Gas Chromatography-Mass Spectroscopy Investigations on the Flavor Chemistry of Oat Groats

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Dried oat groat volatiles were characterized by GC-MS in an effort to study the inherent flavor chemicals in oats before processing. Volatile isolation by vacuum distillation and Tenax headspace trapping, both before and after hydration, resulted in compositionally different isolates. Dry vacuum distillates primarily contained $C_{10}H_{16}$ monoterpenes, alkylbenzenes, and hexanal. Vacuum steam distillation resulted in large increases of volatiles presumed to originate via residual enzyme activity on oat lipids. Major amounts of C_4-C_6 alcohols, hexanal, 1-octen-3-ol, nonanal, and (*E,E*)- and (*Z,E*)-3,5-octadien-2-one were found in the hydrated groat isolates. Headspace isolation, dry and hydrated, also exhibited this difference. Oat groats do not appear to contain large amounts of inherent flavor components but depend on further processing for oat flavor development.

Oat cereal products have been consumed throughout the world for centuries but little is known about the flavor chemistry associated with them. Maga (1978) has recently reviewed the literature on cereal volatiles in general and was surprised at the scarcity of information available on the flavor chemistry of these important food staples.

Oats have a unique and characteristic flavor containing nutty, grain impressions. The only study on oat volatiles is a report by Hrdlicka and Janicek (1964) describing the presence of carbonyls in toasted oat flakes. They regarded carbonyls as one of the factors in toasted oat flavor but not totally responsible for the flavor impressions. Heydanek and McGorrin (1980) have recently reported that pyrazines are also important in the overall perception of oat flavor.

In an effort to define the important aspects of flavor chemistry associated with oats and oat-containing products, the present study was undertaken. This report details the isolation and identification of flavor volatiles from dried oat groats by GC-MS. Dried oat groats are the commercial precursors of oatmeal, oat flakes, and oat flour. It is of interest to detail the volatile compsition of this starting material in order to facilitate further study on flavor development in oat systems.

EXPERIMENTAL SECTION

Materials. Dried oat groats (A grade, *Avena sativa*) were obtained from commercial production streams. The dehulled oats which have been dried to 7.5% moisture represent the commercial starting material for the manufacture of oatmeal, oat flakes, and oat flour.

Authentic chemical compounds were obtained from commercial sources (e.g., Aldrich Chemical Co., Eastman Chemical Co., Pyrazine Specialities, and California Aromatics).

Chemical Synthesis. The following chemical standards were synthesized for structure elucidation.

2-(n-Pentyl)furan. By substantial modification of the general method of Gilman and Calloway (1933), a solution of 1.12 g (16.6 mmol) of furan in 4 mL of dry benzene was added dropwise to a solution of 2.00 g (16.6 mmol) of valeryl chloride and 4.30 g (16.6 mmol) of anhydrous stannic chloride in 20 mL of benzene at 0° over 25 min. After being stirred an additional 15 min at 0 °C and 10 min at 25 °C, the reaction mixture was extracted into ether, washed with sodium bicarbonate, and dried to afford 1.83 g (72%) of a yellow oil identified as 1-(2-furyl)pentan-1-one: bp 116-119 °C (16 mmHg); IR (neat) 2980, 1670 (C=O), 1570, 1460 cm^{-1}; MS (70 eV) 152 (0.7, M^+), 123 (5), 110 (73), 96 (6), 95 (100), 81 (7), 67 (8), 41 (7), 39 (22).

The ketone (430 mg, 2.8 mmol) was added to a solution of 320 mg (5.7 mmol) of potassium hydroxide and 0.5 mL of 85% hydrazine hydrate in 2.8 mL of diethylene glycol and heated at 100 °C for 1.5 h. The hydrazone intermediate was decomposed by heating at 220 °C for 3 h. Product isolation (Et_2O, $NaHCO_3$) furnished 224 mg (58%) of a pale yellow oil identified as 2-(*n*-pentyl)furan: bp 162 °C; MS (70 eV) 138 (10, M^+), 120 (4), 105 (9), 95 (4), 82 (22), 81 (100), 67 (4), 53 (12), 41 (4).

