

## Odor-Active Compounds in Cooked Rice Cultivars from Camargue (France) Analyzed by GC–O and GC–MS

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Volatile compounds of cooked rice from scented (Aychade, Fidji) and nonscented (Ruille) cultivars grown in the Camargue area in France were compared to that of a marketed Asian scented one (Thai) by gas chromatography–olfactometry (GC–O) and gas chromatography–mass spectrometry (GC–MS). GC–O analyses of the organic extracts resulted in the perception of 40 odorous compounds. Only two compounds, oct-1-en-3-one and 2-acetyl-1-pyrroline, were almost always perceived. Hierarchical cluster analysis showed that most of the difference between rice odors was linked to quantitative differences with only 11 compounds being specific to some of the rice. Sixty compounds were identified and quantified by GC–MS, including a few new odor-active components. Principal component analysis enabled us to differentiate scented cultivars from a nonscented one, and scented rice cultivars from Camargue from a Thai sample. Calculated odor-active values evidenced that the Thai sample odor differed from that of scented Camargue cultivars because of the degradation of lipids and of cinnamic acid compounds.

**KEYWORDS:** Camargue area; rice cultivars; cooked rice aroma; organic extract; representativeness; GC–O; detection frequency; GC–MS

### INTRODUCTION

Scented rice is one of the important components of the diet in Asian countries. It develops a strong and specific aroma, and its popularity has been recently increasing in North America and Europe, including France. Efforts have been undertaken for the last decades to promote the production of aromatic cultivars in Camargue, a traditional rice cultivation area in the south of France. New scented cultivars adapted to temperate climate have been developed, and three of them (Aychade, Fidji, and Giano) are being tested in Camargue area (1).

A roasty popcornlike flavor compound, 2-acetyl-1-pyrroline (2AP), was reported for the first time by Buttery et al. (2, 3) as an important contributor to the scented character of cooked rice. This has been confirmed since by several studies (4, 5). However, other volatile compounds, such as aldehydes, shiki-

mate derivatives, and sulfur- and nitrogen-based compounds, were also reported as probable contributors to the overall cooked aroma of scented rice (6, 7).

Cooked rice aroma was evaluated through different extraction procedures such as dynamic headspace (7), steam distillation continuous extraction (8–11), solvent extraction (12, 13), and solid-phase microextraction (14, 15).

The aim of the present study was to evaluate the flavor compounds of cooked rice from two scented rice cultivars from the Camargue area (Aychade and Fidji) and to compare them with those of a well-known Asian scented rice cultivar (Thai). Moreover, a nonaromatic cultivar from the Camargue area (Ruille) was also analyzed for comparison with the aromatic cultivars. Gas chromatography–olfactometry (GC–O) technique was used to study the aroma profile of the samples since it allows selecting odor-active compounds in a complex mixture (16, 17). It has been associated with gas chromatography–mass spectrometry (GC–MS) for quantitative assessment of the odor-active compounds. Before GC–O and GC–MS analyses, a study was undertaken to obtain representative aromatic extracts as similar as possible to the aroma of the original product (18, 19).

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## MATERIALS AND METHODS

**Chemicals.** All aroma compounds labeled as standard in tables were purchased from Sigma-Aldrich Chimie and were of GC purity. Synthetic 2AP was a kind gift from Dr. Yoshihashi (Japan International Research Center for Agricultural Sciences, Ibaraki, Japan).

**Rice Samples.** Three scented cultivars (Aychade, Fidji, and Giano) and a common nonscented cultivar (Ruille) were harvested in 2006 in the Camargue area (Centre Francais du Riz, CFR, Arles, France). One scented commercial rice sample (Thai, Taureau Ailé, Lyon, France) was purchased as milled grain in a French supermarket. Paddy grains from Camargue were sun-dried, dehulled, and milled in the laboratory to remove ca. 12% bran. All samples were stored in an air-conditioned room (20 °C, 70% relative humidity) until analysis.

**Extraction of Flavor Compounds.** The method is based on Jezussek et al. (6) with some modifications. Rice (5 g) and mineral water (Volvic, 10 mL) were introduced in a glass beaker. The sample was cooked in open steam for 20 min. After resting for 10 min, the freshly cooked rice was frozen under liquid nitrogen and 30 g of anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) was added. This was kept in the freezer overnight, then ground to flour in a basic analytical crusher (IKA-WERKE) under liquid nitrogen. The resulting powder was suspended in 60 mL of different organic solvent mixtures stated below, spiked with 2,4,6-trimethylpyridine (collidine, 1  $\mu\text{g}$ ) as internal standard, and magnetically stirred at room temperature for 1 h under nitrogen. The organic phase was recovered, and the powder was re-extracted with 40 mL of organic solvent. The organic extracts were pooled, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated to ca. 5 mL through a Vigreux column and finally to ca. 0.5 mL through a Kuderna-Danish concentrator fitted with a Snyder column (Supelco).

**Representativeness of the Organic Extracts.** Four solvents were tested: dichloromethane, dichloromethane/pentane (1:2, v/v), ether, and ether/pentane (1:1, v/v). Cooked scented rice (Giano) was subjected to the aroma extraction as above, but without collidine addition. Resulting organic extracts were concentrated to ca. 1 mL. This procedure was performed four times, and the extracts were pooled. The pooled extract was then distributed in 20 2-mL flasks (0.2 mL in each). The solvent was removed under nitrogen flux, and the flasks were hermetically closed. The reference consisted of freshly cooked rice obtained as above (Giano) that was presented in hot glasses (ca. 20 g in each). Sixteen panelists from the laboratory (five females and 11 males) were asked to evaluate the closeness between the odor of the organic extracts and that of the cooked rice (18, 20). They were asked to smell the odor of the reference first and memorize it. They were informed to heat the flasks containing extracts by keeping them in their hands for about 10 s before opening them to evaporate residual solvent before smelling. They were asked to place a mark for each of the four extracts (randomly numbered) on an unstructured 10-cm scale, anchored with "different from the reference" on the left and "similar to the reference" on the right. The distances between the left anchor of the scale and the position of the marks were measured. This test was performed in duplicate.

