



Article

The Volatile Compounds and Aroma Description in Various *Rhizopus oligosporus* Solid-State Fermented and Nonfermented Rice Bran

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Abstract: Rice bran is known to have beneficial nutrients. Current studies suggest that solid-state fermentation affects the rice bran's volatile profile. The aim of this study is to identify the volatile compounds and aroma description of fermented and nonfermented rice bran (FRB and NFRB) of Ciherang, Inpari30, IR64 and Inpari42. The fermentation was conducted using *Rhizopus oligosporus* solid-state fermentation. Headspace-solid phase microextraction coupled with GC/MS was performed, and the aroma was translated by 10 trained panelists through quantitative descriptive analysis (QDA). The result showed that 72 and 68 compounds were identified in FRB and NFRB, respectively. They are aldehydes, ketones, alcohols, acids, esters, fatty acid, phenol, benzenes, furan, thiazole, pyrazines, pyridine, lactones, terpenes, and hydrocarbons. The PCA showed that FRB was dominated by alcohols, whereas NFRB was dominated by aldehydes. The QDA described nine aromas, i.e., rancid, smoky, musty, grassy, green, earthy, cereal, and sweet in NFRB. The fermentation process added fermented attributes to the aroma description to FRB and enhanced the rancid, smoky, and musty aromas. These studies indicated that fermented rice bran might increase the volatile compound of rice bran. Thus, it may provide opportunities to develop the production of fermented rice bran as a functional ingredient.

Keywords: aroma description; fermentation; principal component analysis; rice bran; volatile compounds

1. Introduction

Rice bran (RB) is a by-product of milling rice that is produced in an estimated million tons per year. This high production has a great potential to be developed as functional ingredients, due to its high nutritional and bioactive compounds such as dietary fiber, vitamins, minerals, lipids, protein, γ -oryzanol, ferulic acid, and tocopherol [1].

The sensory profiles of RB odor are the most decisive in terms of acceptance. Volatile compounds are chemical compounds that play an essential role in regulating odors in food. The volatile compounds of RB have been detailed by a few investigators utilizing GC-MS to identify the compounds. The main volatiles of RB are aldehydes, ketones, acids, esters, hydrocarbons, alcohols, and alkanes, among which hydrocarbons, acids, and aldehydes

contribute to the off-flavor [2]. These compounds have an unacceptable odor that makes them difficult to consume.

Rancidity in RB is created by lipid oxidation and by the lipase and lipoxygenase in free fatty acids (FFA) such as oleic and linoleic acids which produce the off-flavor in RB [2–4]. The degradation of lipid results in an aldehyde group, one of which is hexanal as a marker that comes from linoleic acid degradation [3]. This is used as a degradation indicator in rice bran flavor quality.

Rice-based products that are fermented by variety of microorganisms are capable of producing various volatile compounds such as alcohol and acids in RB [4]. The fermentation process can remove the hexanal, reduce alcohol, and oxidize it into acid by microorganisms [5]. Fermentation has been shown to improve the bioactivities of RB in recent studies. There is an increase in the total phenolic compound and antioxidant activity of RB using *Rhizopus oryzae* [6] and *R. oligosporus* [7] in fermented rice bran (FRB).

In recent studies, 55 volatile compounds were identified in fermented rice bran (FRB) and nonfermented rice bran (NFRB) in the Indonesian popular cultivars IPR30 and Cempo Ireng [8]. They were categorized within aldehydes, ketones, acids, alcohols, esters, benzene, pyrazines, furans, phenols, terpenes, thiazoles lactone, and pyridines. Solid-state fermentation, performed by implementing *R. oligosporus*, can enhance the acceptance of RB [8].

Ciherang (C), Inpari30 (IPR30), Setra Ramos (IR64), and Inpari42 (IPR42) were the most widely circulated rice types in the Indonesian market because of their affordable price and relative suitability to the tastes of urban communities [9]. These four varieties were considered for use in this study because their accessibility ensured that the bran obtained was also abundant.

The descriptive analysis approach was recognized as a great and optimal measurement tool in assessing sensory attributes in various food products [10], especially fermented food products [11] and Indian milk products such as cham cham [10]. The training panelists measure product-specific quality attributes to produce product descriptions that could be statistically analyzed, which is a key principle of qualitative descriptive analysis (QDA) [10].

In comparison with our group in the previous study [8], these studies aimed to come up with novel information about the relationship between volatile compounds and aroma description in C, IPR30, IR64, and IPR42 fermented and nonfermented RB varieties.

2. Materials and Methods

2.1. Materials

This research used the superior seed quality of four varieties of rice: Ciherang (C), Inpari30 (IPR30), Setra Ramos (IR64), and Inpari42 (IPR42). The IPR30 was obtained from the Indonesian Center for Rice Research, Subang, West Java, Indonesia. C, IR64, IPR42 were purchased from rice collectors in Bantul, Yogyakarta, Indonesia. The culture *R. oligosporus* 6010 was purchased from the Center for Food and Nutrition Studies, Faculty of Agricultural Technology, Universitas Gadjah Mada, Yogyakarta, Indonesia. The alkane C8-28 was obtained from Sigma-Aldrich (St. Louis, MO, USA) as used by authentic standards.

2.2. Rice Bran Preparation

The samples of C, IPR30, IR64, and IPR42 rice varieties were milled using a grinder LM 24 (Bantul, Yogyakarta, Indonesia) to remove the husks and epidermis, carried out for approximately one minute, and grinder N50 inch (Bantul, Yogyakarta, Indonesia) for less than one minute to get the bran. The method used for RB preparation was carried out as in previous studies [7], followed by 40 mesh sieving. All samples were stored in a dark zipper pouch at 5 °C before being sterilized at 121 °C for 15 min in an autoclave. The purpose of sterilization is stabilization. All bran treatments were the same, both NFRB and FRB were sterilized for stabilization purposes as in previous studies [7].