(E,E)- and (Z,E)-3,5-Octadien-2-one. A solution of 1.00 g (11.9 mmol) of (*E*)-2-pentenal in 5 mL of THF was added dropwise to a suspension of 624 mg (13.0 mmol) of 50% sodium hydride and 754 mg (13.0 mmol) of acetone in 30 mL of THF at 25 °C. After the mixture was stirred for 1 h at 25 °C, the reaction was quenched with 10% aqueous HCl, the solvent was evaporated, and the resulting yellow oil was taken up into ether, washed, and dried over sodium sulfate. Analysis by GC-MS showed the oil to consist of a 40:1 mixture of (*Z,E*)- and (*E,E*)-3,5-octadien-2-ones, whose mass spectra were identical: MS (70 eV) 124 (28, M^+), 109 (10), 96 (6), 95 (100), 81 (52), 79 (41), 77 (14), 65 (9), 53 (16), 43 (20), 41 (7), 39 (11). In addition, the mixture contained traces of 3-hydroxypentanal and unreacted (*E*)-2-pentenal.

Capillary GC-MS Analysis. GC-MS analysis was done on a Hewlett-Packard 5840A GC coupled to a H-P 5982 mass spectrometer via a Pt-Ir open splitter. Data was acquired on a H-P 5933 disc-drive data system. Chromatography was carried out with a 0.5 mm i.d. Pyrex glass WCOT column coated with SE-30. Temperature programming was done by holding the starting temperature at 40 °C for 3 min and then programming at 3 °C/min to 170 °C and holding. Helium at a $\bar{u}$ of 27 cm/s was the carrier gas. Electron ionization was at 70 eV.

Kovats GLC indexes were measured relative to other components in the mixture as internal standards. Authentic compound retention was determined as a mixture with the series of *n*-alkanes.

Total Volatile Isolation. Dry vacuum distillation of oat groats was done in the following manner. An 8-kg sample of oat groats was placed in a 12-L flask and immersed in a 55 °C water bath. The oat groats were then vacuum distilled at 0.02 torr for 4 h. Condensation was effected by immersing a 1-L receiver flask, which was connected in series to two high efficiency spiral cold finger

