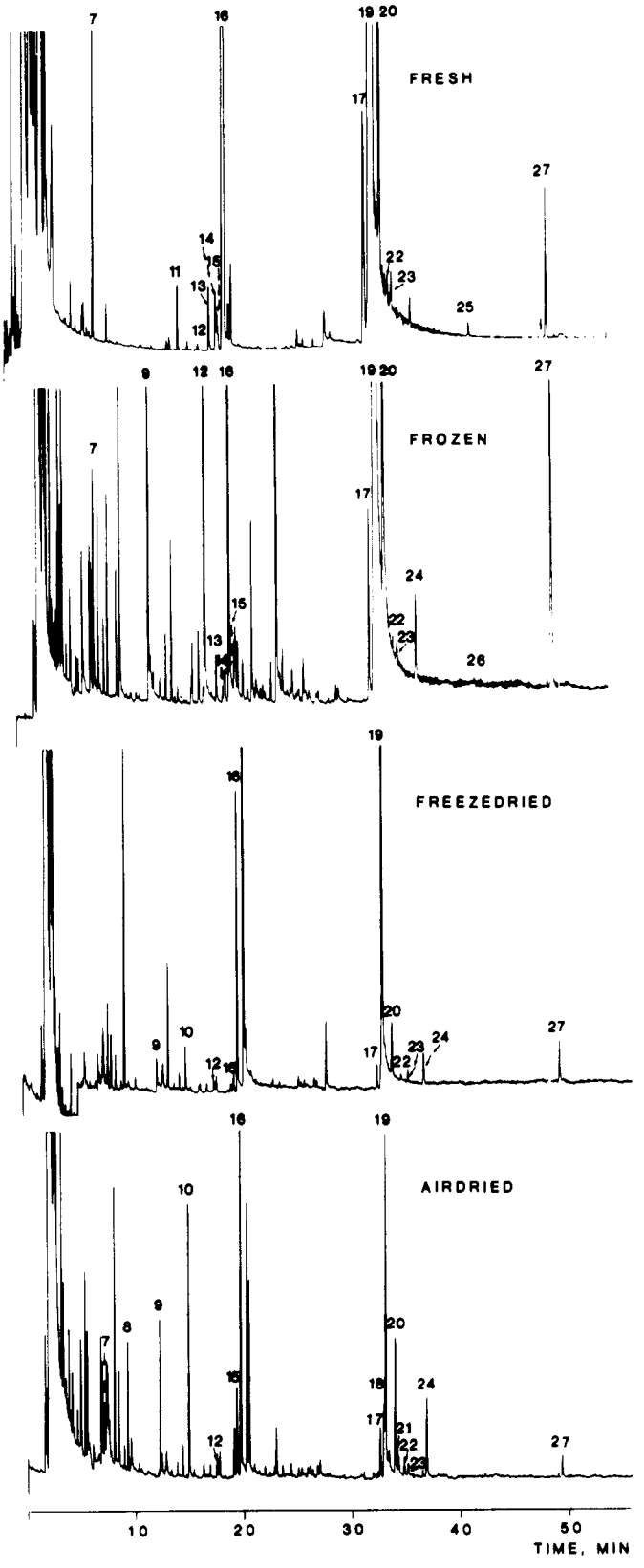

Figure 1. Chromatograms of fresh, frozen, freeze-dried, and air-dried chives analyzed with DB-1701 capillary column.

Freezing and drying treatments alter the perceived intensities of the odor attributes, and significant differences between chive samples were found (Table III). All chive samples differed from each other in intensities of onionlike and haylike odors. No difference between the frozen and freeze-dried samples was observed in fresh-green odor. Strongest intensity of fresh-green odor was detected in frozen samples. Characteristic of dried samples

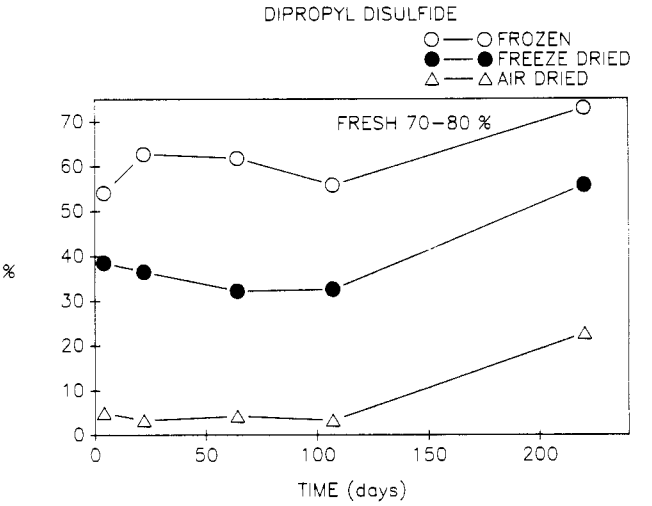

Figure 2. Proportion, in percentage, of dipropyl disulfide in the headspace of fresh, frozen, freeze-dried, and air-dried chive.

