Volatile Compounds from Potato-like Model Systems

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The volatile reaction products of aqueous mixtures comprising combinations of methionine, glucose, linoleic acid, and starch heated in a modified Likens−Nickerson apparatus were extracted and analyzed by gas chromatography−mass spectrometry (GC-MS). The majority of volatile compounds were formed from linoleic acid degradation, hexanal, 2,4-decadienal, and 2-pentylfuran being identified in the greatest amounts. Dimethyl disulfide and dimethyl trisulfide were detected in every system containing methionine. 3-(Methylthio)propanal (methional) and other sulfur compounds were detected when methionine was heated with another precursor. No binding of volatile compounds to starch was observed; rather, starch appeared to act as an additional source of reactive carbohydrate. Almost all the components identified have been identified among the aroma components of cooked potato. No pyrazines, pyridines, or thiazoles were identified, probably due to the relatively low temperature/high moisture conditions.

Keywords: *Potato volatiles; glucose; methionine; linoleic acid; Maillard reaction; model systems*

INTRODUCTION

The common potato (*Solanum tuberosum*) is grown in many parts of the world, even in extreme climates (Maga, 1994). One reason for its popularity is that the tuber can be prepared in a number of different ways including boiling, baking, and frying. In addition, it is used in the dehydrated form by the food industry and is an important component of many ready meals. The volatile components of the various forms of cooked potato, and also of the raw vegetable, have been the subject of considerable study (Whitfield and Last, 1991; Maga, 1994). More than 250 compounds have been identified in boiled or baked potatoes alone (Maga, 1994; Nijssen et al., 1996).

In most cooked foods, including potatoes, the Maillard reaction and lipid degradation are two of the most important reactions leading to the formation of aroma compounds. The compounds most important to baked potato aroma include pyrazines, 2,4-decadienal, and 3-(methylthio)propanal (methional) (Buttery et al., 1973). Coleman et al. (1981) also identified thiazoles, furans, acids, alcohols, esters, and ketones, while Nursten and Sheen (1974) detected aldehydes, alcohols, 2-ethylfuran, and 2-pentylfuran in boiled potato aroma, and Josephson and Lindsay (1987a) demonstrated the importance of *cis*-4-heptenal in potato flavor.

Herz and Shallenberger (1960) suggested that the reaction involving methionine and glucose heated at 100 °C was important in the generation of potato flavor. More recently, methionine/glucose and methionine sulfoxide/glucose model systems were reported to produce cooked potato-like aromas on heating for 1 h at 180 °C, and methional, furans, and pyrazines were among the major reaction products (Yu and Ho, 1995).

Volatiles generated from lipid−amino acid−sugar interactions have been reviewed by Whitfield (1992).

Most of the published work concerning the effect of lipid on the Maillard reaction has been undertaken by Farmer, Mottram, and Whitfield (e.g., Whitfield et al., 1988; Farmer et al., 1989; Farmer and Mottram, 1990). They used a meat model system, i.e., aqueous ribose-cysteine-phospholipid model systems heated at 140 °C for 1 h and established that, in the presence of phospholipid, furans, thiophenes, and pyridines possessing long-chain alkyl substituents (deriving from lipid) are formed while levels of other heterocyclic compounds are modified (Farmer and Mottram, 1990). The study presented here was concerned with the investigation of the volatile products of a boiled potato-like model system. Various combinations of methionine, glucose, linoleic acid, and starch were used. Levels of reactants were chosen to reflect those typically present in uncooked potato flesh. The aim was to obtain a clearer understanding of the chemistry involved in the production of volatile compounds in cooked potato.

EXPERIMENTAL PROCEDURES

Materials. α-D-Glucose (99+%, ACS reagent), L-methionine (98+%), and potato starch (soluble) were obtained from Aldrich Chemical Co., Gillingham, U.K., and linoleic acid (*cis*-9,*cis*-12-octadecadienoic acid, 99+%) was purchased from the Sigma Chemical Co., Poole, Dorset, U.K. Redistilled pentane and diethyl ether were used as solvents.