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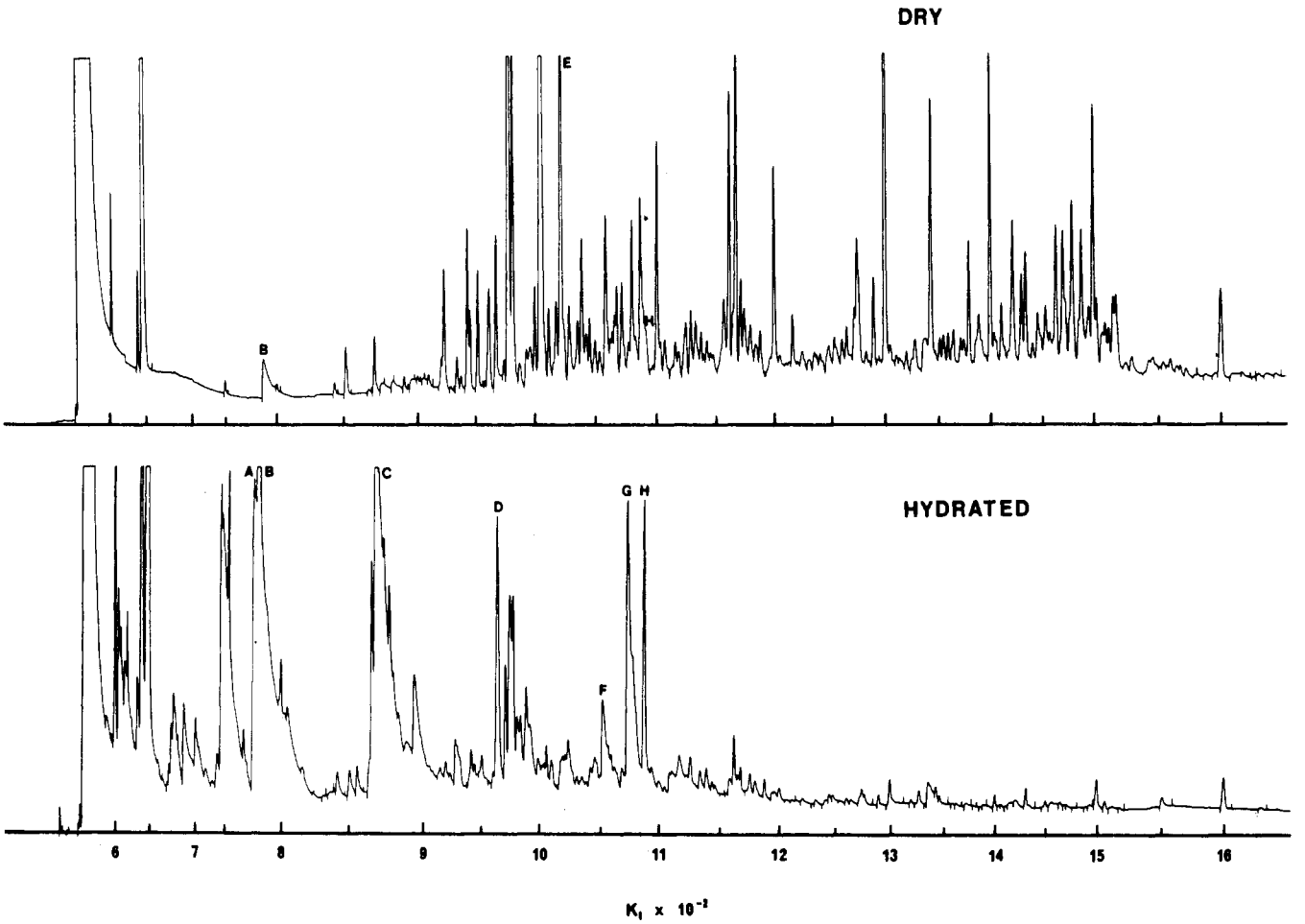

Figure 1. SE-30 capillary GC FID responses of oat groat total volatile isolates as a function of Kovats retention indexes. Dry isolation (upper) and hydrated isolation (lower) are recorded at 256× and 1×, respectively. Peak identifications correspond to those listed in Table I.

traps, all in dry ice–2-propanol. The frozen distillate (~50 mL) was thawed in cold water and rinsed out of the flask and traps with a total of 25 mL of methylene chloride. Further extraction (3 × 5 mL of CH_2Cl_2) was carried out, and the organic layers were combined, dried with Na_2SO_4, and reduced to a 5-mL volume by distillation through a low holdup column. One milliliter of this extract was evaporated under a stream of dry nitrogen to 50 μL, and a 3-μL aliquot was injected onto a SE-30 GC capillary column for generation of the chromatogram in Figure 1.

The hydrated isolate from oat groats was similarly prepared except that 8 L of odor-free water was added to the distillation flask. The groat slurry was allowed to stand 1 h in the 55 °C water bath before distillation in vacuo at 20 torr was begun. An additional spiral cold water condenser was added in front of the 1-L flask in a dry ice–2-propanol bath to precondense the larger volume of water distilled. A total of 1300 mL of distillate was collected in 4 h of distillation. The combined distillates were made 10% (w/v) with NaCl and extracted (5 × 80 mL) with CH_2Cl_2. The extracts were dried, reduced in volume, and concentrated for GC analysis as above.

