Volatile Component Analysis of Commercially Milled Head and Broken Rice

M.A. MONSOOR AND A. PROCTOR

ABSTRACT: The volatile components of commercially milled head and broken rice samples and the effect of water washing on volatile components were investigated over 30 d of storage at 37 °C and 70% relative humidity. Pentanal, pentanol, hexanal, pentylfuran, octanal, and nonanal were the major volatile components identified by gas chromatography-mass spectrometry (GC-MS) analysis in both head and broken rice samples. The concentration of volatile components was greater in broken rice than in head rice and increased significantly during the storage period for both rice types. Water washing was found to be effective in reducing the volatile components for both head and broken rice.

Keywords: broken rice, GC-MS analysis, head rice, volatile components, water washing

Introduction

In the United States, 16% of the rice for domestic consumption is used by the brewing industry (USDA 2001). One fraction of very small broken kernels is traditionally called "brewers rice" because brewers used to use this fraction of broken rice for beer manufacture. Modern beer makers use head rice despite its higher price because it is believed to produce less undesirable volatile compounds relative to broken rice, although no studies have been conducted to show this. Development of undesirable volatile compounds during milled rice storage and transport can subsequently adversely affect beer flavor quality.

Factors affecting the off-flavor development in milled rice are temperature, time, exposure to oxygen, and the amount of surface bran lipids on the milled rice. Lipids in residual bran on the surface of the milled rice are converted to free fatty acids (FFA) by bran lipases (Tanako 1989; Ohta and others 1990). These FFA rapidly oxidize to yield off-flavors (Yasumatsu and others 1966; Aibra and others 1986; Tanako 1993) that can adversely affect beer quality when the rice is used as a source of fermentable carbohydrate. Suzuki and others (1999) found that the amount of hexanal, pentanal, and pentanol in raw brown rice markedly increased during storage at 35 °C for 2, 4 and 6 wk. Widaja and others (1996a, 1996b) studied the changes of volatile compounds of rough, brown, and milled rice and found that there was an increase of total volatile compounds in all 3 forms of rice. They also found that the increase of volatile compounds was far greater for milled than for rough or brown rice because of the presence of aldehydes and ketones commonly formed as a result of the lipid oxidation processes of the residual bran. Lam and Proctor (2003a) showed that oleic and linoleic acids were the major FFA produced during rice lipid hydrolysis and most of the volatile components in milled rice are the breakdown products of their monohydroperoxides (Lam and Proctor 2003b). They (Lam and Proctor 2003b) also studied the formation of volatile compounds of partially and fully milled rice during storage at 37 °C and 70% relative humidity for 50 d. Seventeen volatile compounds were identified in both partially and fully milled raw rice that included alkanes, alcohols, aldehydes, ketones, furans, alkenes, and aromatics. They also found that the concentrations of 2-nonenal, hexanal, and octanal were high and changed significantly during storage in both partially and fully milled rice. However, there have been no studies of the oxidative behavior of broken kernels relative to that of head rice.

Monsoor and Proctor (2003) found that broken rice had significantly greater surface lipid and FFA than head rice. They later described how surface lipid, FFA, and lipid oxidation could be dramatically reduced by a simple rapid water washing treatment (Monsoor and Proctor 2002, 2003). However, the off-flavor volatile components of head and broken rice were not investigated. Therefore, this study will examine the effect of water washing on the off-flavor development on head and broken rice. More specifically, the objectives of this study were (1) to determine the volatile component of commercially milled head and broken rice during storage and (2) to determine the effect of water washing on the volatile component of head and broken rice.

Materials and Methods

Rice sample

Commercially milled head and broken rice of mixed long grain varieties (Wells, Cocodrie, and Drew) were obtained from Riceland Foods (Stuttgart, Ark., U.S.A.). The volatile component formation of these samples was monitored during storage.

Storage study

The head and broken rice samples were placed on aluminum trays in a laboratory humidity oven (Hotpack, Philadelphia, Pa., U.S.A.) at 37 °C and 70% relative humidity (RH). This condition was chosen to represent the optimum temperature and relative humidity for rice bran lipase. Triplicate rice samples (100 g) were taken from the oven for each rice population at 0 d, 15 d, and 30 d of storage for volatile component analysis. The head and broken rice samples from each storage duration were randomly divided into 2 populations (50 g each). One population was subjected to water washing and the other used as an unwashed control.