**GC–O Analysis.** The extracts from dichloromethane/pentane mixture (1:2, v/v) were analyzed by GC–O on a chromatograph model 6890 (Hewlett-Packard), equipped with a flame ionization detector (FID, 250 °C) and a sniffing port and mounted with fused silica capillary column DB-Wax (J&W Scientific, i.d. 0.32 mm, 30 m, film thickness = 0.5  $\mu\text{m}$ ). Operating conditions were as follows: volume injected, 2  $\mu\text{L}$ ; splitless injection (30 s); injection temperature, 240 °C; temperature program from 40 to 240 °C at 6 °C  $\text{min}^{-1}$  and then held constant for 10 min. Helium was used as carrier gas in constant flow mode (1.5 mL  $\text{min}^{-1}$ ) with a linear velocity of 44  $\text{cm s}^{-1}$ . The GC effluent was split 1:1 between the FID and the sniffing port (240 °C). Humidified air was added in the sniffing port at 100 mL  $\text{min}^{-1}$ .

**Odor Detection Frequency.** GC–O frequency analysis was performed following the methodology described by Charles et al. (21) with some modifications. Panelists (five women and three men) from the laboratory UMR Flavic, experienced with odor recognition and with the sniffing of GC–O effluents, performed the sniffing of the extracts in duplicate and one session per day. The duration of the sniffing was 40 min. For each odor stimulus, panelists recorded the detection time and gave a verbal description of each perceived odor. They quoted "unknown descriptor" when the odor was not recognized. The detection

**Table 1.** Similarity of the Odors of the Four Organic Extracts to Cooked Rice Reference

solvent test	similarity scaling, $\text{cm}^a$
ether	2.3 A
ether/pentane	3.1 A
dichloromethane/pentane	4.9 B
dichloromethane	5.0 B

<sup>a</sup> On a scale of 10 cm. Values followed by the same letter are not significantly different (Newman–Keuls test,  $p < 0.05$ ).

frequency for an odor having the same retention time and a similar description was calculated (sum of the number of odor detections at this retention time: maximum = 16). Homemade software COCONUT (22) was used for data acquisition.

**GC–MS Analysis.** Injection of 2  $\mu\text{L}$  of the dichloromethane/pentane extract was performed on a chromatograph model 6890 HP. Operating conditions were the same as those mentioned above. Mass spectrometry was performed on a mass selective detector model 5973 (Agilent Technologies) operated in the electron ionization mode (70 eV). The mass spectrometer scanned mass from  $m/z$  29 to 350. Ion source was set at 230 °C and transfer line at 260 °C.

**Identification of the Volatile Compounds.** The identification of the volatile compounds was carried out by comparing with mass spectra of standards (when available), by using the mass spectra libraries (Wiley, NIST, INRA database), and by comparing the retention indexes with those of available standard compounds and those of the literature. Linear retention indexes (RI) were calculated using a series of alkanes ( $\text{C}_{10}$ – $\text{C}_{30}$ ) injected daily in the same chromatographic conditions.

**Quantitative Measurements.** The extractions of aroma compounds from cooked rice were performed in triplicate. The total ion chromatogram (TIC) area from GC–MS analysis served to quantify volatiles using collidine as internal standard and the concentration calculated as collidine equivalent. The concentrations of coeluted compounds were calculated after estimating the total area of the target compound by dividing the area of a representative ion over its relative abundance in TIC from the library. The selected ions were 82, 105, 81, 73, 92, 88, and 60 for hexanal, acetophenone, (*E,E*)-nona-2,4-dienal, pentanoic acid, 2-phenylethanol, 2-ethylhexanoic acid, and heptanoic acid, respectively.

**Statistical Analysis.** Hierarchical cluster analysis (HCA) was performed with XL-STAT Prov.7 (Addinsoft) using the Ward aggregation method and squared Euclidean distance. Analysis of variance, mean comparison tests (Newman–Keuls), and principal component analysis (PCA) were performed using Statistica v. 7.1 (StatSoft).

## RESULTS

**Representativeness of the Organic Extracts.** A scented rice cultivar from the Camargue area (Giano) was used for choosing the organic solvent giving an aroma extract resembling that of the original product. Panelists (16 people) were asked to score the similarity between the odor of the four organic extracts and the odor of the freshly cooked rice. Analysis of variance ( $p < 0.05$ ) of the distance marked by each panelist to the reference did not evidence any panelist effect. The solvent effect was, however, significant, and dichloromethane and dichloromethane/pentane (1:2, v/v) gave extracts with the higher similarity values with the reference. These extracts were thus considered the most representative ones (Table 1).

It should be noted that the highest value did not exceed half of the scale. Dichloromethane/pentane (1:2, v/v) mixture was chosen for further work because it needs a lower temperature for concentrating, compared to dichloromethane, thus limiting the potential loss of volatile compounds.

**GC–O Results.** Dichloromethane/pentane extracts of the four rice samples (Aychade, Fidji, Ruille, and Thai) were analyzed in duplicate by GC–O with eight trained panelists. Linear retention index values, compound associated, odor description, and frequency of detection are presented in Table 2. Forty