Headspace Analysis. Isolation of headspace volatiles was performed by using a Hewlett-Packard 7675A automated purge and trap sampler, coupled to a Hewlett-Packard 5840A gas chromatograph. The purge sampler was modified with a standard taper 24/40 male adapter to accommodate larger sample volumes. A 300-g sample of dried oat groats was placed in a 1-L round-bottom flask, connected to the headspace trapping system, and heated either dry or in the presence of 300 mL of water at 30 °C in a thermostated water bath. Dry helium, 170 mL/min, swept 10.2 L of headspace volatiles over a 1-h period onto a porous polymer catridge (1.3 cm^3 of 60–80-mesh Tenax GC contained in a 3 mm i.d. × 105 mm stainless steel tube) held at 25 °C. The volatiles were thermally desorbed at 200 °C directly onto the 50-m SE-30 capillary column for GC–MS as described above, except that the gas chromatograph was temperature programmed for the following conditions: an initial 8-min hold at −10 °C during desorption, increasing to 26 °C at 12 °C/min, followed by 3 °C/min to 170 °C and holding for 30 min.

Sensory Evaluation of Distillates. Heydanek and McGorrin (1980) showed that true flavor fidelity was maintained in vacuum steam distillates from oat-based food sources. The flavor of vacuum distillates in this study was compared to that of the original material by taste evaluations of the distillate water. Five cubic centimeters of the distillate water was tasted from a spoon and compared to either direct mastication of the intact groats or the water from a 50% suspension of groats. The original groats as well as the distillates obtained had a raw oat grain, hay–feedy, grassy flavor with only a trace of cooked oat identity as determined by a five-membered trained laboratory panel.

RESULTS AND DISCUSSION

Total Volatile Analysis. In an effort to develop a base of knowledge on the flavor chemistry of oats, both total volatile and purge trap headspace analyses were employed in the study of dried oat groat volatiles. Total volatile anlaysis was designed to evaluate materials with a Kovats