2.3. Rice Bran Fermentation

The preparation of the culture and fermentation process followed the method used in a previous study [7] with slight modifications. Each nine grams sterilized RB and 20% (v/v) of distilled water were mixed and inoculated with 15% (v/w) of *R. oligosporus* (10^6 spore/mL) in a petri dish on sterile Potato Dextrose Agar (PDA) and incubated for 72 h at 30 °C. Following the mycelium growth evenly distributed, all samples of FRB and NFRB were dried by freeze-drying at BenchTop Pro with Omnitronics (Ipswich, UK) at −20 °C for 48 h.

2.4. Headspace Solid Phase Microextraction (HS-SPME)/GC-MS Analysis

HS-SPME was also used by referring to the previous study [12] with slight modifications. Fiber 50/30 µm DVB/CAR/PDMS 2 cm was used in this study from SUPELCO Bellefonte, PA (USA). The FRB and NFRB weighed as much as 3 g and were put into the SPME Agilent 22 mL vial then closed using a Septa PTFE/Silicon. Furthermore, in an 80 °C water bath for 30 min, the fiber DVB/CAR/PDMS was laid onto the headspace of a vial in a water bath. Afterward, the fiber was removed from the vial and injected into the GC-MS/Olfactometry injector at 250 °C desorption for 10 min. The SPME was carried out cleaning procedure after absorption.

Gas Chromatography-Mass Spectra (GC-MS) (Agilent Technologies, Palo Alto, CA, USA) 7890 A and MS Agilent 5975 C with triple exist detector XL EI/CI) was used to analyze the volatile compounds in RB. The GC-MS system was equipped with a splitless injection port at 250 °C. DB-WAX was (30 m × 250 µm × 0.25 µm) used as column. Initially, the oven temperature was set at 40 °C, which was later increased to 110 °C by the speed 5 °C/min and up again to 230 °C by the speed of 8 °C/min and then maintained for 5 min. The interface temperature was set at 250 °C. The carrier gas was helium with a flow rate of 0.8 mL/min.

The comparison of mass spectra with the NIST05A database was used to identify volatile compounds. In addition, further confirmation was carried out by comparing the retention index with the existing reference. Retention data of linear alkane solution (C8C28) in hexane were used to calculate linear retention index (LRI). Peak area percentage was obtained from the quantification of the comparison of the peak area to the sample weight and expressed in peak area percentage.

$$\text{peak area percentage} = \frac{\text{peak area}}{\text{peak area of total compound}} \times 100\% \quad (1)$$

2.5. Aroma Description

The aroma of FRB and NFRB was analyzed by QDA in a focus group discussion (FGD) [13] with 10 trained panelists (7 females and 3 males). The panels were trained based on ISO 8586-2012 for 3 h. The performance panel was carried out 3 times based on ISO 11132 followed by final evaluations. All panelists supplied informed consents before the examination.

Panelists were asked to provide an assessment of the aroma attributes contained in the sample. Eight samples consisting of four FRB and four NFRB were presented individually at room temperature by using a trivial code to avoid bias during testing. Then the panels were asked to inhale the aroma of the sample for 5 s and neutralize it with the aroma of coffee.

2.6. Statistical Analysis

The principal component analysis (PCA) was used to analyze the FRB and NFRB volatile compounds based on their peak area percentage and aroma description by trained panels. Pearson's correlation was applied for the mapping of cultivars based on volatile compounds using XLSTAT 2021 software.

3. Results and Discussion

3.1. Volatiles of Fermented and Nonfermented Rice Bran

The FRB and NFRB volatile compounds are listed in Table 1. Totals of 69 and 65 compounds were identified in FRB and NFRB volatile compounds, respectively. FRB included 10 alcohols, 8 aldehydes, 8 ketones, 7 acids, 7 esters, 6 naphthalene, 4 phenol, 2 furan, 1 benzene, 2 thiazole, 2 pyrazines, 2 pyridine, 2 lactones, 4 terpenes, and 4 hydrocarbons. NFRB included 12 aldehydes, 6 ketones, 8 alcohols, 9 acids, 3 esters, 4 phenols, 7 naphthalene, 1 benzene, 2 furans, 2 thiazoles, 1 pyridine, 2 lactone, 3 terpenes, and 4 hydrocarbons. As seen in Table 1, it could be concluded that the volatile compounds of FRB were more distinctive than NFRB, including increased peak area percentages. These results are relevant to previous results [3] that the fermentation process is the crucial way to improve the volatile compounds.

Regarding the sample, these compounds are generally considered to be derived from carbohydrates, triglycerides, protein, free amino acids, and their derivatives [14]. As shown in Figure 1, among these varieties, aldehydes were the dominant volatile, followed by alcohol, benzene, and acid, especially in FRB. The aldehyde group was formed through lipid oxidation, with the greatest contribution to the aroma in all categories, due to the comparatively lower odor threshold. The aldehyde content such as hexanal, heptanal, and nonanal increase during the storage. This was due to the increase in lipoxygenase and lipase activity [15]. Consequent to all the above-mentioned factors, the rancidity increased.