**Table 2.** Odor-Active Compounds of Four Cooked Rice Cultivars Detected in GC–O Analysis

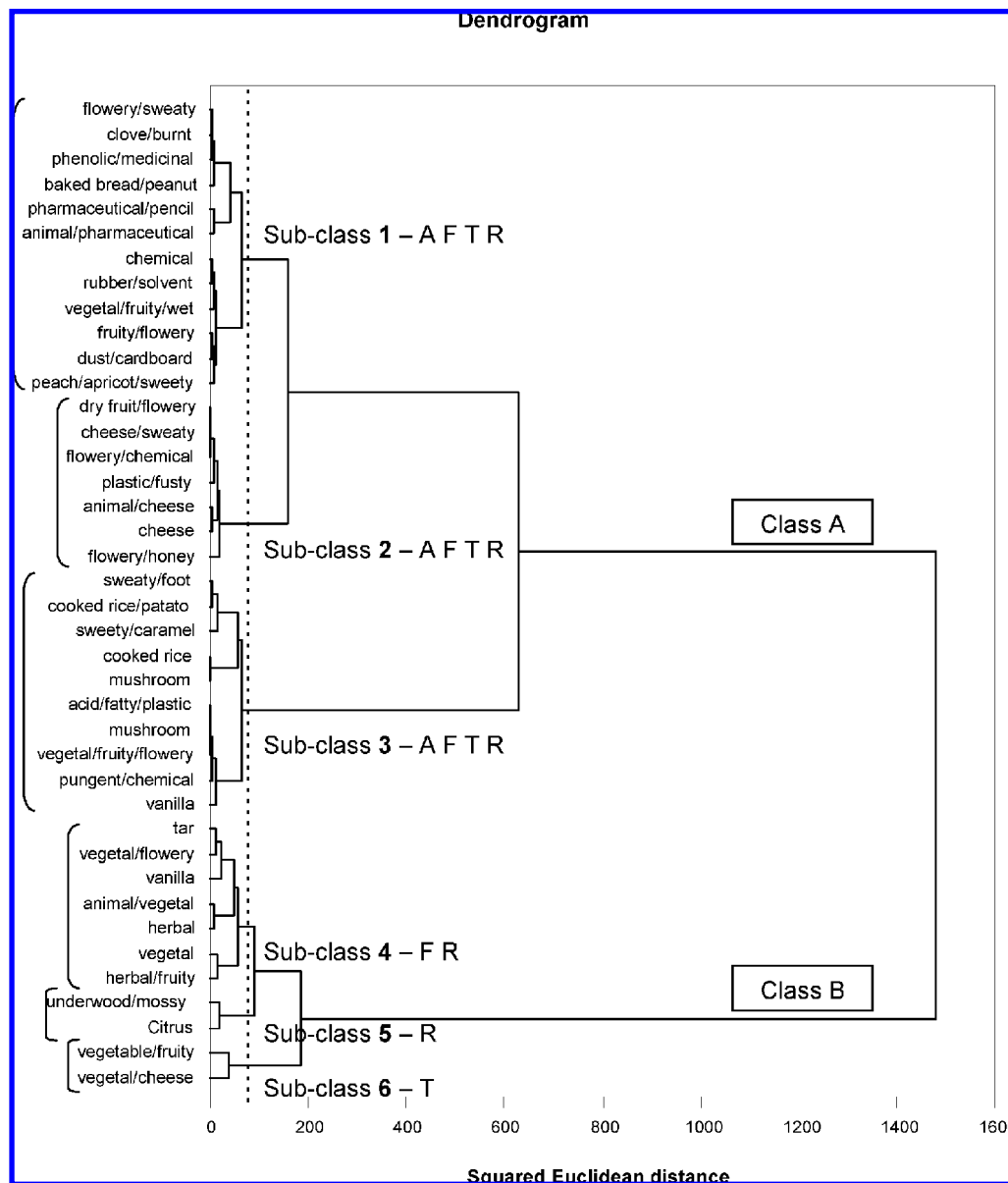
RI <sup>a</sup>	odor description <sup>b</sup>	compounds <sup>c</sup>	frequency of detection <sup>d</sup>			
			Aychade	Fidji	Thai	Ruille
1106	herbal	hexanal	0	8	5	6
1200	herbal/fruity	2-/3-methylbutan-1-ol	0	8	0	5
1292	citrus-like	octanal	0	0	0	5
1302	mushroom	oct-1-en-3-one	15	16	16	15
1334	cooked rice	2-acetyl-1-pyrroline	16	16	16	15
1370	vegetal	hexan-1-ol	0	6	0	0
1404	mushroom	oct-3-en-2-one	11	15	11	13
1446	cooked rice/potato	oct-2-enal/methional	15	15	13	9
1501	vegetal/fruity/flowery	decanal	12	14	12	14
1531	dust/cardboard	octa-3,5-dien-2-one/non-2-enal	5	5	9	8
1608	vegetal/flowery	longifolene	5	9	0	6
1625	vegetal/cheese	propanoic acid	0	0	5	0
1633	cheese	<u>2-methylpropanoic acid</u>	10	13	9	11
1676	sweaty/foot	butanoic acid	14	14	15	11
1693	baked bread/peanut	( <i>E,E</i> )-nona-2,4-dienal	5	10	8	10
1716	flowery/honey	phenylacetaldehyde	11	10	6	13
1747	cheese/sweaty	2-methylbutanoic acid	10	10	10	10
1784	dry fruit/flowery	( <i>E,Z</i> )-deca-2,4-dienal	10	9	9	10
1801	acid/fatty/plastic	pentanoic acid	12	14	11	13
1847	animal/pharmaceutical	hexanoic acid/2-methoxyphenol	7	10	12	9
1890	fruity/flowery	2-phenylethanol	5	5	7	8
1944	rubber/solvent	benzothiazole	7	6	6	7
1992	pungent/chemical	phenol/heptanoic acid	13	14	10	14
2011	vegetal/fruity/wet	unknown	5	8	7	7
2023	sweet/caramel	$\gamma$ -nonalactone	11	14	14	7
2072	animal/vegetal	unknown	0	9	5	10
2132	peach/apricot/sweaty	$\gamma$ -decalactone	7	5	10	7
2170	clove/burnt	2-methoxy-4-vinylphenol	7	10	6	9
2182	flowery/sweaty	$\delta$ -decalactone	8	8	6	8
2200	flowery/chemical	unknown	11	10	10	11
2222	animal/cheese	nonanoic acid	8	12	9	10
2247	vegetable/fruity	unknown	0	5	12	0
2272	plastic/fusty	unknown	11	10	12	12
2301	pharmaceutical/pencil	2,4-bis(1,1-dimethylethyl)phenol	6	11	12	5
2379	phenolic/medicinal	4-vinylphenol	7	11	7	7
2394	underwood/moss	unknown	0	0	0	11
2426	tar	indole	5	5	0	9
2474	chemical	3-methylindole	6	5	7	6
2501	vanilla	unknown	0	6	0	10
2543	vanilla	vanillin	10	14	13	16