Preparation of Model Systems and Volatile Isolates. Model systems contained water (400 mL) and combinations of glucose (13 g L^{-1}, 0.0721 mol L^{-1}), methionine (20 g L^{-1}, 0.1415 mol L^{-1}), linoleic acid (0.5 g L^{-1}) and starch (50 g L^{-1}) (see Table 1). A blank (water only) was also prepared. Solutions with linoleic acid were mixed before heating with a Ultra-Turrax T25 blender (Janke and Kunkel, IKA-Labortechnik, Staufen, Germany) to allow a better dissolution. Potato starch (50 g L^{-1}) was added when the solution was boiling to a homogeneous mixture. They were refluxed (100−105 °C) for 2 h in a 1 L round-bottomed flask of a modified Likens−Nickerson simultaneous steam distillation−solvent extraction apparatus (Maarse and Kepner, 1970). The extraction solvent

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Table 1. Composition of Model Systems and Odor after Heating

code	reactants	odor of heated model system
G	glucose	nd[a]
M	methionine	sulfury
L	linoleic acid	nd
S	starch	nd
M/G	methionine and glucose	boiled potato, boiled cabbage
L/G	linoleic acid and glucose	nd
L/M	linoleic acid and methionine	sulfury
L/M/G	linoleic acid, methionine and glucose	roasted potato, boiled vegetables, hash browns
L/M/G/S	linoleic acid, methionine, glucose and starch	roasted potato, boiled vegetables, hash browns

[a] nd, no detectable odor.

was pentane:ether (9:1, v:v, 30 mL) and it contained 0.1 mL of a solution of 1,2-dicholorobenzene in ether (130.6 mg L^{-1}) as internal standard.

The pH of the model systems before heating was 6.3, except for the blank and G (pH 5.3) and S (pH 7.0). A Russell (Auchtermuchty, U.K.) autoclavable pH electrode was used to monitor the pH during heating. The pH was adjusted and maintained at pH 6 by the addition of 0.5 M NaOH or HCl solutions during heating.

Model systems and the blank were prepared in triplicate.

Following extraction, residual water was removed from the solvent extract by storing it at −18 °C for 18 h. Extracts were then concentrated to 0.5−1.0 mL using a Kuderna−Danish apparatus, fitted with a Vigreux distillation column, at 45 °C. The resulting volatile isolates were analyzed by gas chromatography−mass spectrometry (GC-MS) within 1 week of preparation.

GC-MS. Volatile isolates were analyzed using a Hewlett-Packard (HP, Bracknell, U.K.) 5890 series II gas chromatograph coupled to a HP 5972 series mass spectrometer, equipped with a BPX5 fused silica capillary column (50 m × 0.32 mm internal diameter, 0.25 μm film thickness) obtained from SGE (Milton Keynes, U.K.). Significant analytical conditions were helium carrier flow rate, 1.0 mL/min; injector temperature, 250 °C; transfer line temperature, 280 °C; injection volume, 1 μL; injection technique, split/splitless; temperature program, 50 °C for 5 min followed by a ramp rate of 4 °C/min to a final temperature of 250 °C, held for 5 min. The MS was operated in the electron impact (EI) full scan mode; electron energy, 70 eV; ion source temperature, 175 °C; scan time, 1 s; mass range, 35−400 amu.

Retention time and quantitative data were obtained using HP MS Chemstation software G1034C. Linear retention indices (LRI) for individual compounds were calculated based on data obtained for a series of external standard C$_6$−C$_{22}$ *n*-alkanes in diethyl ether using the same analytical conditions. Identifications of components were made by comparing retention mass spectra and linear retention indices (LRI) with those of authentic compounds. Where both were consistent, identifications were considered positive. When only mass spectra data were consistent, identities were tentative. Individual components were quantified by the use of the internal standard (1,2-dicholorobenzene).