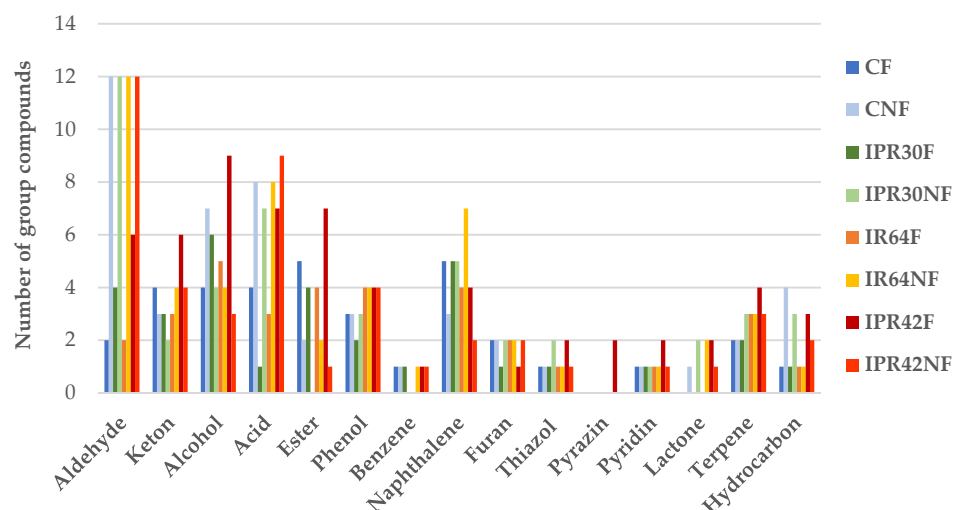


Figure 1. Number of volatile compounds in Ciherang fermented (CF), Inpari42 fermented (IPR42F), Inpari30 fermented (IPR30F), IR64 fermented (IR64F) Ciherang nonfermented (CNF), Inpari42 nonfermented (IPR42NF), Inpari30 nonfermented (IPR30NF) and IR64 nonfermented (IR64NF) rice bran.

In this study, D-limonene and nonanal had the highest peak area percentage with 4755 and 3748 in CNF. This is in accordance with the physical characteristics of its color, which is more yellowish than other varieties. In addition, according to a previous study in 2013 [45], RB had a rich bioactive component: β -carotene [11], so that it gives a citrus aroma and spices that are generally associated with terpenes [46]. Nonanal, contributing fatty, citrus, and green aroma has a low odor threshold of one ppb. Other than that, benzaldehyde is found to be an important fragrance for wild rice [47] as it supplies an almond and nutty-like odor. The uniqueness of the type of nutty and roasted aroma mainly results from various volatile active compounds, 2-pentylfuran, benzaldehyde, and furfural [47].

Table 1. Volatile compounds of fermented and nonfermented rice bran.

No	Volatile Compounds	Code	LRI ^a	LRI Ref ^d	Identifi Cation ^e	Peak Area Percentage								Odor Description
						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
Aldehyde														
1	Hexanal	Ad1	0	1065 [16]	MS + LRI	nd	2403.09	nd	nd	0.636	3255	0.646	0.176	grass, tallow, fat ^b
2	Heptanal	Ad2	1184	1186 [17]	MS + LRI	nd	nd	nd	nd	0.443	0.388	0.329	0.431	fat, citrus, rancid ^b
3	Octanal	Ad3	1287	1286 [18]	MS + LRI	nd	0.001	nd	nd	0.277	0.146	0.417	0.473	fat, soap, lemon, green ^b
4	Nonanal	Ad4	1391	1392 [17]	MS + LRI	nd	nd	nd	nd	3.748	1860	2375	1214	fat, citrus, green ^b
5	3-Furaldehyde	Ad5	1425	na	MS	nd	0.002	nd	nd	0.351	0.730	0.342	0.254	-
6	Furfural	Ad6	1448	1448 [18]	MS + LRI	0.056	0.004	0.006	0.383	0.468	0.746	0.521	0.605	bread, nutty, roasted, almond, sweet ^b
7	Decanal	Ad7	1495	1484 [17]	MS + LRI	nd	nd	nd	nd	0.749	0.761	0.615	0.800	soap, orange peel, tallow ^b
8	Benzaldehyde	Ad8	1517	1513 [17]	MS + LRI	nd	nd	nd	0.251	1111	0.764	0.639	0.745	almond, nutty, burnt sugar ^b
9	Benzene acetaldehyde	Ad9	1636	1643 [17]	MS + LRI	nd	nd	nd	nd	0.309	0.377	0.370	0.425	green, honey, alcoholic, chemical, sweet, caramel, bread, coffee, mostly ^c
10	3-methylbenzaldehyde	Ad10	1644	na	MS	nd	nd	nd	0.302	0.540	0.473	0.370	0.763	sweet fruity ^c
11	Cinnamaldehyde	Ad11	2046	2045 [19]	MS + LRI	nd	nd	nd	0.663	0.062	0.209	0.151	0.394	cinnamon, paint ^c
12	Vanillin	Ad12	2576	2578 [20]	MS + LRI	nd	nd	nd	nd	0.153	0.261	0.135	0.288	vanilla ^b
Ketone														
13	3-Penten-2-one	Kt1	1120	na	MS	0.546	nd	nd	nd	nd	nd	nd	nd	fruity ^c
14	3-Octen-2-one	Kt2	1388	1435 [21]	MS + LRI	0.056	0.001	nd	nd	0.220	nd	nd	nd	herbal ^c

Table 1. Cont.

No	Volatile Compounds	Code	LRI ^a	LRI Ref ^d	Identifi Cation ^e	Peak Area Percentage								Odor Description
						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
15	5-ethylidihydro-5-methyl-2(3H)-furanone	Kt3	1667	1684 [22]	MS + LRI	nd	nd	nd	0.392	nd	nd	0.245	nd	creamy ^c
16	6,10-dimethyl (E)-5,9-undecadien-2-one	Kt4	1857	1865 [17]	MS + LRI	nd	nd	nd	1612	nd	nd	nd	0.275	-
17	1-(1H-pyrrol-2-yl)-ethanone	Kt5	1977	1967 [23]	MS + LRI	nd	nd	0.004	0.404	nd	nd	0.189	0.363	musty ^c
18	dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone	Kt6	2037	na	MS	nd	nd	nd	0.288	nd	nd	nd	nd	cotton candy ^c
19	6,10,14-trimethyl-2-Pentadecanone,	Kt7	2130	2110 [24]	MS + LRI	0.030	0.005	0.005	0.324	0.258	0.379	0.393	0.330	green, fat, floral
20	5,6,7,7a-tetrahydro-4,4,7a-trimethyl-2(4H)-benzofuranone	Kt8	2368	na	MS	0.108	0.001	0.001	0.049	0.128	0.186	0.118	0.106	fruity ^c

Table 1. Cont.