<sup>a</sup> Linear retention index calculated on a DB-Wax column with a series of alkanes C<sub>10</sub>–C<sub>30</sub>. <sup>b</sup> Odor description by panelist during olfactometry. <sup>c</sup> The underlined compounds are those identified for the first time in rice GC–O analyses. <sup>d</sup> Detection frequency (eight panelists); the data are from duplicate analyses.

odorous compounds were perceived, of which seven could not be identified. Twenty-nine odors were found in all four rice extracts. Fifteen compounds were perceived more than 40 times out of 64 (cumulative frequency of detection for the four rice cultivars); five were aliphatic acids, three aldehydes, three ketones, two unknown, one phenol, and one nitrogen compound. Some acids, such as butanoic acid and 2-methylbutanoic acid, were responsible for animal (cheese, sweaty) unpleasant notes, while aldehydes such as hexanal and decanal were associated with pleasant descriptors such as herbal, fruity, and flowery. Only two odors were detected almost every time (oct-1-en-3-one, mushroom attribute; and 2-acetyl-1-pyrroline, cooked rice attribute). All the panelists detected 2AP and described it as cooked rice odor. This confirms the importance of 2AP in scented cooked rice aroma (2, 6). Surprisingly, this compound was also perceived in the nonscented cultivar, Ruille, with a high frequency of detection (15/16). Similarly, Jezussek et al. (6) reported 2AP as the odor-active compound of (nonscented) “indica” rice, but with a low flavor dilution (FD) factor. They also reported for the first time three compounds showing the highest FD factor (bis(2-methyl-3-furyl)-disulfide, 2-amino acetophenone, and 4,5-epoxy-(*E*)-dec-2-enal) for four rice samples. They were not detected in our samples. However, we detected for the first time in cooked rice four compounds (oct-

3-en-2-one, 2-methylpropanoic acid,  $\gamma$ -decalactone, and  $\delta$ -decalactone) that have a high frequency of detection. Other compounds such as oct-3-en-2-one (mushroom note), decanal (vegetal/fruity/flowery notes), butanoic acid (sweaty/foot note), and pentanoic acid (acid/fatty/plastic notes) were detected with a high frequency (70%). Therefore, they may be possible contributors to overall cooked rice flavor. The unknown compound, with a linear retention index value of 2394 was detected only in Ruille, with a frequency of detection of 11. Octanal (citruslike aroma) was also only perceived in this cultivar but with a lower frequency (5/16). Furthermore, hexan-1-ol (vegetal) was detected only for Fidji and propanoic acid (vegetal/cheese) only for Thai.

To assess a typology of odors perceived in scented and nonscented cooked rice, an HCA was performed. The resulting dendrogram revealed two main clusters (A and B) and six subclusters for a squared Euclidean distance of 64 (**Figure 1**). The first cluster (A) includes odors detected at a similar frequency of detection for the four rice cultivars; the frequency of detection of odors increased from subcluster 1 to 3. The second cluster (B) concerns odors detected with the frequency of detection differing between cultivars. It comprised three subclusters. Subcluster 4 distinguishes Fidji and Ruille cultivars thanks to descriptors such as vegetal/flowery, vanilla, animal/



**Figure 1.** HCA dendrogram of aroma attributes obtained from the four cooked rice cultivars. A: Aychade, F: Fidji, T: Thai, R: Ruille.

vegetal, and herbal/fruity. Underwood/mossy and citruslike notes in subcluster 5 were able to differentiate the Ruille cultivar from the others. Moreover, subcluster 6 with the descriptors vegetable/fruity and vegetal/cheese enabled us to differentiate the Thai cultivar.

**Volatile Compound Levels.** The level of volatile components recovered in the organic extracts of the four cooked rice cultivars subjected to GC–O was determined by GC–MS (Table 3). Sixty compounds were quantified, including 13 aldehydes, 10 alcohols, 10 acids, 7 ketones, 6 phenols, 5 nitrogen compounds, 5 aromatic hydrocarbons, 3 lactones, and 1 terpene.

As expected, the level of 2AP was much higher in the scented cultivars than in the nonscented one; only traces of 2AP could be detected in the latter. The highest level of 2AP was measured for Fidji, followed by Aychade and Thai. In parallel, the level of some other volatiles was significantly higher for scented cultivars as, for example, for oct-1-en-3-ol, octan-1-ol, hexan-1-ol, butanoic acid, and hexanoic acid.