MS 20040182 Submitted 3/23/04, Revised 5/6/04, Accepted 7/7/04. *The authors are with Dept. of Food Science, Univ. of Arkansas, 2650 N Young Ave., Fayetteville, AR 72704. Direct inquiries to author Proctor (E-mail: aproctor@uark.edu).*

Water washing

Before volatile component analysis, the portion of the stored rice samples subjected to water washing study (50 g) was washed with equal volumes (50 mL) of deionized water for 5 min as described by Monsoor and Proctor (2002). The other portion of the stored rice sample from the same storage duration was kept unwashed to compare the volatile components of water washed rice with an unwashed control.

Aqueous extraction of volatile components

Water was selected as a solvent to extract volatiles because beer acquires off-flavors from the rice by aqueous extraction. An aqueous extraction protocol developed by the brewing industry (Anheuser Busch Co., St. Louis, Mo., U.S.A.) was used to extract milled rice water soluble volatiles. In the brewing industry, the aqueous extracts of milled rice commonly called "rice tea" is prepared to evaluate their flavor notes by a group of trained panelists to determine the acceptability of milled rice as a brewing adjunct. The head and broken rice (water washed and unwashed control) samples (10 g) from each storage duration were extracted with 100 mL of water for volatile components at 70 °C for 10 min. The aqueous extracts were obtained by filtering through cheesecloth to separate the rice. The extracts (10 mL) were placed in an amber gas chromatography (GC) vial (15 mL), and the vial was sealed airtight with a Teflon-coated rubber septum and an aluminum cap. Triplicate rice samples from the stored head and broken rice produced 3 aqueous extracts for each stored head and broken rice.

Chromatographic analysis of volatile component

Headspace volatiles of the triplicate samples were extracted by solid phase microextraction (SPME) using a carboxen/polydimethylsiloxane (CAR/PDMS) (Supelco Inc., Bellfonte, Pa., U.S.A.) fiber. An internal standard 1,2 dichloro benzene (10 parts per billion [ppb]) was added to each vial before volatile component analysis. Volatile components in the aqueous extracts were absorbed to the CAR/PDMS fiber inserted into the headspace of the vial placed in a Varian Autotherm Heater (70 °C) for 30 min with agitation. The absorbed volatile components in the fiber were desorbed in an injector port of a gas chromatograph at 270 °C for 2 min, and then separated by GC. The Varian STAR 3400 CX gas chromatograph (Varian Analytical Instruments, Walnut Creek, Calif., U.S.A.) was equipped with a Varian 8200 CX autosampler (Varian Analytical Instruments) and a CP-Sil8 CB-MS fused silica capillary column (30 m × 0.25-mm inner dia, 0.25-m film thickness; Varian Analytical Instruments). The GC oven temperature was increased from 50 °C to 200 °C at a rate of 5 °C/min. The holding time for initial and final temperatures was 5 and 1 min, respectively.

Volatile component identification

A Varian (Saturn 2000) mass selective detector (scan range, 35 to 350 mg) was used to identify the volatile components. The mass selective interface and ionization source temperature, manifold temperature, and transfer line temperature were 280 °C, 80 °C, and 200 °C, respectively. All mass spectra were obtained at 70 eV. The compounds were identified by a combination of NIST mass spectral library (Saturn GC-MS Workstation, version 5.51) and the gas chromatographic retention times of the standard compounds.

Volatile component quantification

Quantification of individual volatile components in aqueous extracts was based on an internal standard method. The concentration (ppb) of the volatile components was calculated using the response factor (RF) determined for each volatile compound. To obtain RF for individual volatile components, a reference mixture consisting of a known amount of the standard volatile compounds and internal standard (1,2-dichloro benzene) was subjected to the gas chromatography-mass spectrometry (GC-MS) analysis. The RF for the individual volatile compounds was calculated as follows: $RF_s = (A_{is}/C_{is})\ (C_s/A_s)$, where A_{is} and C_{is} refer the area and concentration of the internal standard, and C_s and A_s refer to the concentration and area of the given standard volatile compounds. The formula used to calculate the concentration for volatile compounds was $C_{vc} = (A_{vc}.C_{is})/(A_{is}.RF_{vc})$ (Ha and others 1989), where C_{vc}, A_{vc}, and RF_{vc} are the concentration, peak area, and response factor, respectively, of the volatile compounds, and C_{is} and A_{is} are the concentration and peak area, respectively, of the internal standard.

