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Flavour volatiles of 'malt beverage' from roasted Sorghum

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Flavour volatile compounds of sorghum malt beverage were collected and concentrated on a Tenax GC and identified on a GC-MS. Collected and separated compounds were evaluated for their aroma impression. Twenty eight volatile compounds were detected in the sorghum malt beverage. These were made up of pyrazines, furans, aldehydes, ketones, esters and alcohols. The ketones, aldehydes and esters contributed a much higher percentage peak area to the malt beverage volatiles than the heterocyclic compounds and alcohols. The sorghum malt beverage produced a characteristic nutty, sweet chocolate aroma which was not easily tied to a single volatile compound. Copyright © 1996 Elsevier Science Ltd

INTRODUCTION

Malt is the major ingredient in the production of alcoholic beverages such as beer, stout and ale (Hough *et al.*, 1971). In Nigeria, barley malt is not available because of the recent ban on its importation. Also, barley is a temperate grain which cannot be grown locally in commercial quantity that will satisfy the industry's requirement. Presently, the industry uses locally available cereals, particularly malted and unmalted sorghum, for brewing with the addition of external enzymes for saccharification.

Much work has been done on the malting and brewing qualities of sorghum (Skinner, 1976; Okafor & Aniche, 1980; Lasekan, 1991), but so far there has been little published study on the flavour volatiles of this beverage. To ensure the continued growth of the brewing potential of sorghum malt, it is necessary to understand the components influencing its flavour. This will also enhance scientific manipulation of the beverage in the immediate future. The present study describes the collection and identification of the flavour volatile compounds of sorghum malt beverage.

MATERIALS AND METHODS

Sorghum variety SSV3 collected from the International Crops Research Institute for semi-arid tropics (ICRISAT), Kano, Nigeria, was used for this study. The variety has a yellow colour and thin pericarp, and is of the non-waxy endosperm type with an endosperm texture which is intermediate between corneous and floury. Its malt has a high enzyme content (Ilori *et al.*, 1990).

Malt beverage production

Sorghum (5 kg) was cleaned by removing foreign and broken kernels. The sample was then steeped in 500 ppm formaldehyde solution for 18 h, and malted as previously described (Lasekan, 1993). The sorghum malt sample (500 g) was roasted in a forced air oven (200°C, 2 h) to give a brownish endosperm colour. Fifty grams (50 g) of roasted malt was milled with a roller mill to a particle size of about 600 μm (Ilori *et al.*, 1990). The milled sample was mashed using the conventional mashing profile of barley malt, adapted for sorghum malt (Ilori *et al.*, 1990). After saccharification, filtration was carried out by pouring the mash onto a 200 μm mesh screen to obtain about 1.2 l of wort. The wort was further clarified by centrifugation for 15 min at 16,000 r.p.m. (Centrifuge type UJ3, No. 60749, manufactured by PMT, Tomson, Zoetermeer, Holland). Sugar (18%) was added to the wort to increase the original gravity to 1.0582. Twenty percent (20% v/v) of caramel prepared from one kilogram of granulated sugar (Ogundiwin & Ilori, 1991) was added to the wort to give a colour value of 21.0° EBC.

Three replicates of malt beverage for gas chromatography-mass spectrometry (GC-MS) were filtered and pasteurised in a water bath at 60°C for 30 min in 25 ml McCartney bottles and stored at 0°C until analysed.

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Trapping of volatiles

Trapping of volatile was by the reported methods of Uzochukwu *et al.* (1994). Volatiles were trapped on Tenax GC (SGE Pty Ltd, Australia) by bubbling helium through one arm of a T-shaped test-tube containing 2 ml of the sample. This drew the volatiles into the second arm of the tube, to which a glass-lined metal tube packed with the Tenax GC was attached. Collection was for 30 min at 25°C. The rate of helium flow was 10 ml/min. An overnight collection to obtain more of the higher boiling components was also carried out on the malt beverage.

GC

GC was carried out on a Hewlett Packard 588 level 4 Chromatograph equipped with a flame ionisation detector. The column was a 50 m×0.32 mm id stainless-steel capillary column coated with CP Sil 19 CB (Chrompack, Netherlands) of film thickness 0.22 μm. The injection system was an SGE unijector in concentrator-headspace mode (SGE Pty Ltd, Australia) at 250°C. The volatiles trapped on the Tenax GC were desorbed in the unijector onto the CP Sil column, the first 80 mm of which was cooled in a stream of gaseous-liquid nitrogen to condense the volatiles. Oven temperature was held at 5°C for 5 min, then 5–60°C at 30 min^{-1}, 60–210°C at 6°C min^{-1} and held at 210°C for 10 min.