No	Volatile Compounds	Code	LRI ^a	LRI Ref ^d	Identifi Cation ^e	Peak Area Percentage								Odor Description
						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
Alcohol														
21	Ethanol	Al1	0	913 [25]	MS + LRI	nd	nd	nd	2058	nd	nd	nd	nd	sweet ^b
22	2-methyl-1 propanol	Al2	110	1093 [26]	MS + LRI	nd	0.010	0.017	1527	0.499	nd	nd	nd	ethereal ^c
23	3-methyl-1- butanol	Al3	1212	1220 [25]	MS + LRI	nd	0.014	nd	nd	nd	nd	nd	nd	fermented ^c
24	1-Octen-3-ol	Al4	1453	1456 [27]	MS + LRI	nd	nd	nd	nd	0.338	nd	nd	nd	mushroom-like odor ^c
25	2,3- Butanediol	Al5	1546	1494 [28]	MS + LRI	1122	0.010	0.024	1933	0.420	nd	nd	nd	creamy ^c
27	4-ethyl-1,3- benzenediol	Al6	1572	na	MS + LRI	nd	nd	nd	nd	0.221	0.247	0.130	nd	-
28	2- Furanmethanol	Al7	1662	1686 [25]	MS + LRI	nd	nd	nd	0.307	0.206	nd	0.789	0.330	bready ^c
29	2- hexadecanol	Al8	1745	na	MS	nd	nd	nd	0.262	nd	0.152	nd	nd	-
30	benzyl alcohol	Al9	1882	1879 [17]	MS + LRI	nd	nd	nd	0.397	nd	nd	nd	nd	sweet, floral ^b
31	phenylethyl alcohol	Al10	1920	1920 [29]	MS + LRI	0.430	0.001	0.006	0.653	0.142	0.138	0.113	0.252	floral, slightly rose, sweet, clove-like ^c
32	Nicotinyl alcohol	Al11	2236	na	MS	0.906	0.009	0.010	1090	0.261	0.371	0.274	0.424	green ^c
Acid														
33	Acetic acid	As1	1449	1450 [16]	MS + LRI	nd	nd	nd	nd	0.648	0.121	0.845	1630	sour ^b
34	Butanoic acid	As2	1628	1628 [17]	MS + LRI	nd	nd	nd	0.949	0.171	nd	nd	0.129	cheesy, buttery ^c

Table 1. Cont.

No	Volatile Compounds	Code	LRI ^a	LRI Ref ^d	Identifi Cation ^e	Peak Area Percentage								Odor Description
						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
35	Hexanoic acid	As3	1847	1846 [26]	MS + LRI	0.273	nd	0.004	0.559	0.568	1823	0.373	1260	fatty, sour fatty cheesy ^c
36	Heptanoic acid	As4	1955	1954 [30]	MS + LRI	nd	nd	nd	0.148	0.105	0.323	0.116	1641	cheesy, rancid, sour ^c
37	Octanoic acid	As5	2064	2065 [17]	MS + LRI	0.244	0.002	0.003	0.276	0.102	0.450	0.139	0.340	sweet, cheese ^b
38	Nonanoic acid	As6	2163	2177 [17]	MS + LRI	0.053	nd	nd	0.055	0.653	0.508	0.439	0.259	green, fat ^b
39	Dodecanoic acid	As7	2492	2502 [26]	MS + LRI	nd	nd	nd	nd	nd	0.066	0.115	0.062	-
40	Tetradecanoic acid	As8	2704	2706 [26]	MS + LRI	0.083	nd	nd	0.109	0.261	0.351	0.502	0.137	waxy, fatty, soapy, coconut ^c
	Ester													
41	Ethyl hexanoate	Es1	1233	1230 [31]	MS + LRI	0.367	nd	nd	0.286	nd	nd	nd	nd	apple peel, fruity ^b
42	Methyl tetradecanoat	Es2	2015	1994 [18]	MS + LRI	0.448	0.004	0.005	0.247	nd	nd	0.122	nd	orris
43	Isobutyl palmitat	Es3	2374	na	MS	nd	0.035	0.037	3587	0.081	nd	nd	nd	grape ^b
44	9-Octadecenoic acid methyl ester	Es4	2452	na	MS	0.042	nd	nd	0.148	nd	nd	nd	nd	-
45	ethyl oleat	Es5	2052	2044 [32]	MS + LRI	1233	0.007	0.006	3081	nd	nd	nd	nd	fatty, fruity, oily ^c
46	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	Es6	2502	na	MS	0.047	nd	nd	2571	0.089	nd	0.113	0.075	fatty ^c

Table 1. Cont.

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						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
47	Ethyl linoleate	Es7	2535	2491 [18]	MS + LRI	nd	0.005	0.004	1954	nd	nd	nd	nd	fatty, fruity, oily ^c
Phenol														
48	2-methoxyphenol	Ph1	1862	1872 [17]	MS + LRI	1743	0.008	0.017	0.983	nd	0.606	0.207	0.114	phenolic ^c
49	Butylated Hydroxytoluene	Ph2	1912	1912 [23]	MS + LRI	nd	nd	0.003	0.256	0.070	0.147	0.101	0.121	phenolic ^c
50	phenol	Ph3	2008	2002 [33]	MS + LRI	1805	0.005	0.008	1325	0.050	nd	0.217	0.434	phenol ^b
51	2-Methoxy-4-vinylphenol	Ph4	2202	2200 [34]	MS + LRI	0.070	nd	0.007	0.710	1030	1502	1754	0.771	woody ^c
Benzene														
52	Styrene	Bz1	1254	1250 [35]	MS + LRI	0.306	0.003	nd	0.592	1094	nd	0.413	0.524	balsamic, gasoline ^b
Naphthalene														
53	Naphthalene	Na1	1738	1734 [17]	MS + LRI	1078	0.009	0.012	1220	0.686	1390	0.978	1862	tar ^b
54	2-methylnaphthalene	Na2	1853	1877 [36]	MS + LRI	0.383	0.002	0.002	0.326	0.112	0.256	0.110	0.153	floral ^c
55	1-methylnaphthalene	Na3	1889	na	MS	0.381	0.002	0.004	0.484	0.178	0.371	0.274	nd	naphthyl ^c
56	2,6-dimethylnaphthalene	Na4	1998	na	MS	0.067	0.001	0.001	nd	nd	nd	0.123	nd	grass ^b
57	2,7-dimethylnaphthalene	Na5	2006	2012 [36]	MS + LRI	0.106	0.001	nd	nd	nd	nd	0.129	nd	-
58	2,3-dimethylnaphthalene	Na6	2073	2122 [37]	MS + LRI	nd	nd	nd	0.102	nd	0.063	0.000	nd	earthy ^c
59	1,6,7-trimethylnaphthalene	Na7	2112	2122 [37]	MS + LRI	nd	nd	nd	nd	nd	0.075	0.040	nd	fruity strawberry