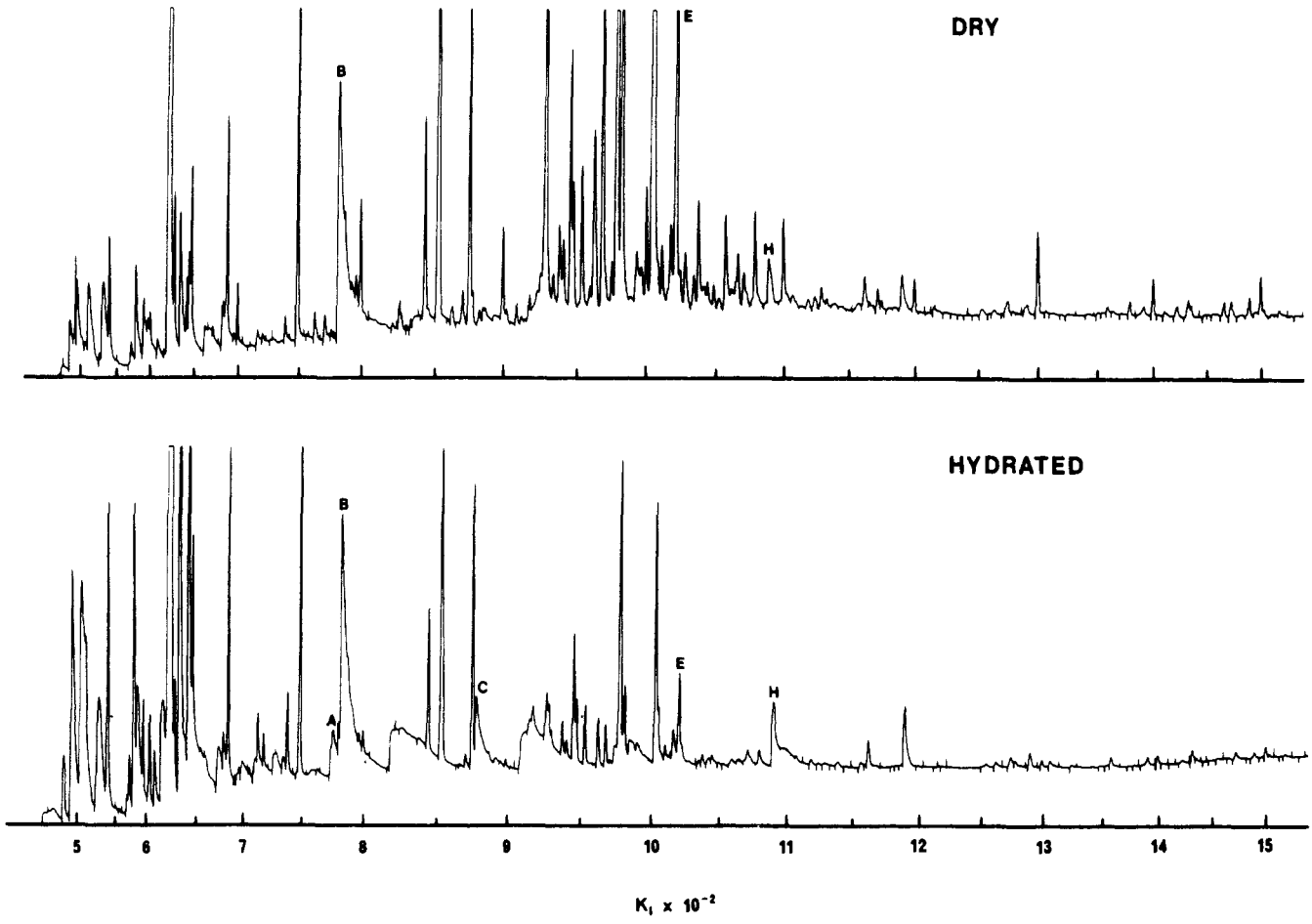

Figure 2. SE-30 capillary GC FID responses of Tenax-trapped oat groat headspace volatiles as a function of Kovats retention indexes. Groats purged dry (upper) and hydrated (lower) are recorded at the same attenuation scale. Peak identifications correspond to those listed in Table II.

index (K_I) of >600, and the headspace technique employed to identify the lower boiling components. During our studies a certain variability in the types and amounts of volatiles was noted in the system containing added water for vacuum steam distillation. For comparison purposes, oat groats volatiles were isolated under high vacuum without added water and the volatile profile changed dramatically. Figure 1 illustrates the differences in total volatiles noted between dry isolation and with water added to the distillation. The relative concentrations between the two chromatograms varies widely. A major peak such as limonene (peak E) in the dry vacuum isolate represents a 10-ppb concentration. Hexanal (peak B) in the hydrated isolation represents 3–5 ppm.

Table I lists the components identified by GC–MS analysis of both total volatile isolates. The dry vacuum isolate, which represented the weak grainy, hay–straw odor of dry groats, contains mostly hydrocarbon materials. The principal components are $C_{10}H_{16}$ terpenes, alkylbenzenes, and some oxygenated constituents. No single component appears responsible for the entire odor.

In contrast, volatiles isolated by vacuum steam distillation showed a much higher level of oxygenates as well as a completely different profile of components. The distillates now had a more green cereal-type odor/flavor. The major components observed are 3-methyl-1-butanol, 1-pentanol, 1-hexanol, hexanal, 1-octen-3-ol, (*E*,*E*)- and (*Z*,*E*)-3,5-octadien-2-one, and nonanal. In addition, many other components associated with metabolism or enzymatic activity such as alcohols and aldehydes were found; those most notable from a flavor standpoint include 3-methylbutanal, 2,4-decadienal, and benzaldehyde. Traces of the major components from the dry vacuum isolate were also found in the hydrated isolate. It appears that when the isolation is carried out in the presence of water, considerable enzymatic activity is still present in the dried oat groat and the volatiles produced by this activity are superimposed on the volatiles inherent in the dry groat.

Considering the type of volatiles produced, C_5 and C_6 alcohols and aldehydes, it appears that lipoxygenase and aldehyde oxidoreductase activity are present in hydrated dried oat groats. Since these volatiles comprise some 80% of the total isolated volatiles from the hydrated system, their specific production seems indicative of these enzymes. Similar production of C_5 and C_6 alcohol and aldehyde components has been shown to arise from the above enzyme systems in green beans and seeds (deLumen et al., 1978), soybeans and corn (Leu, 1974), and alfalfa seeds (Esselman and Clagett, 1974). Oats are known to contain lipoxygenase (Heimann et at., 1975) and, in general, a very complex enzyme system (Shukla, 1975). Therefore it is not surprising to find enzymatic activity; however, it is critical to a flavor study to know that enzymes are active and functional during a flavor isolation process.