Statistical analysis

Analysis of variance (ANOVA) was conducted on the data using the JMP Version 5.0.1 (SAS Inst. Inc., Cary, N.C., U.S.A.) with rice type (head and broken rice), treatment (washed and unwashed rice), and storage time (0 d, 15 d, and 30 d) as factors for analysis. A Student *t* test was used to differentiate mean values, with significance defined at $P < 0.05$.

Results and Discussion

Volatile components in the aqueous extracts

The compounds identified in aqueous extracts of the rice samples were 4 alkanes (hexane, 3-methyl pentane, dodecane, and tetradecane), 12 aldehydes (pentanal, 3-methyl butanal, 2-pentenal, hexanal, 2,4 hexadienal, heptanal, benzaldehyde, octanal, 2-octenal, nonanal, 2-nonenal, and decanal), 6 alcohols (pentanol, hexanol, 2,7-dimethyl octanol, heptanol, 2-butyl octanol, and 2-ethyl decanol), 4 aromatics (1,3,5-trimethyl benzene, 2-pentyl furan, 1,2 bis-cyclobutane, and butylated hydroxytolune), and 1 ketone (2-heptanone) (Figure 1; Table 1). However, 70 volatile compounds were identified from rice flour (Bullard and Holguin 1977). The higher number of volatile compounds was probably due to heating and subsequent reaction during milling. Twelve volatile components were identified in stored brown rice (Suzuki and others 1999) and 17 compounds in partially and fully milled rice (Lam and Proctor 2003b). Most of the compounds reported in Table 1 have been identified in cooked (Yajima and others 1978, 1979; Tsugita 1986; Buttery and others 1988) and raw rice (Withycombe and others 1978; Suvarnalatha and others 1994; Suzuki and others 1999; Lam and Proctor 2003b). No new compounds were formed during storage in addition to those formed at 0 d, but the concen-

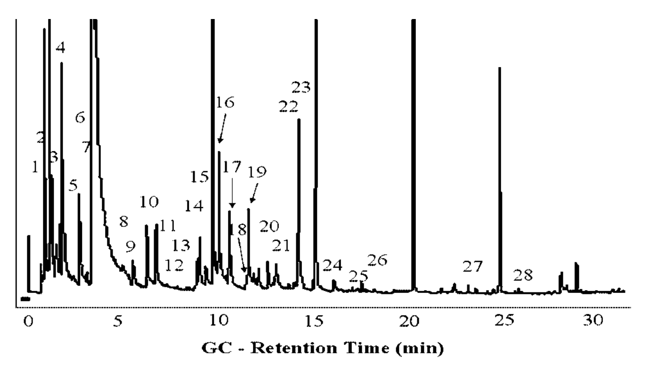

Figure 1—Gas chromatograms of headspace volatile compounds in broken rice stored at 37 °C and 70% RH for 30 d

tration increased over time. Therefore, volatile compounds formed at the beginning of the study probably did not decompose to forms secondary volatile compounds during storage.

The unsaturated fatty acids, oleic and linoleic, represent more than 60% of the total milled rice fatty acids and acts as the precursor for most of the volatile components. The main volatile decomposition products formed from oleic, linoleic, and linolenic acids are those expected from the cleavage of the alkoxyl radicals formed from the hydroperoxides of autoxidized and photosensitized oxidized fatty esters (Frankel 1998). Octanal, heptanal, decanal, nonanal, 2-nonenal, and 2-heptanone are produced by oleate hydroperoxide decomposition; linoleate decomposition produces hexanal, pentanol, pentanal, 2-pentylfuran, and 2-octenal; hexanal and 2-pentenal were produced from linolenate hydroperoxide decomposition (Frankel and others 1981, 1983, 1987; Frankel 1998; Min and Boff 2002; Lee and others 2003). Compounds such as 2, 7 dimethyl octanol, 1, 3, 5 – trimethyl benzene, and 1, 2 bis cyclobutane was probably formed from the interconversion of isomeric hydroperoxide that occurs as a secondary reaction during thermal decomposition under the GC conditions, between hydroperoxides and their decomposition products, or possibly formed during aqueous extraction.