Table 1. Cont.

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						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
Furan														
60	2-Pentylfuran	Fr1	1229	1230 [38]	MS + LRI	0.062	0.001	0.001	nd	0.208	0.157	0.242	0.114	fruity, green, nutty, earthy, beany, vegetable ^c
61	2,3-dihydro-Benzofuran	Fr2	2398	2392 [39]	MS + LRI	0.092	nd	0.001	0.089	0.100	0.125	0.105	0.134	musky odor
Thiazole														
62	Thiazole	Th1	1804	na	MS	nd	nd	nd	0.178	nd	0.151	nd	nd	fishy ^c
63	Benzothiazole	Th2	1964	1950 [39]	MS + LRI	0.154	nd	0.002	0.371	0.238	0.315	0.335	0.366	gasoline, rubber-like ^b
Pyrazine														
64	2,6-dimethyl-pyrazine	Pz1	1318	1308 [40]	MS + LRI	nd	nd	nd	0.179	nd	nd	nd	nd	-
65	trimethyl pyrazine	Pz2	1387	1433 [25]	MS + LRI	nd	nd	nd	0.064	nd	nd	nd	nd	roast, potato, must ^b
Pyridine														
66	trimethyl-pyridine	Pr1	1373	na	MS	0.207	0.002	0.003	0.164	0.450	0.182	0.392	0.359	nutty
67	2-piperidinone	Pr2	1586	na	MS	nd	nd	nd	1308	nd	nd	nd	nd	-
Lactone														
68	Butyrolactone	Lc1	1623	na	MS	nd	nd	nd	0.351	0.100	0.159	0.122	0.230	creamy-milk, oily, fatty ^c
69	Pantolactone	Lc2	2037	2033 [41]	MS + LRI	nd	nd	nd	0.162	nd	0.746	0.416	nd	cotton candy ^b
Terpene														
70	3-Carene	Tp1	1140	1138 [18]	MS + LRI	0.390	0.004	0.005	0.355	0.794	0.577	0.701	1079	lemon, resin ^b
71	D-Limonene	Tp2	1197	1190 [23]	MS + LRI	0.979	0.009	0.040	1415	4755	1869	2404	3384	lemon, orange ^b

Table 1. Cont.

No	Volatile Compounds	Code	LRI ^a	LRI Ref ^d	Identifi Cation ^e	Peak Area Percentage								Odor Description
						Fermented				Nonfermented				
						CF	IPR30F	IR64F	IPR42F	CNF	IPR30NF	IR64NF	IPR42NF	
72	Caryophyllene	Tp3	1593	1593 [42]	MS + LRI	nd	nd	0.025	1344	nd	2371	0.478	0.614	spicy ^c
73	Epizonarene	Tp4	1758	na	MS	nd	nd	nd	0.434	nd	nd	nd	nd	-
	Hydrocarbon													
74	o-Xylene	Hd1	1134	1174 [18]	MS + LRI	nd	nd	nd	0.280	nd	nd	nd	6674,91	geranium ^b
75	o-Cymene	Hd2	1266	1260 [42]	MS + LRI	nd	nd	nd	0.254	nd	nd	nd	6048,25	-
76	Tetradecane	Hd3	1396	1400 [43]	MS + LRI	0.185	0.002	0.005	0.216	0.671	0.876	nd	0.393	alkane ^b
77	cyclodecanone	Hd4	1815	na	MS	nd	nd	nd	nd	0.156	0.180	nd	nd	-
78	2-Pentadecanone	Hd5	2025	1998 [44]	MS + LRI	nd	nd	nd	nd	0.021	0.000	0.066	0.095	floral ^c
79	Indole	Hd6	2454	2450 [42]	MS + LRI	nd	nd	nd	0.280	2371.57	nd	nd	nd	burnt, mothball ^b

^a Retention index was determined using n-alkane C8 to C28; ^b Flavornet.org <https://www.flavornet.org/flavornet.html> (accessed on 1 November 2021).; ^c The Good Scents Company Information System <http://www.thegoodscentscompany.com/search2.html> (accessed on 1 November 2021). ^d Linear Retention Index (LRI) Reference is obtained from journal references were analyzed by DB-WAX column; ^e Identification methods: retention index (RI) and mass spectra data (MS); nd (no detection); na (not available).

Generally, the results of heating processes such as the Maillard reaction and lipid oxidation are reported to produce volatile compounds that are unique to grain products [2]. The Maillard reaction, which forms the main volatile heterocyclic compounds, includes furan, pyridine pyrazine, thiophene, thiazole, pyrrole, imidazole, etc. [15]. Amino acids and reducing sugars are present in the Maillard reaction during heat treatment, resulting from lipid degradation as aldehydes and ketones [48].