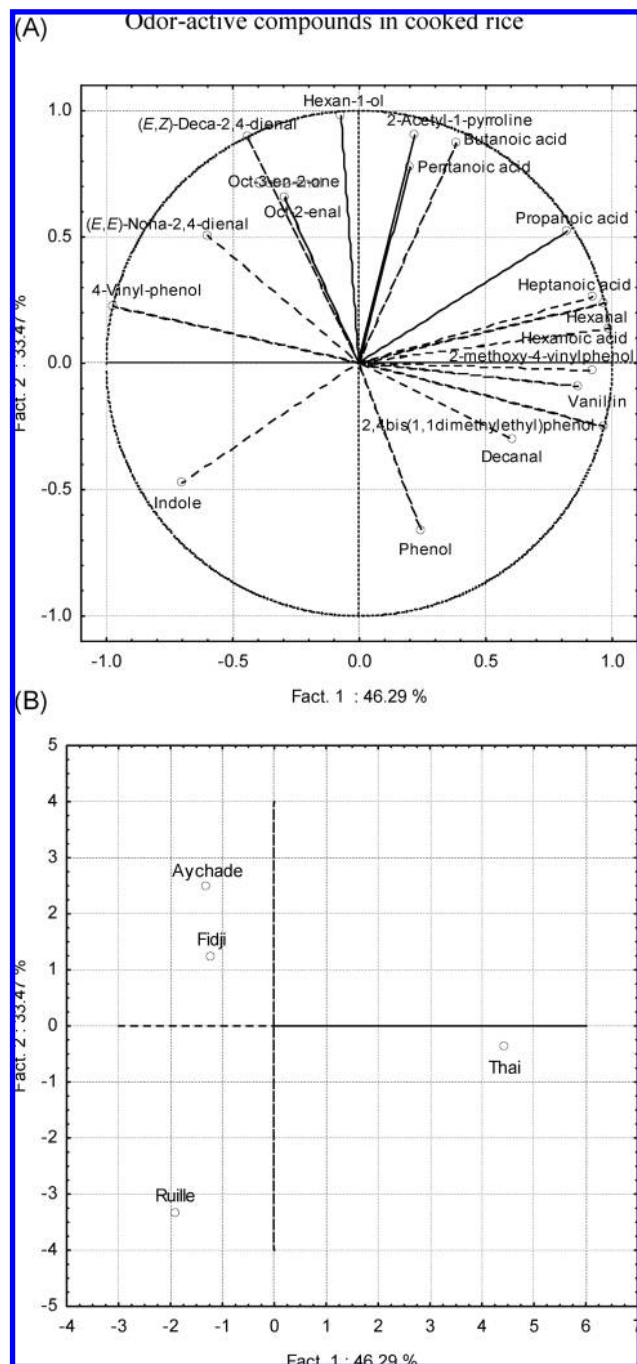
GC–MS results take into account all the volatiles present in the extracts. It was therefore more relevant to focus only on odor-active compounds. A PCA was thus performed on a short

compound list restricted to compounds detected by GC–O and quantified by GC–MS for at least one cultivar. Nineteen odor-active compounds were selected. Eleven compounds cited in GC–O and identified were at trace level by GC–MS, and six unknowns could not be quantified. It should be noticed that some of the mostly cited compounds, such as oct-1-en-3-one or  $\gamma$ -nonalactone, were at trace level. The first two principal components (PCs) explained 46.29 and 33.47% of the total variance, respectively. Figure 2 shows a bidimensional plot of variable loadings and cultivar scores on PC1 and PC2. Thai and Ruille were clearly distinct and separated from Aychade and Fidji. The score on PC1 discriminated Thai from the Camargue cultivars and PC2 scented from nonscented cultivars. The aroma compounds with high positive loadings on PC1 were mainly aldehydes and acids, opposite 4-vinylphenol. Thai was indeed richer in hexanal and hexanoic acid, in particular. Positive loadings of PC2 were observed in particular for hexan-1-ol, 2-acetyl-1-pyrroline, and (*E,Z*)-deca-2,4-dienal and negative ones for phenol and indole. Ruille was clearly separated from scented cultivars by PC2 because of its higher level in indole and phenol and its lower level in some compounds such as