Ten FFA oxidation products (pentanal, pentanol, hexanal, 2-heptanone, heptanal, heptanol, 2-pentylfuran, octanal, 2-octenal, and nonanal) were selected for further studies based on products of major rice FFA, availability, and odor threshold value. Pentanal, pentanol, and hexanal, the major volatile compounds of rice, have a characteristic grassy and fatty flavor (Kochhar 1993). The oleic acid oxidation products octanal and nonanal have a very low odor threshold value (0.7 and 1.0 ppb) (Buttery and others 1999), and their sharp, citrus, and fatty flavor (Kochhar 1993) made them significant for off-flavor development in beer. Heptanol and pentanol had threshold values of 3.0 and 4.0 ppm, respectively (Fazzalari 1978). Linoleic oxidation product 2-pentylfuran has been reported as the major compound responsible for undesirable beany flavor (Chang and other 1983; Min and Boff 2002) has a threshold value of 6.0 ppb (Buttery and others 1999). The oleic acid oxidation products 2-heptanone and 2-octenal have threshold values of 140 and 3.0 ppb, respectively (Buttery and others 1999).

Effect of water washing on head rice selected volatile compounds during storage

The effect of water washing on the concentration of commercially milled head rice volatile compounds during storage is presented in Table 2. Volatile compounds of head rice increased significantly during storage. Pentanal, pentanol, and hexanal were the major volatile compounds comprising about 60% to 70% of the total volatile compounds in head rice. The initial concentration of pentanal, pentanol, and hexanal for head rice was 14.88, 11.22, and 15.02 ppb, respectively; the concentration of these volatile compounds increased to 34.40, 12.57, and 28.26 ppb, respectively, during 30 d of storage at 37 °C and 70% RH. The rates of formation of aldehydes (pentanal, hexanal, heptanal, octanal, and nonanal) and ketone (2 heptanone) during storage were greater than those of pentanol, heptanol, and 2 pentylfuran. Suzuki and others (1999) also reported that the concentrations of hexanal and pentanal in raw brown rice increased during storage at 35 °C for 2, 4 and 6 wk. This was possibly because the secondary oxidation product aldehydes, the pre-

Table 1—Volatile components identified from milled rice aqueous extracts

Peak nr[a]	Volatiles[b]	RT (min)
1	Hexane	1.57
2	3-Methyl pentane	2.11
3	Pentanal	2.23
4	3 Methyl butanal	2.25
5	2-Pentenal	2.78
6	Pentanol	3.12
7	Hexanal	3.78
8	Hexanol	5.90
9	2 Heptanone	6.69
10	2,4 Hexadienal	6.93
11	2,7-Dimethyl, Octanol	7.10
12	Heptanal	7.11
13	1, 3, 5, - Trimethyl, benzene	9.26
14	Bezaldehyde	9.37
15	Heptanol	9.70
16	2-Pentyl-furan	10.38
17	Octanal	10.91
18	1, 2 –Bis, cyclobutane	11.79
19	1,2 Dichlorobenzene[c]	11.90
20	(E)-2, Octenal	12.90
21	2-butyl, octanol	12.98
22	2-ethyl-decanol	14.28
23	Nonanal	14.50
24	2-Nonenal	16.33
25	Dodecane	17.54
26	Decanal	17.84
27	Tetradecane	23.32
28	Butylated hydroxytoluene	26.09

[a] GC peak numbered in Figure 1.
[b] Identified by the combination of SPME-GC-MS and GC retention time of standards compounds.
[c] Internal standard.

Table 2—Effect of water washing on the volatile components of aqueous extracts prepared from head rice stored at 37 °C and 70% relative humidity for 30 d[a,b]