Alcohols are considered inferior products from the oxidation of unsaturated fatty acids [15]. The second plentiful volatile in FRB were alcohols, consisting of ethanol and 2,3-Butanediol 2058 and 1933 which had the highest peak area percentage, respectively, in IPR42F. The formation of 2,3-Butanediol was from the catabolism of glucose to pyruvate via the glycolysis pathway. This gives it a characteristic buttery and creamy aroma. Although alcohol such as 1-octen-3-ol has relatively high odor thresholds, most of the phenol has relatively low odor thresholds which contribute to the rice flavor. In addition, 1-octen-3-ol (mushroom-like odor) is also one of the most plentiful volatiles in rice [15].

The highest peak area percentage of phenol was found in CF with 1805. During heating, the products produced from the decarboxylation of p-coumaric acid and ferulic acid in plants can be classified as phenol [15]. The most plentiful phenolic acid in RB is 2-methoxy-4-vinyl phenol, gained from ferulic acid, which is thermally decarboxylated. This produces a spicy or clove-like aroma. Those derived from lignin degradation and formed by thermal degradation of ferulic acid are vanillin (vanilla) [49].

Fat oxidation reactions are formed through the process of free radicals. These include oxygenated fats, primary oxidation products (hydroperoxides), and degraded and volatile oxides such as aldehydes, acids, esters, hydrocarbons, and ketones [48]. Ketone is mainly derived from amino acid degradation and beta-oxidation of saturated fatty acids during the fermentation process [50]. Esterification reaction between fatty acid and alcohol, resulting from carbohydrate fermentation or amino acid catabolism, forms an ester group [46]. This reaction is catalyzed by an esterase, wherein the formation of esters by one of the acetic acid precursors during fermentation produces fruity and floral notes.

Among these compounds, the aldehyde is the greatest aroma compound in the NFRB, followed by alcohols, acids, esters, benzenes, ketones, phenols, hydrocarbons, terpenes, furans, fatty acid, thiazoles, pyrazines, pyridines, lactones, and alkaloids. These group compounds dominated the FRB. Most of these are in higher content than NFRB, derived from rice fatty acids during fermentation. The large amount of FFA in RB is responsible for the level of lipid degradation products during fermentation [4].

3.2. Principal Component Analysis of Fermented and Nonfermented Rice Bran

PCA was applied to compare the volatile compounds of FRB and NFRB. The PCA of FRB, NFRB, and a combination of both is shown in Figure 2. In Figure 2a, the volatile compounds in FRB are located in a quadrant different from NFRB, thus indicating that fermentation influenced volatile compound production. The alcohol group was generally found in FRB, such as 2-methyl-1 propanol, 2,3-butanediol, phenylethyl alcohol, and nicotiny alcohol. Furthermore, octanoic acid, ethyl oleate; 6,10,14-trimethyl-2-pentadecanone, 2-methoxyphenol, phenol, and 9-octadecenoic acid methyl ester were also identified. This could be due to the fermentation process which increases the alcohol content. NFRB included hexanal, nonanal, furfural, and decanal aldehydes, benzaldehyde, 3-methylbenzaldehyde, butanoic acid, heptanoic acid, octanoic acid, 2-methoxy-4-vinylphenol, 3-carene, D-limonene, and caryophyllene.