RESULTS AND DISCUSSION

The chosen levels of glucose, methionine, and linoleic acid in the model systems were about the same as levels of total sugars (1.3 g/100 g), total amino acids (1.909 g/100 g) and total fatty acids (ca. 0.06 g/100 g) in raw potato (Paul and Southgate, 1978). The level of starch used was about one-third of the amount in raw potato (14.8−16.6 g/100 g). This was the maximum amount that could be used without any charring occurring during heating. The pH of the model systems was maintained at pH 6, representative of the pH range (5.25−6.67) of potato tubers. This pH range is due to varietal differences (Maga, 1994).

The odors of the heated model systems are given in Table 1. The presence of both glucose and methionine were required for the generation of a potato odor. Roasted potato/hash brown notes were detected only in the presence of linoleic acid. Systems M/G and L/G/M were potato flesh color (yellow) after heating. The color darkened in the presence of starch (system L/M/G/S).

Identification of Compounds in the Model Systems. Twenty-eight compounds were identified in the model systems. Nine of these were tentative (by mass spectral data only) or partial identifications. Their identities, LRI values, and quantitative data are given in Table 2. The components accounted for at least 95% of the total peak area of each system. Only four compounds were identified in blank extracts, and they have been excluded from Table 2.

The identified compounds can be grouped into those generated from the thermal degradation of the individual model system starting materials, i.e., methionine, linoleic acid, glucose and starch, and those generated from the thermal interaction of two or more of these components.

No compounds were detected in G, apart from those which were present in the blank. This reflects the stability of glucose when heated alone in aqueous solution at 100 °C (Belitz and Grosch, 1987).

Alkanes, alkenes, alcohols, and hexadecanoic acid were identified in S. Potato starch may contain traces of lipids which can lead to the formation of these compounds.

Compounds Requiring Methionine for Their Formation. Low levels of two compounds came from the degradation of methionine alone (M), i.e., dimethyl disulfide and dimethyl trisulfide. Amounts were 2−6-fold higher in M/G and L/M. However, the amounts increased greatly, by 13−19-fold, in L/M/G and in L/M/G/S, compared to M. Dimethyl disulfide was the second most abundant compound (after methional) in L/M/G and L/M/G/S, while dimethyl trisulfide was present in amounts comparable to those for hexanal and 2-pentylfuran, some of the most abundant lipid degradation products formed in L/M/G and L/M/G/S.

Yu and Ho (1995) report that the addition of glucose to an aqueous methionine solution increased the yield of dimethyl disulfide by 17-fold. They suggested that a portion of the methional, formed by reaction of glucose with methionine, may degrade to methanethiol, which in turn could yield dimethyl disulfide and dimethyl trisulfide.

When oxidized lipid (or another oxidizing agent) is present, methionine has been shown to be easily oxidized to methionine sulfoxide (Yu and Ho, 1995). These authors have also shown that the formation of dimethyl sulfides was favored over methional in the methionine sulfoxide system compared to the methionine system.

Table 2. Volatile Compounds Identified in Aqueous Model Systems Comprising Combinations of Methionine, Glucose, Linoleic Acid, and Starch