	Unwashed head rice (ppb)			Water washed head rice (ppb)		
Volatiles	0 d	15 d	30 d	0 d	15 d	30 d
Pentanal	14.88 ± 0.08e	21.66 ± 3.22d	34.40 ± 1.77b	12.95 ± 1.77e	16.14 ± 0.70e	21.69 ± 4.91d
Pentanol	11.22 ± 2.37cd	11.01 ± 0.47cd	12.57 ± 2.48c	7.58 ± 1.36def	6.46 ± 0.31f	6.88 ± 1.53ef
Hexanal	15.02 ± 0.99gh	24.02 ± 2.27ef	28.26 ± 1.95cd	12.61 ± 0.60h	15.00 ± 0.18gh	16.87 ± 1.27g
2-Heptanone	1.09 ± 0.09efg	1.47 ± 0.21de	2.03 ± 0.07c	0.77 ± 0.16g	0.99 ± 0.16fg	1.22 ± 0.28efg
Heptanal	1.19 ± 0.23def	1.49 ± 0.42cd	1.94 ± 0.10b	0.86 ± 0.19f	1.09 ± 0.15ef	1.31 ± 0.27de
Heptanol	0.49 ± 0.08b	0.44 ± 0.05b	0.53 ± 0.04b	0.09 ± 0.16d	0.11 ± 0.20d	0.21 ± 0.18cd
2-Pentyl Furan	2.40 ± 0.43fgh	3.14 ± 0.61def	3.56 ± 0.77d	1.73 ± 0.13h	2.23 ± 0.11gh	2.73 ± 0.55efg
Octanal	1.15 ± 0.17d	1.75 ± 0.18c	1.96 ± 0.09c	0.91 ± 0.04d	1.15 ± 0.05d	1.66 ± 0.01c
2-Octenal	NDf	0.18 ± 0.32ef	0.67 ± 0.04cd	NDf	NDf	0.39 ± 0.06de
Nonanal	4.90 ± 1.10de	6.37 ± 0.78c	6.47 ± 0.48c	3.97 ± 0.50e	3.66 ± 0.41e	4.05 ± 0.28e

[a] Values with the same letter in each row are not significantly ($P < 0.05$) different from each other. ND = not detected.
[b] The data presented in Table 2 and 3 were evaluated as single data but were separated for ease of presentation.

Table 3—Effect of water washing on the volatile components of aqueous extracts prepared from broken rice stored at 37 °C and 70% relative humidity for 30 d[a,b]

	Unwashed broken rice (ppb)			Water washed broken rice (ppb)		
Volatiles	**0 d**	**15 d**	**30 d**	**0 d**	**15 d**	**30 d**
Pentanal	27.34 ± 1.99c	54.01 ± 0.96a	54.32 ± 4.56a	20.90 ± 3.53d	23.04 ± 2.74cd	21.83 ± 0.96d
Pentanol	13.35 ± 2.98bc	16.65 ± 3.11ab	17.32 ± 2.55a	10.22 ± 2.70cde	10.46 ± 1.74cde	11.26 ± 2.60cd
Hexanal	31.56 ± 1.54c	45.99 ± 3.94b	58.31 ± 2.66a	21.60 ± 2.71f	23.26 ± 1.87f	27.01 ± 2.79de
2-Heptanone	2.07 ± 0.25c	3.18 ± 0.03b	3.95 ± 0.26a	1.28 ± 0.25ef	1.49 ± 0.46de	1.90 ± 0.49cd
Heptanal	2.11 ± 0.14b	3.31 ± 0.19a	3.64 ± 0.13a	1.32 ± 0.07de	1.45 ± 0.08cde	1.78 ± 0.30bc
Heptanol	1.05 ± 0.07a	0.97 ± 0.20a	1.05 ± 0.12a	0.41 ± 0.09bc	0.49 ± 0.10b	0.51 ± 0.05b
2-Pentyl Furan	5.07 ± 0.53c	8.55 ± 0.21a	6.27 ± 0.20b	2.80 ± 0.26efg	3.47 ± 0.45de	2.53 ± 0.42fg
Octanal	2.61 ± 0.51b	4.27 ± 0.17a	4.24 ± 0.34a	1.61 ± 0.06c	1.94 ± 0.06c	2.40 ± 0.40b
2-Octenal	0.41 ± 0.37de	1.19 ± 0.18b	1.67 ± 0.06a	0.23 ± 0.20ef	0.42 ± 0.13de	0.84 ± 0.01c
Nonanal	6.13 ± 0.88d	8.29 ± 0.35b	10.93 ± 1.72a	4.07 ± 0.20e	4.54 ± 0.34e	8.14 ± 0.48b

[a] Values with the same letter in each row are not significantly ($P < 0.05$) different from each other.
[b] The data presented in Table 2 and 3 were evaluated as single data but were separated for ease of presentation.

cursor for pentanol and heptanol, did not oxidize further to form alcohol during storage. The concentrations of linoleic oxidation products (pentanal and hexanal) were greater than those of oleic acid oxidation products (octanal and nonanal). This was probably because linoleic acid oxidizes 41 times faster than oleic acid (Frankel 1998).