Headspace Analysis. Tenax trapping of headspace volatiles from oat groats, both dry and hydrated, showed similar results to those obtained in the total volatile analysis. Figure 2 compares the headspace profiles obtained by purging dry (upper) and hydrated (lower) groats. Table II lists the components identified by GC–MS analysis of these volatiles. Most notable in the hydrated sample is the increase in C_5–C_6 alcohols and a general increase in the level of oxygenated components. Although the changes do not appear as dramatic in a 1-h incubation

Table I. Compounds Identified in the Total Volatile Fraction of Oat Groats by GC–MS

Kovats GC index (K_I)[a]	compound	std match[b]	dry[c]	hydrated[c]	Kovats GC index (K_I)[a]	compound	std match[b]	dry[c]	hydrated[c]
605	ethyl acetate	++		+	1021 (E)	limonene	++	+	
619	2-methyl-1-propanol	++		+	1023	4-methyldecane	+	+	
628	3-methylbutanal	++		+	1035	*o*-diethylbenzene	+	+	
641	benzene	++	+	+	1042	propyltoluene	+	+	
651	cyclohexane	+	+	+	1044	3-ethyl-1,2-dimethylbenzene	+	+	
669	pentanal	++		+	1046 (F)	(*Z,E*)-3,5-octadien-2-one	++	+	+
689	3-pentanol	+		+	1049	γ-terpinene	++	+	
699	2-pentanol	+		+	1053	*n*-propyltoluene	+	+	
720	pyridine	++		+	1055	2,5-dimethyl-3-ethylpyrazine	++		+
743	3-methyl-1-butanol	++	+	+	1062	3,5-dimethyl-2-ethylpyrazine	++		+
748	toluene	++	+	+	1065	dimethylethylbenzene	+	+	
754 (A)	1-pentanol	++		+	1069 (G)	(*E,E*)-3,5-octadien-2-one	++	+	+
774 (B)	hexanal	++	+	+	1071	dimethylethylbenzene	+	+	
782	ethyl butyrate	++		+	1072	dimethylstyrene	+	+	
798	2-methylpyrazine	++		+	1079	alloocimene	+	+	
800	octane	++	+	+	1084 (H)	nonanal	++	+	+
814	furfural	++	+	+	1091	dimethylethylbenzene	+	+	
845	ethylbenzene	++	+		1097	isophorone	+	+	
854	*m/p*-xylene	++	+		1100	undecane	++	+	
858 (C)	1-hexanol	++		+	1101	2-phenylethanol	++		+
860	γ-butyrolactone	+		+	1103	dimethylethylbenzene	+	+	
867	2-heptanone	++	+	+	1106	dimethylethylbenzene	+	+	
869	styrene	++	+		1108	2-formylimidazole	+	+	+
875	*o*-xylene	++	+	+	1113	4-decanone	+	+	
878	heptanal	++	+	+	1122	ethylstyrene	+	+	
883	2,5-dimethylpyrazine	++	+	+	1156	borneol	++		+
895	*n*-pentyl acetate	++		+	1160	diethyl fumarate	+		+
900	nonane	++	+		1162	naphthalene	++	+	+
910	isopropylbenzene	++	+		1172	$C_{13}H_{28}$	+	+	
930	α-pinene	++	+		1174	2-decanone	++		+
931	benzaldehyde	++		+	1193	benzothiazole	+		+
937	dimethylpyridine	++		+	1200	dodecane	++	+	
940	*n*-propylbenzene	++	+		1216	$C_{13}H_{28}$	+	+	
943	camphene	++	+		1274	2-methylnaphthalene	++	+	
947	*m*-ethyltoluene	++	+	+	1289	1-methylnaphthalene	++	+	
948	2,5-diethylfuran	+		+	1293	2,4-decadien-1-al	++		+
949	*p*-ethyltoluene	++	+	+	1300	tridecane	++	+	
956	1,3,5-trimethylbenzene	++	+	+	1324	γ-nonalactone	++	+	+
961	*tert*-butylbenzene	++	+		1380	$C_{15}H_{32}$	+	+	
964	*o*-ethyltoluene	++	+	+	1400	tetradecane	++	+	+
970	β-pinene	++	+		1411	$C_{15}H_{24}$ sesquiterpene	+	+	
971 (D)	1-octen-3-ol	++		+	1422	$C_{15}H_{24}$ sesquiterpene	+	+	
972	2-octanone	++	+		1434	α-cedrene	+	+	
978	2-(*n*-pentyl)furan	++	+	+	1446	$C_{15}H_{26}$	+	+	
979	1,2,4-trimethylbenzene	++	+	+	1458	biphenyl	+	+	
982	myrcene	++	+		1463	$C_{16}H_{34}$	+	+	
986	3-octanol	+		+	1471	$C_{15}H_{22}$	+	+	
988	dichlorobenzene	+	+		1479	$C_{15}H_{24}$ sesquiterpene	+	+	
994	2-octanol	+		+	1488	pentadecene	++	+	
997	*sec*-butylbenzene	+	+		1500	pentadecane	++	+	+
1000	decane	++	+		1507	methyl laurate	+		+
1004	Δ^3-carene	++	+	+	1509	$C_{15}H_{24}$ sesquiterpene	+	+	
1007	1,2,3-trimethylbenzene	+	+		1516	α-cadiene	+	+	
1011	*p*-cymene	++	+		1559	diethyl phthlate	++		+
1016	*o*-methylstyrene	+	+		1600	hexadecane	++	+	+
1018	3-octen-2-one	++	+	+					