GC-MS

The gas chromatograph was a Carlo-Erba, 'Mega Series'. The volatiles trapped on the Tenax GC were desorbed as described above onto the same CP Sil column. The GC was operated in a split–splitless mode, the value being opened after 20 s. Other operating conditions were the same as for the GC examinations described above. For all of the GC analysis, the carrier gas was helium flowing at 1.5 ml min^{-1}. The MS was a Kratos MS 80 RFA model with a D.S. 90 data system. Its operating potential was 70 eV at 200°C. Ionisation was by electron impact. The identification of compounds was performed by comparing mass spectra using a library search program and published collections. Relative retention behaviour of authentic samples was also checked against those obtained for identified compounds, to be certain that compounds eluted at the proper points.

AROMA ASSESSMENT

To evaluate the aroma significance of separated components, odours associated with GC peaks were assessed by GC sniff, using the method of Williams & Tucknott (1978), which involves performing two chromatographic runs, the second with the flames extinguished. Odour components were then recorded, and the markers established on the recorder chart were correlated with peaks. The proportions of components, excluding solvents, were calculated using peak areas obtained from the chromatograms using the Hewlett Packard 588 instrument. GC sniff was conducted by three people on two replicate samples. The result presented is an integration of all descriptions collected for the six sniffing sessions. Descriptions and corresponding retention times were recorded by the same person sniffing, using a stop watch (Uzochukwu *et al.*, 1994).

RESULTS AND DISCUSSION

The results of GC-MS analyses of the volatiles obtained from sorghum malt beverage are outlined in Figure 1 and Table 1. Altogether, twenty eight compounds were identified in the sorghum malt beverage. Among these were three alcohols, seven aldehydes, four ketones, two esters and one lactone; eight heterocyclic compounds of six membered rings, two other heterocylic compounds and one hydrocarbon. The beverage also contained two odorants (Nos. 15 and 20) of unknown chemical structure. The volatiles with the major peaks comprised of butan-1-01, 1-hydroxy-2-propanone, 2-acetylfuran, 2-furan carboxylic acid methyl ester, 3-furan carboxylic acid methyl ester, 2-acetyl-4-methylphenol, styrene, 2-furfural, 3-methyl dihydrofuran 2,5-dione and 2,5-dimethylpyrazine.

In principle, the volatiles detected can be classified into three main groups: compounds which are formed by the degradation of starch, compounds which are formed by reaction between starch and protein (Maillard reaction) and compounds formed during caramel production.

Important products produced by the reaction of carbohydrates and amine compounds are the pyrazines. The pyrazines have been recognized as important flavour constituents of a large number of cooked, roasted and toasted foods (Maga, 1982).

The production of pyrazines from the reaction of carbohydrates and amine compounds has been studied extensively over the past several years. Hodge *et al.* (1972) proposed that amino acids and carbohydrates were important precursors for pyrazines formed during the non-enzymatic browning reaction. Ferretti *et al.* (1970) obtained pyrazines from the reaction of lactose with casein. Formation pathways have also been proposed for pyrazines. Rizzi (1972) proposed that sugars react with amines with the formation of alpha-amino carbonyl intermediates, which condense to produce pyrazine compounds.

Other detected volatiles such as benzaldehyde and phenylacetaldehyde have been reported in extruded cereals (Pfannhauser, 1993) and in model systems of corn starch, zein and corn oil (Huang *et al.*, 1987).

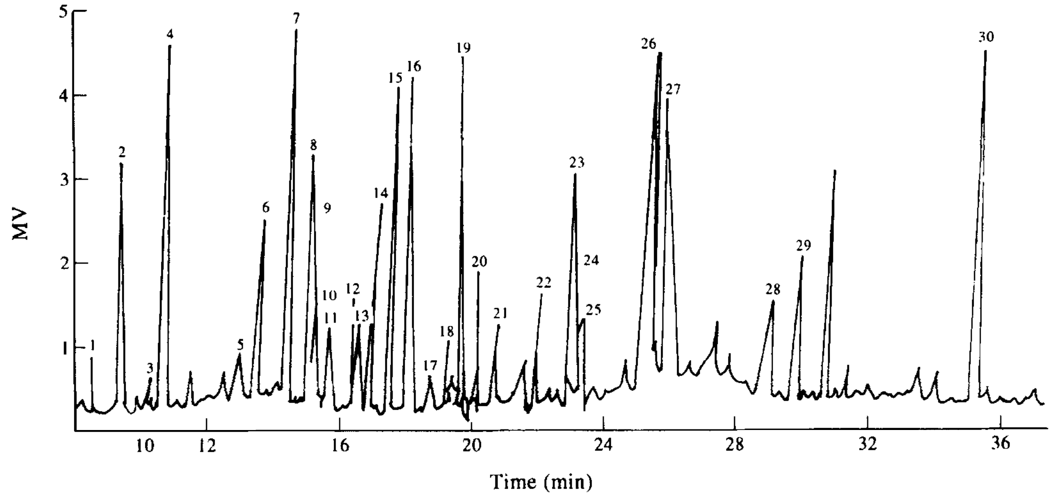

Fig. 1. Gas chromatogram of sorghum malt beverage. Peak numbers correspond to those of the compounds in Table 1.