Water washing significantly reduced the concentration of the volatile components for commercially milled head rice. This was probably due to the effect of water washing on the total surface lipid of the milled head rice. Monsoor and Proctor (2002) found that about 60% to 70% of the milled head rice total surface lipids were removed by water washing. The concentrations of pentanal, pentanol, and hexanal of unwashed 0 d stored head rice were 14.88, 11.22, and 15.02 ppb, respectively. The corresponding values for water-washed head rice were 12.95, 7.58, and 12.61 ppb. The similar effect of water washing was observed for all the selected volatile compounds. The greater effect of water washing was observed when higher concentrations of volatile compounds were present on the milled rice surface. The concentrations of pentanal, pentanol, and hexanal were reduced to 21.69, 6.88, and 16.87 ppb, respectively, from the initial 34.40, 12.57, and 28.26 ppb, respectively, for head rice sample stored for 30 d. This was possibly because of the milled rice surface saturation with the volatile compounds formed during storage. The volatile compounds formed on day 0 probably did not saturate the milled rice surface and remain attached to the rice during water washing. On the other hand, the volatile compounds formed in 15-d and 30-d stored rice were significantly greater than those formed in 0-d stored rice and easily removed by water washing.

Effect of water washing on broken rice selected volatile compounds during storage

The effect of water washing on the concentration of commercially milled broken rice volatile compounds during storage is presented in Table 3. The major volatile compounds were pentanal, pentanol, and hexanal, which make up about 60% to 70% of the total volatile compounds of broken rice. Each volatile component is as a greater concentration in unwashed broken rice than unwashed head rice for any storage time (Table 2 and 3). This is supported by Monsoor and Proctor (2003) who reported that broken rice had a significantly higher FFA and conjugated diene (CD) formation rate than head rice. The large quantity of FFA is probably due to the higher surface lipids in broken rice from the residual bran present in broken rice. As we have seen, in head rice, the volatile compounds of broken rice also increased significantly during storage. The concentration of pentanal, pentanol, and hexanal increased to 54.32, 17.32, and 58.31 ppb, respectively, from the initial 27.32, 13.35, and 31.56 ppb, respectively, during 30 d of storage at 37 °C and 70% RH. The similar trend of formation also observed with other volatile compounds during storage. The concentrations of octanal and nonanal were lower than pentanal and hexanal in stored broken rice. The relatively lower amount of octanal and nonanal indicated that the rate of oxidation for oleic acid was slower than for linoleic acid. This is in agreement with the findings of Lam and Proctor (2003b) who found that the primary volatile oxidation products were of linoleic acids.

Water washing significantly reduced the concentration of the volatile components for broken rice samples. The concentration of pentanal, pentanol, and hexanal were reduced to 21.83, 11.26, and 27.01 ppb, respectively, from the initial 54.32, 17.32, and 58.31 ppb, respectively, for broken rice sample stored for 30 d. Water washing is also found to be effective in reducing the volatile compounds of 0 d and 15 d stored broken rice. Monsoor and Proctor (2003) reported that water-washed broken rice had slower FFA and CD formation rate relative to an unwashed control. Broken rice with higher concentrations of volatile compounds were more susceptible to water washing than broken rice with lower concentrations of volatile compounds as we saw with head rice.

Conclusions

The concentration of linoleic oxidation products, pentanal, pentanol, and hexanal were high and increased significantly in both head and broken rice samples, reflecting a higher rate of oxidation of linoleic acid than oleic acid. The concentrations of volatile compounds of broken rice were significantly greater than those obtained with head rice. The higher concentration of volatile components in broken rice is responsible for greater off-flavor development and lower quality of broken rice relative to head rice. Water washing was effective in reducing the concentration of volatile compounds from both head and broken rice. A simple water wash can increase the economic value of broken rice by reducing the end product off-flavor development. Therefore, water washing of broken rice may be used before beer manufacture to improve the end product flavor quality.

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