[a] K_I index and peak identification corresponding to Figure 1. [b] (+) matches published MS and has appropriate K_I. (++) matches authentic compound K_I and MS. [c] (+) denotes the presence in the isolated volatiles from these preparation methods.

at 60 °C by headspace analysis, the Tenax trapping method is not ideally suited for retaining lower boiling alcohols. Therefore, we are not sure all of the alcohol present was retained or purged; however, distinct increases in these components were noted. Again the two chromatograms represent widely differing component concentrations. In the dry isolate limonene (peak E) represents 10 ppb, whereas the hexanal content (peak B) of the hydrated sample correlates with a 3-ppm spike of hexanal added to a water–groat slurry and immediately purged.

The most abundant components noted in the dry purge system are 1,2-dichloroethane, hexanal, toluene, *p*-xylene, 2-pentylfuran, and myrcene. In the lower boiling fraction eluting before hexanal, there are no components other than dimethyl sulfide and dimethyl disulfide that appear to be potentially flavor significant. In contrast, the hydrated isolate contained 2-methylpropanal, ethanol, acetone, 2-butanone, 3-methylbutanal, 3-pentanone, and pentanal in the lower boiling fraction. These results again suggest residual enzymatic activity present in the hydrated groat.

The combined use of total volatile analysis and headspace trapping effectively allowed analysis of the entire spectrum of volatiles arising from oat groats. The presence of large amounts of alcohols in both hydrated systems

