

Volatiles and Oxidative Flavor Deterioration in Fried Chicken

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Volatiles isolated from fried chicken undergoing oxidative flavor deterioration were gas chromatographed. Chromatograms did not reveal major or consistent qualitative changes during flavor deterioration. The quantity of volatile material increased proportionally to the time and severity of exposure to oxygen. The increase in quantity of *n*-hexanal reflected the extent of oxidative deterioration particularly well.

METHODS for determining the extent of lipid oxidation usually measure only one stage in the course of oxidative lipid deterioration. Therefore, it is not surprising that these methods correlate only inconsistently with subjective estimates of staleness and rancidity (4). Accumulating evidence indicates that volatile products produced by oxidizing lipids cause rancid off-flavor (1, 4). Hence, a method that involves comparison of chromatograms of fried-chicken volatiles before and after rancidification might give more consistent results. The objective of this study was to determine whether some gas-chromatographic feature of fried-chicken volatiles correlates with the onset of staleness and rancidity.

Experimental

Preparation of Fried Chicken. Cut-up, tray-packed frying chickens, held for a few weeks at -10° F., were thawed, dredged, and deep-fat fried. The dredge was 60% hard wheat flour, 30% potato flour, and 10% salt by weight. The chicken parts were cooked in peanut oil at 140° C. to an internal temperature of 185° F. Just after frying, parts were packed one-half carcass per can, and the cans were sealed with an air headspace and stored at -30° F. After the two-week frying period, half of the cans were nitrogen-packed by evacuating and venting each three times through a small hole. After the hole was sealed, the nitrogen-packed cans were placed in -30° F. storage, and the remaining cans were stored at 10° F. Prior to this division, the cans were arranged so that samples for each storage period and for each storage condition included chickens from each batch of frying oil and from each chronological cooking position within each batch of frying oil.

Taste Panel. Eight trained judges compared fried chicken stored in air at 10° F. with fried chicken stored for a similar time in nitrogen at -30° F. A triangle test was used; judges were asked to identify the sample(s) having

more off-flavor and to comment on the type of off-flavor. Evaluations were made in quadruplicate. Significance of results was determined from the tables of Roessler, Warren, and Guymon (7), and by the triangle-intensity method of Davis and Hanson (3).

Isolation of Volatiles. Volatiles were distilled from the chicken in vacuo and at 100° C. and are hereafter referred to, respectively, as vacuum volatiles and steam volatiles. Details of distillation, extraction of the distillate with isopentane, and concentration of the isopentane extract have been described (6). Each batch of meat volatiles was isolated from about 1400 grams of ground and mixed muscle of eight half-carcasses. Similarly, each batch of skin volatiles was isolated from about 450 grams of the ground skin of eight half-carcasses. Volatiles were isolated in duplicate and stored under nitrogen at -30° F. until chromatographed.

Gas Chromatography. All chromatograms were obtained on a gas chromatograph with thermal conductivity detectors and a $1/4$ -inch o.d. \times 8-foot stainless-steel column packed with 15% Tween-20 on 100- to 120-mesh firebrick. Isothermal column temperatures were 65° and 150° C. The volume of concentrated volatiles was adjusted to 90 to 100 μ l. by the addition or evaporation of isopentane, and one third of this volume was injected into the chromatograph. Each duplicate concentrate of volatiles was chromatographed at least once and usually twice. *n*-Hexanal, *n*-nonanal, and 2,4-decadienal were identified as previously described (6).

Slight variation in quantity of volatiles was encountered between chromatograms of duplicate concentrates, more so at 3 months than at 0 or 10 months' storage. However, these quantitative variations were not great enough to interfere with the interpretation of the results.

Gas Analysis. Oxygen in the headspace of cans was determined by the method of Stone and Skavinski (8), with KOH for CO_2 absorption and chromous chloride for oxygen absorption.

Results

Oxidative Flavor Deterioration during Storage. Disappearance of oxygen from the headspace of air-packed chicken (Table I) and the taste panel results (Table II) show that oxidative flavor deterioration did take place in the air-packed chicken. "Staleness" in meat and "rancidity" in skin were used most frequently to describe the off-flavor. As shown particularly well by the T-I values (3), off-flavor development in meat correlates well with storage time (Table II). Surprisingly, the T-I values show significant off-flavor in air-packed skin during the 0- to 1-month period (Table II). However, the degree of off-flavor must have been slight because duplicates were not identified at a significant level during the 0- to 1-month period (Table II). The greater consistency with which off-flavor was detected in meat than in skin confirms the observation of Hanson, Fletcher, and Lineweaver (5) that oxidative off-flavor in fried chicken is as strong in meat as it is in skin and skin coating or perhaps more so. Therefore, we studied meat volatiles more than skin volatiles.