**Table 3.** Quantification of Volatile Compounds of Organic Aroma Extracts from Four Cooked Rice Cultivars

compounds	RI exp <sup>b</sup>	RI ref <sup>c</sup>	identification <sup>d</sup>	quantification <sup>e</sup> (μg/kg)				
				Aychade	Fidji	Thai	Ruille	RSD <sup>f</sup>
hexanal <sup>a,r</sup>	1074	1084 <sup>g</sup>	MS, RI, O, Std	70 A	61 A	122 B	42 A	14
ethylbenzene	1132	1125 <sup>g</sup>	MS, RI	340	325	403	292	42
1,4-dimethylbenzene	1141	1127 <sup>g</sup>	MS, RI	391 A	416 A	495 B	375 A	27
1,3-dimethylbenzene	1146	1150 <sup>g</sup>	MS, RI	900 A	946 A/B	1314 C	995 B	34
butan-1-ol	1147	1145 <sup>g</sup>	MS, RI, Std	39	45	116	74	33
pent-3-en-2-ol	1174		MS, Std	76 A	70 A	115 B	76 A	8
1,2-dimethylbenzene	1192	1183 <sup>g</sup>	MS, RI	622 A	680 A	868 B	420 C	32
2-/3-methylbutan-1-ol <sup>f</sup>	1214	1208/1205 <sup>g</sup>	MS, RI, O, Std	trace	trace	trace	trace	
pentan-1-ol	1259	1255 <sup>g</sup>	MS, RI, Std	107 A	trace C	343 B	7 C	23
octanal <sup>f</sup>	1297	1280 <sup>g</sup>	MS, RI, O, Std	50	40	44	40	6
oct-1-en-3-one <sup>f</sup>	1308	1305 <sup>f</sup>	MS, RI, O, Std	trace	trace	trace	trace	
butylbenzene	1320	1308 <sup>f</sup>	MS, RI	27 B	49 A	64 A	73 A	10
hept-2-enal (isomer)	1333	1320 <sup>g</sup>	MS, RI, Std	275 B	176 C	58 A	64 A	19
6-methylhept-5-en-2-one	1346	1340 <sup>g</sup>	MS, RI, Std	26 A	59 B	29 A	39 A	9
2-acetyl-1-pyrroline <sup>f</sup>	1348	1320 <sup>g</sup>	MS, RI, O, Std	215 A/B	264 B	186 A	trace C	28
hexan-1-ol <sup>f</sup>	1361	1360 <sup>g</sup>	MS, RI, O, Std	59 B	43 A	35 A	19 C	5
4-hydroxy-4-methyl-pentan-2-one	1372	1343 <sup>h</sup>	MS, RI	430 B	601 C	75 A	86 A	27
nonan-2-one	1397	1388 <sup>g</sup>	MS, RI, Std	trace	trace	trace	trace	
nonanal	1402	1385 <sup>g</sup>	MS, RI, Std	167 B/C	152 A/B	188 C	134 A	13
2-butoxyethanol	1412		MS, RI	77 A	154 B	68 A	96 A	22
oct-3-en-2-one <sup>f</sup>	1418	1388 <sup>g</sup>	MS, RI, O, Std	60 B	trace A	trace A	trace A	12
oct-2-enal <sup>f</sup>	1439	1408 <sup>g</sup>	MS, RI, Std	48 B	trace A	trace A	trace A	4
methional <sup>g,r</sup>	1440	1458 <sup>g</sup>	RI, O, Std	trace	trace	trace	trace	
N,N-diethylformamide	1444		MS	8	13	15	12	2
oct-1-en-3-ol	1458	1465 <sup>i</sup>	MS, RI, Std	129 A	91 B	103 B	22 C	14
2-ethylhexan-1-ol	1498	1492 <sup>g</sup>	MS, RI, Std	25 A	68 B	32 A	81 B	16
ethanoic acid	1499	1477 <sup>j</sup>	MS, RI, Std	299 A	338 A	193 B	92 C	49
decanal <sup>f</sup>	1506	1510 <sup>j</sup>	MS, RI, O, Std	92 A/B	61 A	107 B	92 A/B	16
octa-3,5-dien-2-one <sup>f</sup>	1530	1521 <sup>g</sup>	MS, RI, O	trace	trace	trace	trace	
non-2-enal <sup>f</sup>	1533	1510 <sup>g</sup>	MS, RI, O, Std	trace	trace	trace	trace	
benzaldehyde	1536	1520 <sup>j</sup>	MS, RI, Std	47	38	99	80	12
octan-1-ol	1558	1561 <sup>g</sup>	MS, RI, Std	50 B	38 A	46 A/B	15 C	5
longifolene <sup>h,r</sup>	1588	1577 <sup>h</sup>	MS, RI, O	trace	120	92	trace	44
propanoic acid <sup>f</sup>	1595		MS, RI, O, Std	289 A	198 B	382 C	103 D	23
2-methylpropanoic acid <sup>f</sup>	1632		MS, O, Std	trace	trace	trace	trace	
acetophenone <sup>a,i</sup>	1666	1645 <sup>g</sup>	MS, RI, Std	2 A	4 B	3 B	3 B	1
butanoic acid <sup>f</sup>	1689	1650 <sup>j</sup>	MS, RI, O, Std	330 A	218 A	276 A	105 B	44
(E,E)-nona-2,4-dienal <sup>a,r</sup>	1696	1705 <sup>j</sup>	MS, O	7 A	3 B	2 C	3 B	1
phenylacetaldehyde <sup>f</sup>	1709		RI, O, Std	trace	trace	trace	trace	
2-methylbutanoic acid <sup>f</sup>	1740		MS, RI, O, Std	trace	trace	trace	trace	
(E,Z)-deca-2,4-dienal <sup>f</sup>	1776	1770 <sup>h</sup>	MS, RI, O, Std	163 A	117 B	25 C	18 C	15
pentanoic acid <sup>a,r</sup>	1798		MS, RI, O, Std	27 A/B	35 A	28 A/B	18 B	5
(E,E)-deca-2,4-dienal	1822	1820 <sup>j</sup>	MS, RI, Std	252 B	130 A	35 A	22 A	18
hexanoic acid <sup>f</sup>	1898	1872 <sup>j</sup>	MS, RI, O, Std	568 A	638 A	1104 B	443 C	29
2-methoxyphenol <sup>f</sup>	1912	1883 <sup>j</sup>	RI, O	trace	trace	trace	trace	
2-phenylethanol <sup>a,r</sup>	1926	1931 <sup>j</sup>	MS, RI, O, Std	3	3	5	3	1
benzothiazole <sup>f</sup>	1970	1948 <sup>j</sup>	MS, RI, O	55	46	15	35	16
2-ethylhexanoic acid <sup>a</sup>	1999		MS, RI	12 A	15 A	14 A	trace B	1
phenol <sup>f</sup>	2017	2008 <sup>j</sup>	MS, RI, O, Std	75 A	39 B	89 A	100 A	11
heptanoic acid <sup>a,r</sup>	2017		MS, O, Std	12 A	14 A	19 B	10 A	2
γ-nonalactone <sup>j</sup>	2052	2042 <sup>g</sup>	MS, RI, O	trace	trace	trace	trace	
γ-decalactone <sup>a,r</sup>	2161	2136 <sup>j</sup>	MS, RI, O	trace	trace	trace	trace	
2-methoxy-4-vinylphenol <sup>l,r</sup>	2210	2198 <sup>g</sup>	MS, RI, O	373 B	254 A	563 C	317 A/B	42
δ-decalactone <sup>m,r</sup>	2210	2208 <sup>g</sup>	MS, RI, O, Std	trace	trace	trace	trace	
nonanoic acid <sup>f</sup>	2245		MS, RI, O, Std	trace	trace	trace	trace	
2,4-bis(1,1-dimethylethyl)phenol <sup>f</sup>	2313	2283 <sup>g</sup>	MS, RI, O	1069 A	1114 A	1671 B	1174 A	39
4-vinylphenol <sup>n,r</sup>	2405	2427 <sup>g</sup>	MS, RI, O	1127 A	1079 A	trace B	962 C	31
indole <sup>o,r</sup>	2463	2450 <sup>g</sup>	MS, RI, O	252 B	108 A	69 A	365 C	44
3-methylindole <sup>p,r</sup>	2474		RI, O	trace	trace	trace	trace	
vanillin <sup>q,r</sup>	2586	2569 <sup>g</sup>	MS, RI, O, Std	260 A	139 B	399 C	222 A	24