compound	ref LRI[a]	exptl LRI[b]	micrograms of compound recovered (from 400 mL of solution)[f]								
			G[c]	M[c]	L[c]	S[c]	M/G[c]	L/G[c]	L/M[c]	L/M/G[c]	L/M/G/S[c]
Compounds Generated from Methionine											
dimethyl disulfide[i,k]	722	753	nd[d]	3	nd	nd	6	nd	6	39	54
dimethyl trisulfide[k]	990	985	nd	1	nd	nd	4	nd	6	18	19
Compounds Generated from Linoleic Acid											
1-pentanol[h,i,k]	765	771	nd	nd	3	nd	nd	2	1	2	3
hexanal[h,i,j,k]	809	808	nd	nd	16	nd	nd	28	15	21	17
heptanal[h,i,j,k]	913	910	nd	nd	2	nd	nd	2	nd	nd	1
2-heptenal[i,j]	940	968	nd	nd	6	nd	nd	8	7	8	10
1-octen-3-ol[h,i,j]	986	987	nd	nd	1	nd	nd	3	nd	nd	nd
2-pentylfuran[h,i,j,k]	993	994	nd	nd	nd	nd	nd	1	12	18	17
2-ethylhexanal[j,k]		1035	nd	nd	nd	nd	nd	nd	4	nd	nd
(E)−2-octenal[h,i]	1069	1070	nd	nd	3	nd	nd	4	4	4	4
an alcohol		1102	nd	nd	2	nd	nd	2	1	1	nd
(E)−2-nonenal[h,i,j]	1172	1171	nd	nd	1	nd	nd	1	2	1	2
octanoic acid[k]	1186	1182	nd	nd	6	nd	nd	4	nd	nd	nd
decanal[i,j,k]	1204	1214	nd	nd	1	nd	nd	1	1	1	nd
2,4-nonadienal[h,i,j]	1218	1230	nd	nd	2	nd	nd	2	2	2	2
(E,Z)−2,4-decadienal[h,j,k]	1309	1308	nd	nd	26	nd	nd	43	5	7	9
(E,E)−2,4-decadienal[h,i,j,k]	1318	1334	nd	nd	53	nd	nd	76	15	26	37
Compounds Generated from Starch[g]											
an alkane		1197	nd	nd	nd	2	nd	nd	nd	nd	1
tridecane[k]	1300	1297	nd	nd	nd	4	nd	nd	nd	nd	2
a branched chain alcohol		1391	nd	nd	nd	1	nd	nd	nd	nd	2
a branched chain alkane		1448	nd	nd	nd	2	nd	nd	nd	nd	1
hexadecanoic acid	1966	1980	nd	nd	nd	69	nd	nd	nd	nd	97
Compound Generated from Interaction between Glucose and Methionine											
methional[i,j,k]	909	924	nd	nd	nd	nd	36	nd	nd	106	145
Compounds Generated from Interactions between Glucose, Methionine, and Linoleic Acid											
2-methyl-5-(methylthio)furan		1018	nd	nd	nd	nd	nd	nd	nd	2	3
methyl methane-thiosulfonate ($CH_3SO_2SCH_3$)		1083	nd	nd	nd	nd	nd	nd	nd	1	nd
2-hexylthiophene		1280	nd	nd	nd	nd	nd	nd	nd	1	nd
Compounds Generated from Interactions between Glucose, Methionine, Linoleic Acid, and Starch											
2,4,5-trithiahexane		1154	nd	nd	nd	nd	nd	nd	nd	nd	1
dimethyl tetrasulfide[k]	1251	1250	nd	nd	nd	nd	nd	nd	nd	nd	tr[e]

[a] Reference LRI, linear retention index of the authentic compound (Acree and Arn, 1997; Baltes and Bochman, 1987; Kondjoyan and Berdagué, 1996; laboratory data compilations). [b] Experimental LRI, LRI of the sample compound. [c] See footnote to Table 1. [d] nd, not detected. [e] tr, trace. [f] Means of at least three replicate model systems (relative standard deviation ranging from 2 to 25%). [g] These compounds are all likely to come from degradation of trace lipid in starch. [h] Identified in raw potatoes (Nijssen et al., 1996). [i] Identified in boiled potatoes (Nijssen et al., 1996). [j] Identified in baked potatoes (Nijssen et al., 1996). [k] Identified in french fried potatoes (Nijssen et al., 1996).

Levels of dimethyl disulfide were more than 7-fold greater in their methionine sulfoxide-glucose system compared to methionine-glucose, and dimethyl trisulfide and dimethyl tetrasulfide were also identified.

It appears that, in the current study, the addition of lipid and of lipid plus starch to the sugar−methionine system results in unexpectedly higher levels of dimethyl sulfides compared to systems M, M/G, and L/M. Glucose facilitates the degradation of methionine while oxidized linoleic acid can facilitate its oxidation to methionine sulfoxide, which in turn yields sulfides more readily than methionine (Yu and Ho, 1995). Starch represents an additional source of reactive carbohydrate. Given the presence of glucose, methionine, lipid, and starch in potato, it is surprising that dimethyl disulfide has only been detected among volatile compounds of boiled or baked potato by Gumbman and Burr (1964) (Table 2).