Skin Vacuum Volatiles. The over-all quantity of volatiles was somewhat less in the air-packed skin than it was in the nitrogen-packed skin after 10 months' storage (Figure 1, compare total areas under chromatograms I and II). Furthermore, the peak at about 19 minutes and the 2,4-decadienal peak present in the chromatogram of skin vacuum volatiles (Figure 1, I), are absent from the comparable chromatogram of air-packed skin volatiles (Figure 1, II). Hence, during storage the quantity of skin vacuum volatiles did not increase and accumulate in air-packed skin. Thus, the area under the chromatograms of skin vacuum volatiles proved useless as a measure of oxidative flavor deterioration in the skin.

Skin Steam Volatiles. Steam distillation brought out many more volatiles from skin than did vacuum distillation (compare Figures 1 and 2). The skin

Table I. Oxygen Content of Head-space Gas in Cans of Fried Chicken Packed in Nitrogen and Packed in Air

Storage Time, Months	O ₂ , %	
	Nitrogen pack ^a	Air pack ^b
0	1.3	20.4
0.5	0.0	19.8
2	0.0	16.9
3	0.0	16.5
4	0.0	15.7
8	0.0	12.2
10	0.2	9.4

^a Stored at -30° F.
^b Stored at 10° F.

steam volatiles of air-packed and nitrogen-packed chicken show similar components, but the air pack produced a greater quantity (Figure 2). The greater quantity of steam volatiles is the only chromatographic feature of skin volatiles that reflects the greater oxidative deterioration of skin in the air pack.

Meat Vacuum Volatiles. The quantity of meat vacuum volatiles in the air pack, particularly *n*-hexanal, increased faster during storage than in the nitrogen pack (Figure 3). Furthermore, comparison of the 10-month chromatograms (Figure 3, V vs. VI) reveals that additional volatile components appeared in the air-packed meat. Therefore, oxidation in air-packed meat did cause accumulation of volatile products. This evidence that oxidative deterioration proceeded differently in meat than in skin is compatible with the results of Hanson, Fletcher, and Lineweaver (5), who found a greater flavor change and higher peroxide values in the meat than they did in the skin.

Vacuum volatiles may have disappeared from the nitrogen-packed chicken during the three evacuation and nitrogen flushes used in nitrogen packing. This may explain why more volatile material is evident at 0 months in the air pack than in the nitrogen pack (Figure 3, I vs. II).

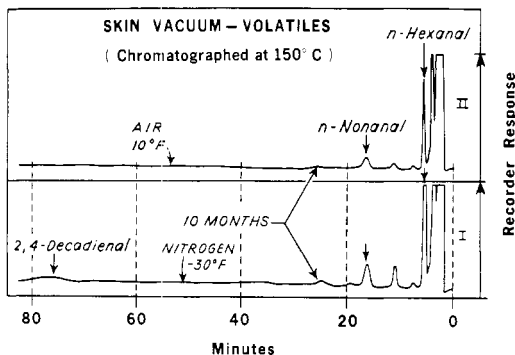


Figure 1. Gas chromatograms of skin vacuum volatiles from air-packed and nitrogen-packed fried chicken after 10 months' storage

Table II. Taste Panel Comparison of Air-Packed Fried Chicken Stored at 10° F. with Nitrogen-Packed Fried Chicken Stored at -30° F.

Storage Time, Months	Meat			Skin		
	Triangle test ^a (duplicates identified), %	More off-flavor judgments in air, 10° F. pack, % ^b	T-I value ^c	Triangle test ^a (duplicates identified), %	More off-flavor judgments in air, 10° F. pack, % ^b	T-I value ^c
0	19	83	177	44	86	152 ^d
0.5	31	50	200	28	89	157 ^d
1.0	31	90	160 ^d	44	79	140 ^e
1.5	31	80	150 ^d	31	90	164
2	38	83	148 ^d	28	44	195
3	62 ^e	80	136 ^e	50 ^d	62	177
4	44	93	128 ^f	44	86	142 ^e
8	62 ^e	90	92 ^f	28	89	151 ^d
10	53 ^d	94	107 ^f	66 ^e	95	87 ^f

^a Total tests per storage period = 32.

^b Based on duplicates correctly identified.

^c T-I values assume chicken stored in air at 10° F. had more off-flavor.