<sup>a</sup> The concentrations were calculated through a representative ion (see Materials and Methods). <sup>b</sup> Linear retention index calculated on a DB-Wax column with a series of alkanes C<sub>10</sub>–C<sub>30</sub>. <sup>c</sup> Linear retention index: <sup>g</sup> from the FlavorNet database (<http://www.flavornet.org>, accessed June 2007), Acree, 2004 (on C20 M stationary phase); in the literature: <sup>h</sup> Kondjayan and Berdagué (37); <sup>i</sup> Pozo-Bayon et al. (17); <sup>j</sup> El-Sayed (38). <sup>d</sup> Identification proposal is indicated by the following: MS, identification by comparing EI mass spectrum with Wiley and NIST mass spectral database; RI, identification by retention indexes with literature data; O, identification with odor description; Std, mass spectrum agreed with standards injected under the same conditions. When identification was not performed with a standard compound, it was considered a tentative identification. <sup>e</sup> Semi-quantification (fresh weight of raw rice) using 2,4,6-trimethylpyridine (RI = 1384) as internal standard. The data correspond to the mean of triplicates; values followed by the same letter are not significantly different (Newman–Keuls test, *p* < 0.05). The same characters mean that there is no difference among the samples; trace < 2 μg/kg. <sup>f</sup> Residual standard deviation. <sup>g</sup> IUPAC name: 3-methylsulfanylpropanal. <sup>h</sup> (1*R*,2*S*,7*S*,9*S*)-3,3,7-Trimethyl-8-methylenetricyclo-[5.4.0.0<sup>2,9</sup>]undecane. <sup>i</sup> 1-Phenyl-ethan-1-one. <sup>j</sup> 5-Pentylloxolan-2-one. <sup>k</sup> 5-Hexylloxolan-2-one. <sup>l</sup> 4-Ethenyl-2-methoxyphenol. <sup>m</sup> 6-Pentylloxan-2-one. <sup>n</sup> 4-Ethenylphenol. <sup>o</sup> 1*H*-Indole. <sup>p</sup> 3-Methyl-1*H*-indole. <sup>q</sup> 4-Hydroxy-3-methoxybenzaldehyde. <sup>r</sup> Aroma compounds detected in GC–O.



**Figure 2.** Biplot of variable scores (A) and rice loadings (B) on the first two axes of the PCA.

hexan-1-ol, 2AP, and butanoic acid. Aychade and Fidji had high positive PC2 loadings because of their high concentrations, among others, of 2AP and (*E,Z*)-deca-2,4-dienal.

## DISCUSSION

Before GC–O and GC–MS analyses, the representativeness of the organic extracts from cooked rice was tested. The extraction with dichloromethane and the dichloromethane/pentane mixture gave the highest similarity score: 5 on a 10-cm unstructured scale. This result was acceptable when compared to those reported for other foods (23–25). Furthermore, these solvents have already been used to prepare representative aroma extracts from various food matrixes and for the extraction of rice flavors (6, 7).

Among the several GC–O methodologies, the frequency of detection method was used because of its simplicity and most importantly because of its suitability for statistical analyses. HCA showed that most of the differences between rice odors were linked to quantitative frequency of citation rather than to specific compounds. Only 11 compounds (among 40) were specific to some of the rice samples. Three of them were unknown.

PCA treatment, performed on compounds detected by GC–O and quantified by GC–MS, enabled us to clearly separate scented cultivars from a nonscented one on PC2 and Camargue scented cultivars from the Asian one (Thai) on PC1. 2AP content is indeed one of the most tightly positively variables correlated with PC2 (i.e., discriminating between scented and nonscented cultivars). As already noticed (26), the nonscented cultivar was not free from 2AP but was only present at trace levels. Some other components also contribute to this PC, particularly hexan-1-ol content, which was higher for scented rice samples, in agreement with Petrov et al. (5). Widjaja et al. (26) found, however, a hexan-1-ol content of one nonscented cultivar intermediate to those of four scented cultivars. At the opposite side of PC2, indole and phenol appear to be characteristic of the nonscented cultivar. This disagreed with previous results: Yajima et al. (27) found a higher content of indole in a scented cultivar, and Widjaja et al. (26) did not show any clear discrimination between scented and nonscented cultivars for this component. None of the component content correlated with PC1 has been published hitherto for discriminating between scented cultivars (5, 26). Thai (Asian scented sample) appears richer in some aliphatic aldehydes and acids. Most of them may be attributed to lipid degradation (28). The higher level for Thai may be linked to longer storage duration for the market sample from Asia. Camargue samples were indeed analyzed six months after harvest, whereas the market sample may have been stored longer. Vanillin and 2-methoxy-4-vinylphenol contents were also higher for Thai. It is, however, important to note that these compounds, together with fatty acids, some aldehydes, and other shikimate derivatives such as 4-vinylphenol, detected in cooked rice are almost absent in raw rice (29). They are probably formed by an enzyme or even more probably by oxidative mechanisms and elevated temperature during rice storage and processing. Vanillin as a flavor component of cooked rice has been previously reported only in one article (6). Its formation from degradation of ferulic acid has been hypothesized (30). Furthermore, it is now well-established that thermal decarboxylation of ferulic and coumaric acids gives rise to 2-methoxy-4-vinylphenol and 4-vinylphenol, respectively. This phenomenon could occur in our analysis conditions because of the high temperature of the injection port. These volatile phenols were, however, already detected in cooked rice by using a cold on-column injection mode (6). It may thus be postulated that the differences in vanillin, 2-methoxy-4-vinylphenol, and 4-vinylphenol levels between Camargue scented cultivars and the Thai sample could arise from the different amounts in the hydroxycinnamic acids. Indeed, the concentration and the composition of hydroxycinnamic acids may depend on the cultivar (29, 31). Consequently, the relative ratio of these compounds may hence have an indirect influence on the global aroma of rice.