Compounds Requiring Linoleic Acid for Their Formation. By far the majority of the compounds identified in this study derive from lipid degradation. They include 10 aldehydes, 3 alcohols, and one furan. The most abundant lipid-derived compounds in this study were hexanal, 2,4-decadienal, and 2-pentylfuran. Oxidation of linoleic acid gives a mixture of mainly the 9- and 13-hydroperoxides, which are readily interconverted (Frankel, 1982). Thus, they give the same volatile decomposition products, but in different amounts. The most abundant compound in systems L and L/G was 2,4-decadienal. Of all the systems studied, the amount of 2,4-decadienal was highest in L/G and lowest in L/M. Interactions between sugars and carbonyl compounds, such as those derived from lipid, have not been studied by flavor chemists (Whitfield, 1992). However, initiation of the oxidation of either carbohydrates or of lipids in physiological systems stimulates the oxidation of the other (Baynes, 1996). These autoxidation reactions generate reactive oxygen species, which will promote lipid oxidation.

The low amount of 2,4-decadienal in L/M may be related to the identification of 2-pentylfuran in that system. Since 2-pentylfuran is formed from 2,4-decadienal, which is itself formed from 9-linoleate hydroperoxide, the higher production of 2-pentylfuran may indicate more extensive degradation reactions in the presence of methionine. Methionine can degrade on its own under the chosen experimental conditions (M) to give products capable of reacting with carbonyl compounds formed by the degradation of linoleic acid and thus promoting lipid oxidation. Another explanation for the lower yield of 2,4-decadienal in L/M is that this compound may react with methionine, giving nonvolatile products.

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The higher levels of 2-pentylfuran in L/M/G and L/M/G/S, compared to L/M, was surprising since some Maillard reaction products are reported to act as antioxidants (Whitfield, 1992). The formation of free radicals in the early stages of the Maillard reaction may provide the key to the formation of the observed higher levels of 2-pentylfuran.

The third abundant lipid-derived compound identified in this study is hexanal. It is a major volatile carbonyl coming from 13-linoleate hydroperoxide (Frankel, 1982). A number of groups have studied the formation of hexanal from linoleic acid, and various mechanisms have been proposed. For example, Schieberle and Grosch (1981) demonstrated its formation from 13-linoleate hydroperoxide and from compounds having a double bond in the ω-6-position, e.g., 9-linoleate hydroperoxide, 2,4-decadienal and 2-octenal. Josephson and Lindsay (1987b) have shown that 2,4-decadienal can undergo retro-aldol condensation to yield 2-octenal (also identified here) which, in turn, may degrade via a similar mechanism to hexanal. Hexanal was present at relatively similar levels (15–28 μg) in all systems containing linoleic acid, in contrast to 2,4-decadienal (30–119 μg) and 2-pentylfuran (not detected, –18 μg). Thus, it appears that whatever the pathways leading to hexanal in the current study, its formation is less affected by glucose and methionine than the formation of 2,4-decadienal and 2-pentylfuran.

The formation of the remaining lipid degradation compounds reported in Table 2 has been explained previously (Schieberle and Grosch, 1981; Frankel, 1982; Josephson and Lindsay, 1987b).

All of the lipid-derived compounds reported here have been identified among the volatile components of cooked potato (Table 2). Many possess fatty aromas.

Compounds Formed by Methionine/Glucose Interactions. The only compound detected in M/G which was not present in M or G was methional, the Strecker aldehyde of methionine. Yu and Ho (1995) reported that methional can be generated from methionine alone through the Shigematsu reaction, by decarboxylation and deamination of the amino acid. Methional was the major component of the extracts from M/G, L/M/G, and L/M/G/S. Its absence from M indicates that the Shigematsu reaction did not occur under the experimental conditions used here.

The amount of methional increased in L/M/G and L/M/G/S, possibly due to the greater amounts of α-dicarbonyl compounds, which are required for the Strecker degradation of methionine.

The odor of methional has been described as "cooked potato" (Table 2). In contrast to its presence as a major reaction product in M/G, L/M/G, and L/M/G/S, methional has been detected quantitatively as a minor compound in all studies on boiled or baked potato flavor (Maga, 1994; Nijssen et al., 1996).