^d Significant at 5% = 161 or less.

^e Significant at 1% = 147 or less.

^f Significant at 0.1% or less.

Chromatography of vacuum volatiles from meat at 150° C. differed only in a distinct peak at about 15 minutes which increased with duration in storage and is probably *n*-nonanal.

Meat Steam Volatiles. The chromatograms at 65° C. of steam volatiles showed clearly that *n*-hexanal formed faster in air packs than in nitrogen packs (Figure 4). The proliferation of peaks as storage goes on is also faster in the air pack than in the nitrogen pack. The unidentified prominent peak at about 10 minutes (Figure 4, VI) is remarkable because it is absent or nearly so from all other chromatograms (Figure 4). The significance of its abrupt appearance in stale meat volatiles to off-flavor development is unknown.

Qualitatively, chromatograms of meat steam volatiles chromatographed at 150° C. proved remarkably similar in spite of storage variables (Figure 5). At 10 months, however, there are distinctly more volatiles from the air pack than from the nitrogen pack. Note the difference in sizes of the 2,4-decadienal peaks at about 73 minutes (Figure 5, IV vs. V).

Discussion

The general qualitative nature of fried-chicken volatiles remained remarkably the same during storage in spite of obvious flavor deterioration. The few qualitative changes that were evident appeared sporadically and were generally much smaller in magnitude than were quantitative changes. Therefore, this study revealed no significant relationship between the qualitative nature of volatiles and flavor deterioration.

On the other hand, the quantity of volatile material in fried-chicken meat was generally proportional to the storage time and, hence, intensity of oxidative off-flavor. Measurement of the increase in the quantity of volatiles, therefore, suggests itself for detecting oxidative flavor deterioration in fried chicken. Determination of the quantity of volatile material in a simple, well defined peak may suffice. *n*-Hexanal in chromatograms of meat volatiles (Figures 3 and 4) varies with the intensity of oxidative off-flavor. Hence, *n*-hexanal promises to serve as an indicator of oxidative deterioration in fried chicken as it does

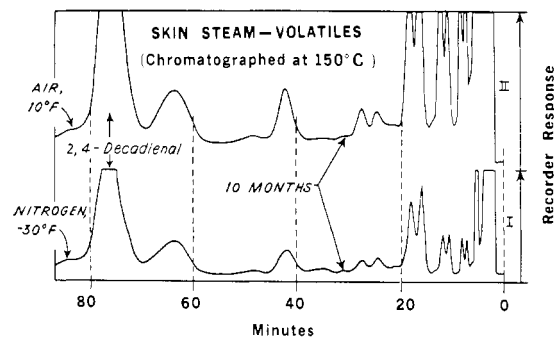


Figure 2. Gas chromatograms of skin steam volatiles from air-packed and nitrogen-packed fried chicken after 10 months' storage

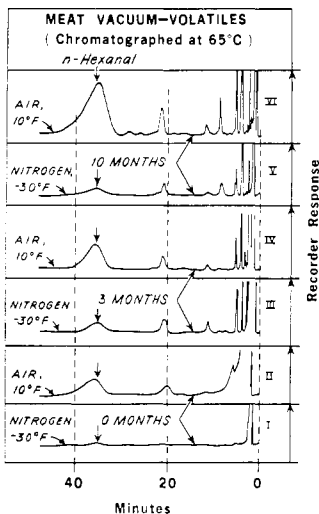


Figure 3. Gas chromatograms of meat vacuum volatiles from air-packed and nitrogen-packed fried chicken at 0, 3, and 10 months' storage

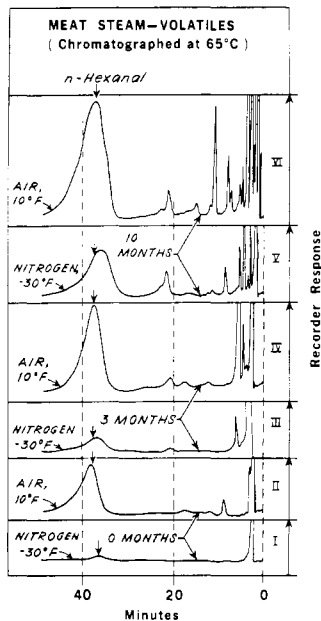


Figure 4. Gas chromatograms of meat steam volatiles from air-packed and nitrogen-packed fried chicken at 0, 3, and 10 months' storage