Separated results of GC–O for one side and of GC–MS for the other side do not allow easy recognition of the real impact of flavor compounds in cooked rice. In GC–O analysis, the compounds are separated from their matrix and moreover the odor threshold of volatiles is usually lower in vapor phase than

**Table 4.** OAVs<sup>a</sup> of Volatiles in the Four Cooked Rice Cultivars

compounds	odor threshold ( $\mu\text{g/L}$ ) <sup>b</sup>	OAV <sup>c</sup>			
		Aychade	Fidji	Thai	Ruille
2-acetyl-1-pyrroline	0.1 <sup>d</sup>	2150.00	2640.00	1860.00	nd
( <i>E,Z</i> )-deca-2,4-dienal	0.07 <sup>d</sup>	2328.57	1671.43	357.14	257.14
2-methoxy-4-vinylphenol	3 <sup>d</sup>	124.33	84.67	187.67	105.67
4-vinylphenol	10 <sup>d</sup>	112.70	107.90	nd	96.20
( <i>E,E</i> )-nona-2,4-dienal	0.09 <sup>d</sup>	74.43	33.21	19.23	38.52
decanal	2 <sup>d</sup>	46.00	30.50	53.50	46.00
hexanal	5 <sup>d</sup>	13.94	12.27	24.39	8.47
vanillin	20 <sup>e</sup>	13.00	6.95	19.95	11.10
oct-2-enal	3 <sup>d</sup>	16.00	nd	nd	nd
indole	140 <sup>d</sup>	1.80	0.77	0.49	2.61
butanoic acid	240 <sup>e</sup>	1.38	0.91	1.15	0.44
hexanoic acid	3000 <sup>d</sup>	0.19	0.21	0.37	0.15
hexan-1-ol	2500 <sup>d</sup>	0.02	0.02	0.01	0.01
propanoic acid	20000 <sup>e</sup>	0.01	0.01	0.02	0.01
phenol	5900 <sup>e</sup>	0.01	0.01	0.02	0.02
pentanoic acid	3000 <sup>e</sup>	0.01	0.01	0.01	0.01
heptanoic acid	3000 <sup>e</sup>	nd	nd	0.01	nd

<sup>a</sup> OAVs were calculated for the compounds perceived in GC–O and quantified. They could not be calculated for oct-3-en-2-one and 2,4-bis(1,1-dimethylethyl)phenol, since their odor threshold values were not available. nd: not determined since the concentration was below the detection limit of quantification (<2  $\mu\text{g/L}$ ). <sup>b</sup> Odor threshold values in water obtained from the literature. <sup>c</sup> Odor unit values were obtained by dividing the concentration of the odorant compound in the cooked rice by its odor threshold in water. <sup>d</sup> Buttery et al. (7). <sup>e</sup> Fazzalari (36).

in food matrixes (32, 33). Furthermore, GC–MS gave concentration results whatever the odor activity. The odor-active values (OAV) of the volatiles were therefore calculated for 17 compounds, perceived in GC–O and quantified, by dividing their concentration by their odor threshold in water (Table 4). Therefore, the OAVs provide only a tendency for the contribution of volatiles to cooked rice flavor, the effect of matrix being neglected.

Among the 17 compounds, 11 were present in the extracts with a concentration above their odor threshold (OAVs > 1): 2AP, (*E,Z*)-deca-2,4-dienal, 2-methoxy-4-vinylphenol, 4-vinylphenol, (*E,E*)-nona-2,4-dienal, decanal, hexanal, vanillin, oct-2-enal, indole, and butanoic acid. Five of them have been found with high OAV or FD values (5–7) for scented and nonscented cultivars: 2AP, 2-methoxy-4-vinylphenol, 4-vinylphenol, hexanal, and vanillin.

The OAV for 2AP was very high in scented rice cultivars, confirming its role in the flavor of these products. Its OAV could not be estimated in the nonscented cultivar as its concentration was below the quantification limit. This compound could be detected by all the panelists in GC–O analysis because of its odor threshold extremely low in air, 0.02 ng/L against 0.1  $\mu\text{g/L}$  in water (34). Similarly, some fatty acids such as butanoic acid, pentanoic acid, and phenol had OAVs less than 1 but displayed a relatively high frequency of detection in GC–O what was probably due to their low odor threshold in the air. The second highest OAVs were observed for (*E,Z*)-deca-2,4-dienal, in particular in the scented cultivars from Camargue for which it was in the same range as the OAV of 2AP. This component may thus have a great impact on the global aroma of scented Camargue cultivars. It is mainly formed upon autoxidation of linoleic acid, suggesting higher levels of the latter in those cultivars. In addition, the level of linoleic acid in rice was negatively correlated with ripening temperature (35). This may be the reason for the lower levels of (*E,Z*)-deca-2,4-dienal in the Thai sample. It is also possible that this aldehyde was partially converted through autoxidation to other compounds such as hexanal during storage. Indeed, the level of the latter

was significantly higher in the Thai sample than that in Camargue cultivars. In GC–O analysis of Asian scented cultivars, the FD factor of (*E,Z*)-deca-2,4-dienal was low, much lower than that of (*E,E*)-deca-2,4-dienal, for example (6), which is regularly cited as a volatile and odor-active component of scented Asian cultivars (5, 7, 26). In our GC–O conditions, (*E,E*)-deca-2,4-dienal could not be perceived, probably because of the intense odor of pentanoic acid eluted just before.

$\gamma$ -Decalactone and  $\delta$ -decalactone could not be quantified in our analysis conditions, because their concentrations were less than 2  $\mu\text{g/kg}$ . However, because of their odor threshold in water, 65 and 100  $\mu\text{g/kg}$ , respectively (36), their contribution to cooked rice aroma may be excluded.

In conclusion, organic extracts of cooked scented rice from Camargue cultivars (Aychade and Fidji) showed quite a similar aroma profile by GC–O analysis in comparison with a well-scented commercial rice, Thai. However, HCA and PCA statistical treatments evidenced quantitative differences for the levels of some flavor compounds, issued from the degradation of lipids and cinnamic acids, between scented Camargue cultivars and the Thai one. Those differences were probably due to the cultivar effect as well as to the growing and postharvest conditions.

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