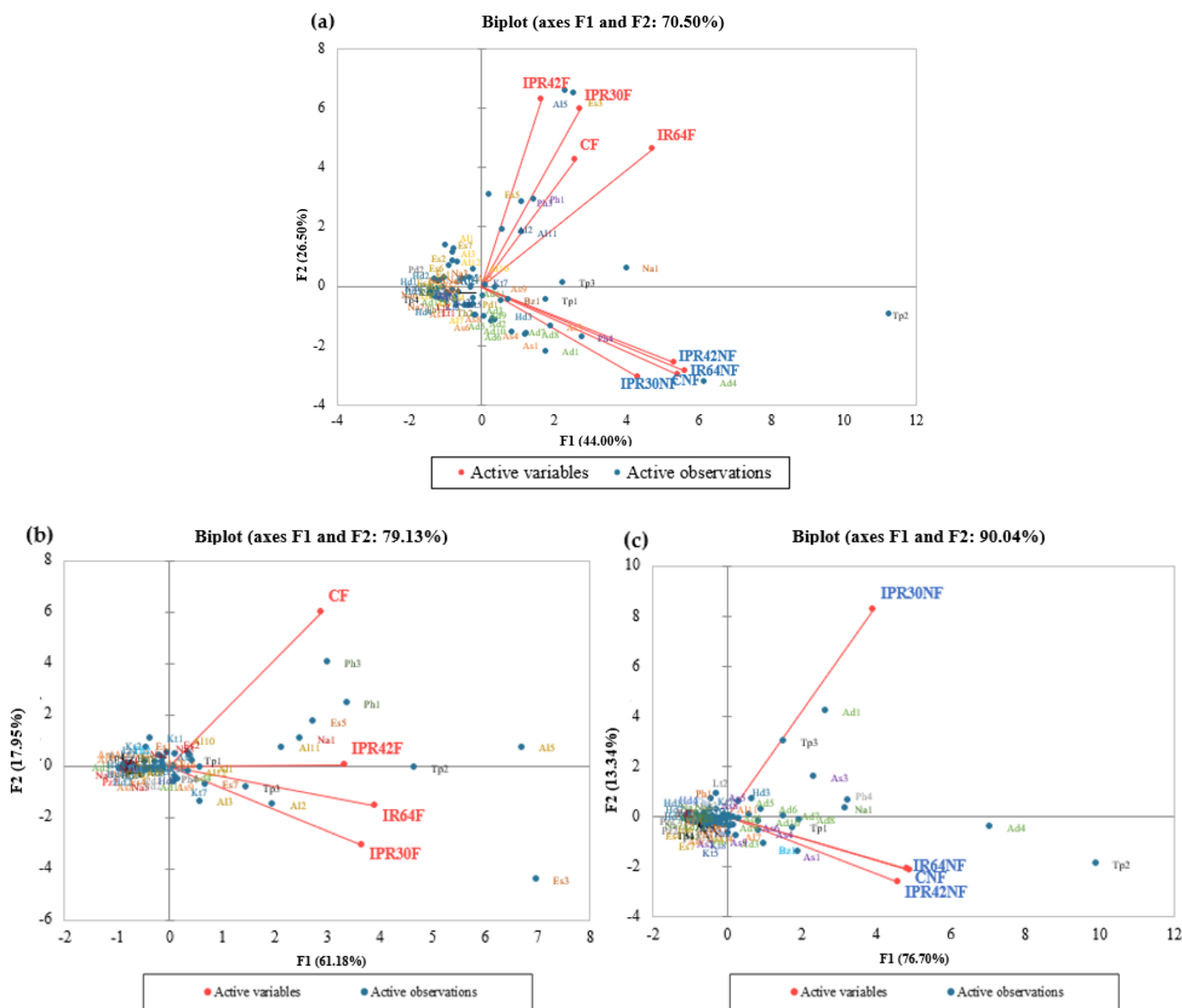


Figure 2. Volatile compound: (a) FRB and NFRB; (b) Ciherang fermented; (CF), Inpari42 fermented (IPR42F), Inpari30 fermented (IPR30F), IR64 fermented (IR64F) rice bran; (c) Ciherang nonfermented (CNF), Inpari42 nonfermented (IPR42NF), Inpari30 nonfermented (IPR30NF), and IR64 nonfermented (IR64NF) rice bran.

During fermentation, the fungal active enzyme produced alcohol [51] from microbial metabolites that can increase the volatile compound. Hydrolyzing starch to glucose via glycolysis produced the desired fermentation product such as ethanol. Other alcohol compounds identified in FRB were benzyl alcohol and phenyl ethyl alcohol indicating a slightly sweet aroma. This is following the origination from the degradation of carbohydrates [52].

The PCA volatile compound FRB (Figure 2b) showed that CF is in contrast from others, in which 2-methoxyphenol, phenol, benzyl alcohol, phenylethyl alcohol, nicotinyl alcohol, D-limonene, ethyl oleate, 1-methylnaphthalene, hexanoic acid, and 9-octadecenoic acid methyl ester were dominant. PCA in NFRB (Figure 2c), IPR30, and IPR42 have more volatile dominants such as hexanal, heptanal, 3-furaldehyde, furfural, benzaldehyde, benzene acetaldehyde, 3-methylbenzaldehyde, hexanoic, heptanoic, octanoic, nonanoic, and tetradecanoic acid, 3-carene, caryophyllene, 2-methoxy-4-vinylphenol, and a different quadrant from IR64 and C.

The acid produced from the aldehyde group is appropriate during the continuous oxidation process, and this acid had other aromas such as cheesy, sweet, sour, and fatty. The storage process can increase the intensity of the odor-active compound. In addition, acid aromas can be formed through the oxidation of aldehydes such as hexanoic acid, which was detected most abundantly in RB. The increase in levels may be the result of hexanal oxidation.

This difference in position is shown in addition to the different treatments. The characteristics of each variety are also different, even though the C and IPR30 varieties came from the IR64 selection. These different characteristics show the aroma profile, and the fermentation shows changes. This is the novel finding of this research.

3.3. Aroma Description of Fermented and Nonfermented Rice Bran

The QDA determined the effects of changes in the rice bran samples, including the fermentation process. Figure 3 represents the distinction of aroma description in FRB and NFRB. No panelist mentioned any fermented or alcohol odor in NFRB (Figure 3a). The PCA results of dominant aroma description in various RB are given in Figure 3c. These results showed the same grouping of volatile compounds, starting with different FRBs and NFRB quadrants.