No pyrazines, furans, thiazoles, oxazoles, and thiophenes, which are known products of the Maillard reaction and which are important compounds in the flavor of baked potatoes, were identified. In most cases, their absence may be attributed to the relatively low reaction temperature/high moisture conditions, while the absence of 5-hydroxymethylfurfural may be attributed to the relatively high pH.

Compounds Formed by Linoleic Acid/Methionine/Glucose Interactions. Three volatile sulfur compounds, i.e., 2-methyl-5-(methylthio)furan, methyl meth-

anethiosulfonate ($CH_3SO_2SCH_3$), and 2-hexylthiophene, were identified in L/M/G and are attributed to thermal interactions of linoleic acid, methionine, and glucose.

As previously mentioned, methionine has been found to be easily oxidized to methionine sulfoxide, especially when an oxidizing agent, such as oxidized lipid, is present. Yu and Ho (1995) identified 2-methyl-5-(methylthio)furan and methyl methanethiosulfonate in methionine sulfoxide and methionine sulfoxide + glucose model systems. Lower levels of 2-methyl-5-(methylthio)-furan were identified on heating methionine alone, but neither compound was identified in the glucose–methionine system. No reaction pathways were reported but the results presented here support a route involving oxidation of methionine to its sulfoxide.

2-Hexylthiophene has been identified in mixtures containing cysteine, ribose, and phospholipids (Whitfield et al., 1988; Farmer and Mottram, 1990). The authors proposed a reaction pathway between 2,4-decadienal and hydrogen sulfide, which can give 2-hexyl-thiophene (Farmer and Mottram, 1990).

The low number and amounts of compounds from the interaction of glucose, methionine and linoleic acid are probably due to two main reasons. First, the temperature of the model systems was low (100–105 °C), and second, lipid oxidation products may mask some interaction products on the gas chromatogram. The three compounds which were identified have not been reported among the flavor components of boiled or baked potatoes or of french fries (Nijssen et al., 1996).

Compounds Formed by Linoleic Acid/Methionine/Glucose/Starch Interactions. Two volatile sulfur compounds, i.e., 2,4,5-trithiahexane and dimethyl tetrasulfide, were detected in small quantities in M/G/L/S only, but have not been reported among the volatile compounds of boiled or baked potato (Maga, 1994; Nijssen et al., 1996).

When starch was added to the system, the amounts of most of the volatile compounds remained the same or increased slightly, compared to M/G/L. This result was unexpected because starch has the ability to bind certain components, thereby making them difficult to extract and thus quantify (Maga, 1994). Hau et al. (1994) studied the binding of aroma volatiles to starch in a closed system at 25 °C and found that the presence of starch in solution decreased the percentage of volatiles in the headspace.

BeMiller (1965) showed that the rate of hydrolysis of starch depends on the pH and the temperature of the system. At slightly acid pH, the hydrolysis rate increases with temperature. The hydrolysis of starch leads to D-glucose and reversion products and greatly increases the number of carbonyl groups. This could account for increases in levels of some compounds in L/M/G/S.

CONCLUSION

This study demonstrates the occurrence of important interactions among flavor precursors in a heated model system. By heating each component alone, as well as pairs of reactants, the ability of one component to influence the degradation of the second has been shown. The data emphasize the importance of studying multicomponent model systems and indicate the drawbacks of binary systems. Nevertheless, the composition of the most complex system studied here, i.e., L/M/G/S, is still far removed from that of the target food, i.e., potato.

Further work is required to understand interactions between, e.g., different amino acids and other fatty acids in such a system and these studies are in progress.

ABBREVIATIONS USED

GC-MS, gas chromatography−mass spectrometry; LRI, linear retention indices; G, system containing glucose; L, system containing linoleic acid; M, system containing methionine; S, system containing starch; M/G, system containing methionine and glucose; L/G, system containing linoleic acid and glucose; L/M, system containing linoleic acid and methionine; L/M/G, system containing linoleic acid, methionine and glucose; L/M/G/S, system containing linoleic acid, methionine, glucose and starch.

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