Table II. Headspace Volatiles (K_I < 800) Identified from Oat Groats Using GC-MS

Kovats GC index (K_I)[a]	compound	std match[b]	dry[c]	hydrated[c]
478	acetone	++	+	+
485	ethanol	++	+	+
493	dimethyl sulfide	+	+	+
506	1,2-propanediol	++	+	+
538	2-methylpropanal	++	+	+
565	2,3-butanedione	++	+	+
572	1-propanol	++	+	+
575	2-butanone	++	+	+
593	2-methylfuran	+	+	+
605	chloroform	++	+	+
607	ethyl acetate	++	+	+
612	2-methyl-1-propanol	++		+
623	1,2-dichloroethane	++	+	+
630	1,1,1-trichloroethane	++	+	+
634	3-methylbutanal	++	+	+
643	3-pentanone	++	+	+
644	benzene	++	+	+
649	carbon tetrachloride	++	+	+
679	pentanal	++	+	+
683	trichloroethylene	++	+	+
688	2-ethylfuran	+	+	+
700	heptane	++	+	+
716	methylcyclohexane	+	+	+
727	dimethyl disulfide	+	+	+
739	1,1,2-trichloroethane	++	+	+
749	toluene	++	+	+
772 (A)	1-pentanol	++		+
778 (B)	hexanal	++	+	+
796	tetrachloroethylene	++	+	+
800	octane	++	+	
827	ethylcyclohexane	+	+	
878	2-(*n*-butyl)furan	+	+	
884 (C)	1-hexanol	++	+	
1021 (E)	limonene	++	+	+
1086 (H)	nonanal	++	+	+

[a] K_I index and peak identification corresponding to Figure 2. [b] (+) matches published MS and has appropriate K_I. (++) matches authentic compound K_I and MS. [c] (+) denotes the presence in the isolated volatiles from these preparation methods.

caused some aberration in the chromatographic systems due to tailing and column overloading. However, the profiles obtained illustrate the fact that these alcohols are abundant in the hydrated system, offering a clue to the processes taking place.

The fact that residual enzyme activity may be present in dried cereal grains must be taken into account whenever flavor isolations are conducted. Two completely different conclusions as to the type of flavor components present in an oat groat would be drawn depending on the isolation technique used. In essence, dried oat groats derive their weak hay grassy odor from very low levels of monoterpenes and hexanal. These would be expected to make little contribution to oat flavor in cooked, hydrated, or processed systems. Therefore, further studies on the flavor of oat containing systems would be expected to show the cooking and/or processing method to be highly important to the ultimate flavor.

In this context, the presence of small but detectable amounts of 2-methyl, 2,5-dimethyl-, and C_4-substituted pyrazines in oat groats would suggest some browning takes place during the initial production drying process. More severe heat treatment would be expected to produce larger amounts of these type components, and in fact, Heydanek and McGorrin (1980) have shown this to be the case in oatmeal.

The presence of relatively large amounts of (*E,E*)- and (*Z,E*)-3,5-octadien-2-one (peaks F and G, Figure 1) are of interest from a formation standpoint. They appear as minor components in the dry isolate but are quite abundant in the isolate from the hydrated system. These dienones have been found in red beans (Buttery et al., 1975), green beans (Murray et al., 1976), cooked asparagus (Tressel et al., 1977), oxidized linolenate (Badings, 1970), and autooxidized methyl docosahexaenoate (Noble and Nawar, 1975). All evidence points to their origin being from a lipid oxidation reaction, either enzymatic and/or chemical, but why they appear specifically in oats is unknown. Badings (1970) measured a flavor threshold of 0.30 ppm in paraffin oil for the dienone and described its flavor as fruity, fatty. Synthesis of this dienone and isolation of the *E,E* and *Z,E* isomers were undertaken. Their odor appears to be more grassy, straw-like than fatty when diluted in air and resembles to an extent the grassy–straw character in oats. Further study on the flavor significance of these components to oat flavor is contemplated.

CONCLUSIONS

Oat groats were found to have varying volatile composition dependent on the method of isolation. Dried oat groats, commercial precursors of most oat products, had very low volatile levels consisting mainly of $C_{10}H_{16}$ terpenes, alkylbenzenes, and hexanal. Little inherent flavor contribution would be expected in further processed oat products.

Dried oat groats hydrated before isolation of volatiles contain a much higher level of oxygenated volatile material, the major components being alcohols and aldehydes most likely produced by residual enzymatic activity in the oat groat. These alcohols and aldehydes are thought to be derived by enzymatic lipid oxidation and reduction of liberated aldehydes to alcohols by an aldehyde oxidoreductase. The present study indicates that research on oat flavor will have to consider residual enzyme activity as well as processing conditions in order to understand the flavor chemistry of oat-containing products.

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