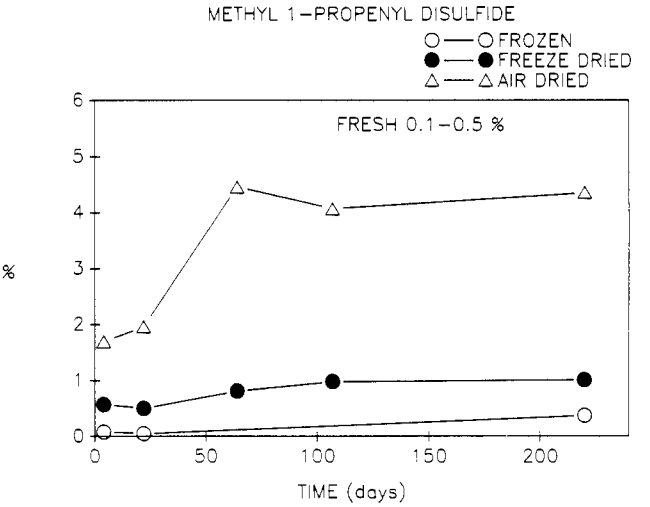

Figure 3. Proportion, in percentage, of methyl 1-propenyl disulfide in the headspace of fresh, frozen, freeze-dried, and air-dried chive.

was low intensity of onionlike odor and high intensity of haylike odor (Figure 4). Frozen chive differed from other samples in fishy odor, which was found most difficult to evaluate by judges (Figure 4).

There are few works published on the sensory properties of onion and chive or on the use of descriptive sensory profiling in characterizing the effect of freezing, freeze-drying, and air-drying of onion and chive on aroma. Chopping and processing of *Allium* species result in differences in the composition of volatile compounds and sensory properties. Dipropyl disulfide, the main compound in fresh and frozen chive in this study, is linked with the odor of fresh onion and dimethyl disulfide with the odor of cooked cabbage (Bernhard, 1969). Dipropyl disulfide attains the olfaction easily, due to its high evaporation, and so affects the aroma of fresh onion and chive. The high evaporation of the compound causes great losses in drying processes (Mazza, 1980), as was also the case in this study (Figure 2). The weakest intensity of onionlike odor was detected in dried samples (Figure 4). On the other hand, according to Galetto and Bednarczyk (1975) methyl propyl disulfide, methyl propyl trisulfide, and dipropyl trisulfide are significant compounds contributing to flavor, whereas dipropyl disulfide has no remarkable effect on the flavor of onion oil. The propenyl derivatives strongly affect onion aroma, the methyl