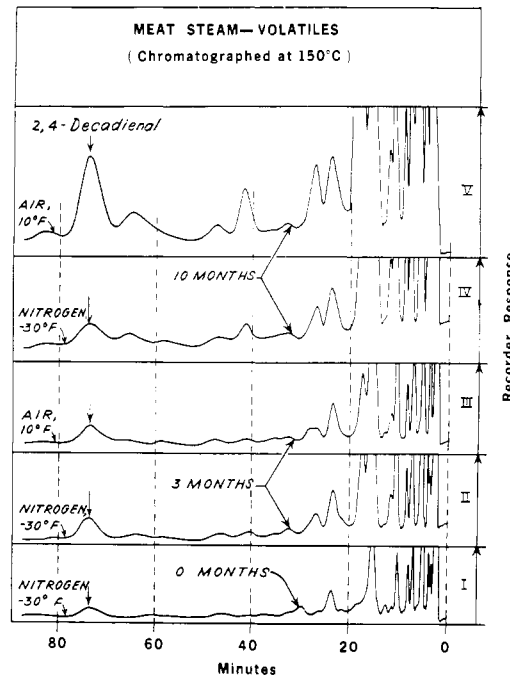


Figure 5. Gas chromatograms of meat steam volatiles from air-packed and nitrogen-packed fried chicken at 0, 3, and 10 months' storage

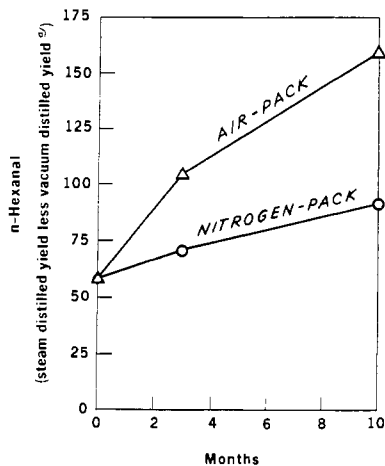


Figure 6. Quantity of *n*-hexanal thermally produced during steam distillation as a function of storage time and storage condition

^aPeak area weight of steam-distilled *n*-hexanal (mg.) minus peak area weight of vacuum-distilled *n*-hexanal (mg.)

during oxidative deterioration of potato granules (2).

Steam distillation at 100° C. generally gave a greater quantity of volatile material than did vacuum distillation. The difference in yield is, therefore, an indication of the quantity of volatile material thermally produced during

steam distillation at 100° C. Plotting the difference in *n*-hexanal yield from the two distillation procedures against storage time (Figure 6) shows that the quantity of thermally produced *n*-hexanal, too, increased faster during storage in the air pack than it did in the nitrogen pack. Therefore, the quantity of volatile material thermally produced during steam distillation was also evidently a function of the extent of oxidative deterioration that occurred during storage.

However, a substantial quantity of thermally produced *n*-hexanal was evident in meat steam volatiles at zero storage time when there was no off-flavor (Figure 6). Hence, mere presence of substantial amounts of *n*-hexanal in volatiles does not necessarily indicate off-flavor. Instead it is the increase in *n*-hexanal, above its initial value when no off-flavor is present, that follows the development of oxidative off-flavor.

In contrast to the substantial amount of thermally produced *n*-hexanal evident at zero storage time, very little vacuum-distilled *n*-hexanal was present at zero storage (peak area weight, 3 mg.). Nevertheless, the quantity of vacuum-distilled *n*-hexanal was greater at 10 months' storage in the air pack (peak area weight, 100 mg.) than in the

nitrogen pack (peak area weight, 17 mg.). Therefore, the increase of vacuum distilled *n*-hexanal also follows the development of off-flavor.

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Literature Cited

- (1) Berry, N. W., McKerrigan, A. A., *J. Sci. Food Agr.* **9**, 693 (1958).
- (2) Boggs, M. B., Buttery, R. G., Venstrom, D. W., Belote, M. L., *Food Sci.* **29**, 487 (1964).
- (3) Davis, J. G., Hanson, H. L., *Food Technol.* **7**, 335 (1954).
- (4) Gaddis, A. M., Ellis, R., Currie, G. T., *Food Res.* **24**, 283 (1959).
- (5) Hanson, H. L., Fletcher, L. R., Lineweaver, H., *Food Technol.* **13**, 221 (1959).
- (6) Phippen, E. L., Nonaka, M., *J. Food Sci.* **28**, 334 (1963).
- (7) Roessler, E. B., Warren, J., Guymon, J. F., *Food Res.* **13**, 503 (1948).
- (8) Stone, H. W., Skavinski, E. R., *Ind. Eng. Chem., Anal. Ed.* **17**, 495 (1945).

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