Table 1. Volatiles identified in sorghum malt beverage

Peak No.	Compound	Odour impression	Retention index (*RI, Polar*) Obs.	Lit.[9]	*Peak area (%)
1.	Hexanal	green grass	1060	1059[1]	0.2
2.	Styrene	fruity			5.3
3.	Pentan-2-ol	pungent, intense	1100	1100[1]	0.2
4.	Butan-1-ol		900	900[2]	8.3
5.	2-Pentylfuran	fresh flower, pungent	1124	1120[1]	0.2
6.	2-Methylpyrazine	nutty	1240	1238[1]	1.2
7.	1-Hydroxy-2-propanone		1172	1177[2]	10.8
8.	2,5-Dimethylpyrazine	nutty, sweet, roasty	1287	1291[3]	5.3
9.	2,6-Dimethylpyrazine	nutty, sweet, fruity	1300	1300[3]	1.2
10.	2-Ethylpyrazine	roasty, sweet	1307	1316[1]	0.6
11.	2,3-Dimethylpyrazine	nutty, sweet	1314	1318[3]	0.3
12.	2-Ethyl-6-methylpyrazine	roasty, sweet	1361	1364[1]	0.6
13.	2-Ethyl-5-methylpyrazine	roasty, sweet	1382	1381[1]	0.3
14.	Trimethylpyrazine	nutty	1391	1391[1]	1.3
15.	Unknown	Aldehydey			5.7
16.	2-acetylfuran		1441	1440[2]	7.4
17.	Butyrolactone	estery	1460	1456[2]	0.2
18.	Non-2-enal	lime, citrus, licorice	1536	1547[2]	0.3
19.	2-Furfural	smoky, burnt	1427	1427[1]	6.3
20.	Unknown				1.2
21.	Benzaldehyde	nutty, almonds	1487	1490[1]	0.4
22.	5-Methyl-2-furfural	nutty	1536	1546[1]	0.6
23.	Phenylacetaldehyde	horney-like fragrance	1610	1614[1]	3.8
24.	2-Furanmethanol		1623	1619[2]	0.7
25.	Octane-2, 3-dione	termitey	1629	1630[1]	0.3
26.	2-Furan Carboxylic acid, methylester	estery	2015	2015[2]	10.3
27.	3-Furancarboxylic acid, methylester	estery	1772	1771[2]	8.2
28.	Deca-2, 4-dienal	Aldehydey	1807	1807[2]	1.2
29.	3-Methyldihydrofuran -2,5-dione	like apple sauce	1840	1842[1]	5.1
30.	2-acetyl-4-methylphenol	Burnet	2178	2178[1]	9.8

* Mean of two determinations.
9: Literature RI: [1] Pfannhauser (1993); [2] Pons *et al.* (1991); [3] Buttery *et al.* (1988).
Obs: observed.

Benzaldehyde and phenylacetaldehyde are thermal reaction products of deca-2, 4-dienal and hexanal (Pfannhauser, 1993). 2-Pentylfuran which is another likely products of deca-2, 4-dienal has also been detected in fresh and stored triticale products, in model extrusion tests (Huang *et al.*, 1987). Benzaldehyde, phenylacetaldehyde, 2-pentylfuran and non-2-enal have also been detected in cooked rice (Buttery *et al.*, 1988).

5-Methyl-2-furfural and pentan-2-ol were detected in rye bread and white bread crusts, respectively (Pfannhauser, 1993). Other volatiles found and not previously described in cereal products are butan-1-ol, 1-hydroxy-2-propanone, 2-furfural, 2-acetyl furan, butyrolactone, 2-furanmethanol, 2-furan carboxylic acid methyl ester, 3-furan carboxylic acid methyl ester and styrene. With the exception of styrene, the other volatiles have been detected in aromatic caramel (Pons *et al.*, 1991). It is probable, therefore, that these volatiles were formed in the caramel. The likely origin of the styrene is the Tenax GC, as Lewis & Williams (1980) have reported the presence of styrene in volatiles obtained by heating blank Tenax GC. The sorghum malt beverage gave a characteristic, nutty sweet chocolate aroma which decreases in intensity with time. This characteristic aroma is most probably due to the combination of volatile compounds detected in this study. However, the contribution of some undetected volatiles to the characteristic sorghum malt beverage aroma cannot be ruled out.

CONCLUSION

Twenty eight volatile compounds were detected in the sorghum malt beverage. The volatiles could conveniently be classified into three main groups, compounds which were formed by the degradation of starch, compounds formed by reaction between starch and protein and those formed in caramel. Although, the present study have shown that the aroma of sorghum malt beverage is made up of an array of different compounds (ketones, esters, alcohols and aldehydes), the possibility of other undetected compounds contributing to the flavour cannot be ruled out.

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