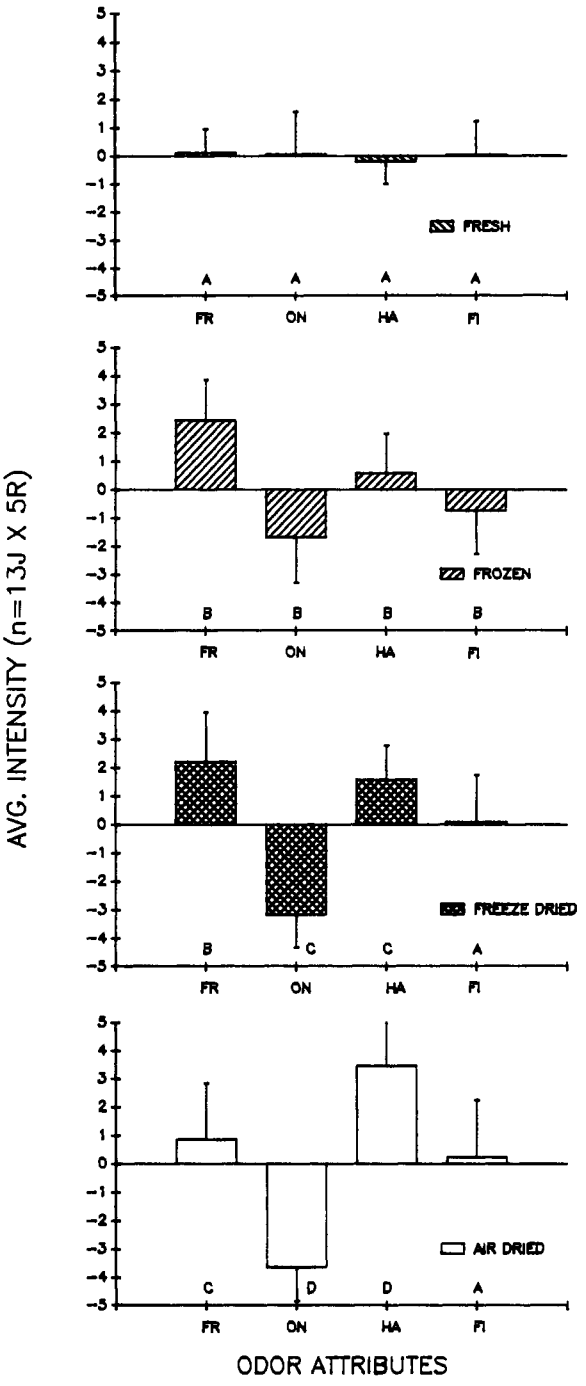

Figure 4. Means and standard deviations for odors of fresh, frozen, freeze-dried, and air-dried chive. Values represent degree of deviation from reference (fresh chive). −5, 0, and +5 on *y*-axis correspond to 0, 5 (=reference), and 10 cm on the scale, respectively. Abbreviations for odor attributes: FR, fresh-green; ON, onionlike; HA, haylike; FI, fishy.

Table III. *F* Values from Analysis of Variance for Attributes

source	df	fresh-green[a]	onionlike[a]	haylike[a]	fishy[a]
judge	12	9.15***	20.83***	6.52***	6.39***
sample	3	59.59***	210.79***	177.71***	6.18***
replication	4	2.68*	1.09	1.34	0.41
judge × sample	36	3.18***	1.64*	4.64***	1.53*
judge × repl[b]	48	1.52*	2.31***		
sample × repl	12	2.25*			

[a] *, **, ***, Significant at $p < 0.05$, 0.01, and 0.001, respectively. [b] Replication.

derivatives add a cabbage tone, and trisulfides give a burnt odor to the aroma (Carson, 1987).

Fresh onion has a rich, pungent odor (Bernhard, 1969). Freeze-dried onion is most reminiscent of fresh onion, as found in sensory tests, although it does not retain the odor and taste of fresh onion (Freeman and Whenham, 1975). In our work, fresh and freeze-dried chive differed from each other in fresh-green, onionlike, and haylike odor attributes.

The intensity of onionlike odor, which probably reflects best, among the attributes evaluated, the amount of sulfur-containing aroma compounds of chive, clearly decreased in frozen, freeze-dried, and air-dried samples, being lowest in dried samples. Air-drying affected most the haylike odor, but the intensity also decreased in frozen and freeze-dried chives (Figure 4).

Consistency of Sensory Results. Judges were a source of variance throughout the study ($p < 0.001$) and replication in the case of fresh-green odor ($p < 0.05$) (Table III). Some significant interactions occurred: judge × replication and sample × replication. Judges had no previous experience in sensory evaluation and used the intensity scale differently, which obviously caused some differences in judgments between replications. A lack of consistency between replications was possibly due to insufficient judge training.

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Registry No. Hexane, 110-54-3; 1-propanethiol, 107-03-9; methyl thiirane, 1072-43-1; 1-methylthiopropane, 3877-15-4; 3-(methylthio)-1-propene, 10152-76-8; pentanal, 110-62-3; dimethyl disulfide, 624-92-0; 2-methyl-2-butenal, 1115-11-3; hexanal, 66-25-1; 2-methyl-2-pentenal, 623-36-9; S-propyl thioacetate, 2307-10-0; 2-hexenal, 505-57-7; 3-hexen-1-ol, 544-12-7; methyl 2-propenyl disulfide, 2179-58-0; methyl 1-propenyl disulfide, 5905-47-5; methyl propyl disulfide, 2179-60-4; 2-propenyl propyl disulfide, 2179-59-1; 1-propenyl propyl disulfide, 5905-46-4; dipropyl disulfide, 629-19-6; 3-ethyl-1,2-dithi-5-ene, 126790-01-0; nonanal, 124-19-6; 3-ethyl-1,2-dithi-4-ene, 126790-02-1; methyl propyl trisulfide, 17619-36-2; 2-decen-1-ol, 22104-80-9; decanal, 112-31-2; dipropyl trisulfide, 6028-61-1.