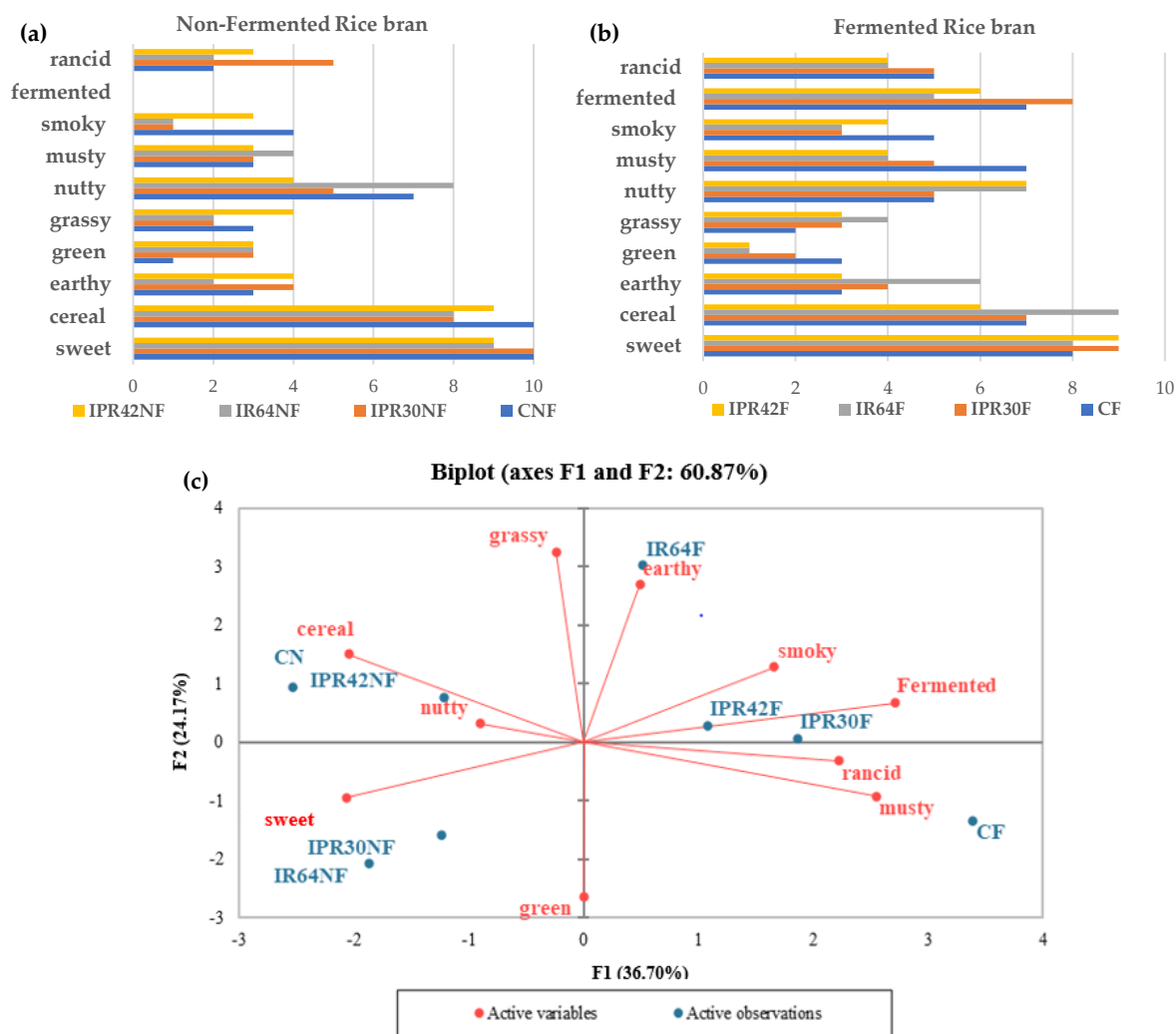


Figure 3. PCA of aroma description: (a) Ciherang nonfermented (CNF), Inpari42 nonfermented (IPR42NF), Inpari30 nonfermented (IPR30NF) and IR64 nonfermented (IR64NF) rice bran; (b) Ciherang fermented (CF), Inpari42 fermented (IPR42F), Inpari30 fermented (IPR30F), IR64 fermented (IR64F) rice bran; (c) FRB and NFRB.

On PCA the volatile compounds indicated that the alcohol group is the dominant group in the FRB. This is following the description of the aroma stated by the panelists of the presence of fermented, rancid, and musty aromas. On the other hand, the NFRB quadrant was dominated by the aldehyde group, where the aldehyde contributes to the fat, green, and rancid aroma. Uniquely, IPR30 and IR64 are stated to have a dominant aroma of sweet and green, while C and IPR42 are cereals, nuts, and grassy. The PCA biplot Figure 3c shows that the diversity in the first and second components is not much different, indicating that the distribution of the aroma description data stated by the panelists is evenly distributed.

3.4. Correlation Analysis between Volatile Compounds with Aroma Description (QDA)

To determine the relationship between volatile compounds and the aroma, which is descriptive in this study, testing was carried out using Pearson's correlation. There was a positive correlation between volatile compounds and aroma description as seen in Table 2. Furfural (0.801), benzaldehyde (0.760), benzene acetaldehyde (0.650), 3-methylbenzaldehyde (0.747), and pantolactone (0.792) have a correlation with sweet aroma. It implies that an increase in peak area percentage of these volatile compounds is highly correlated with the sweet aroma in FRB and NFRB. The results showed that 3-methyl-1-butanol (0.657) and octanoic acid (0.703 and 0.693) were related to fermented and rancid flavors that were produced by aldehyde oxidation and lipid hydrolysis, especial during storage. Moreover, the off-flavor of RB occurs lipid hydrolysis by lipase, and the RB is stabilized by autoclaving to inactive lipase and lipoxygenase. Therefore, the off-flavor of rice bran will not be produced during storage unless the rice bran is not enough stabilized by 15 min autoclaving.

Table 2. Pearson's correlation volatile compounds and aroma description in various fermented and nonfermented rice bran.

Variables	Furfural	Benzaldehyde	Benzene Acetaldehyde	3-methylbenzaldehyde	3-methyl-1-butanol	Octanoic Acid	Pantolactone
Sweet	0.801	0.760	0.650	0.747	0.000	−0.446	0.792
Cereal	0.493	0.575	−0.086	0.691	−0.309	−0.471	−0.143
Earthy	−0.293	−0.265	−0.358	−0.192	0.128	−0.125	0.044
Green	−0.350	−0.429	−0.574	−0.229	−0.051	0.138	0.415
Grassy	−0.070	0.011	0.026	−0.027	0.061	−0.395	−0.493
Nutty	0.241	0.281	0.381	0.120	−0.286	−0.306	−0.267
Musty	−0.583	−0.462	−0.401	−0.539	0.261	0.842	−0.216
Smoky	0.049	0.193	0.267	0.024	0.000	0.674	−0.703
Fermented	−0.569	−0.471	−0.153	−0.620	0.657	0.703	−0.203
Rancid	−0.495	−0.555	−0.318	−0.510	0.394	0.693	0.428

4. Conclusions

The study detected volatile compounds in FRB and 66 in NFRB, including aldehydes, ketones, alcohols, acids, esters, fatty acid, phenol, benzenes, furan, thiazole, pyrazines, pyridine, lactones, terpenes, and hydrocarbons. Volatile compounds from the fermentation process showed more diverse compounds than NFRB. The QDA define nine aromas, i.e., rancid, smoky, musty, green, earthy, cereal, and sweet in NFRB. The PCA plot showed that the volatile compound and aroma description of FRB is in the different quadrant with NFRB. The solid-state fermentation method added fermented attributes to the aroma description in FRB and enhanced the rancid, smoky, and musty aromas. The alcohol group was dominant in FRB, while the aldehyde group dominated NFRB. There was a positive correlation between volatile compounds and aroma description. Consequently, the relationship between volatile compounds and aroma description can be used as a characteristic aroma in various fermented and nonfermented RB varieties.

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