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VOLATILE AROMATIC COMPONENTS OF TWO VARIETIES OF PARBOILED NIGERIAN RICE

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ABSTRACT

The objective of this study is to analyse the volatile components of two parboiled aromatic varieties of Nigerian rice (*Ofada-0S6 and Caroline*). The compounds were extracted using the solvent extraction method and the concentrated extract was analysed with Gas Chromatography-Mass Spectrometry. Some of the volatile compounds varied with the varieties. The classification of components in *Caroline* rice are organic acids, alcohols, aldehydes, esters, alkenes, ketones, and amine while *Ofada* rice components include acids, alcohols, aldehydes, esters, alkanes, alkynes, ketones, phenol and others. The results showed high percentages of organic acids-53.50% and 50.00% for *Caroline* and *Ofada* rice respectively. Volatiles are responsible for flavour sensation and combinations of volatile matrices yield different flavour which characterise the aromatic nature and taste of Nigerian rice.

Key words: OS-6 Ofada, Caroline, Volatile, Organic acids, Aroma

INTRODUCTION

Rice is the leading food crops in the world and a favoured cereal in the diet appearing in different forms (Otegbayo *et al.,* 2001). Nigeria is reputed to have comparative resource advantage in rice (*Oryza sativa L*) production (Nkang *et al*., 2011). It is obvious that the market demand and value of rice depend on its physical qualities such as head rice yield, chalkiness, colour, size characteristics and so on which are subject to processing conditions and storage of paddy (Adekoyeni *et al.,* 2012; Patindol *et al*., 2008; Daniel *et al*., 1998). According to Saeed *et al.,*(2011) aside from appearance, eating and cooking qualities comprise the primary components of rice grain. Analysis of food volatile and non volatile components have long been important in order to evaluate its composition, monitor changes upon processing or cooking, and ascertain the nature of components that provide them with desirable (or undesirable) characters. Volatiles are just one aspect of the plethora of chemicals present in foods and usually responsible for the aromatic and taste characteristics of rice.

One of the popular indigenous rice varieties in Nigeria is *Ofada* rice. The original *Ofada* rice is short grain robust rice believed to be OS6 and ITA 150 varieties (NCRI and ARC, 2007). It is unpolished short grain with red kernel and could be likened to the popular "basmatic rice" from India and Pakistan. The rice is processed traditionally by parboiling method that involves three stages of treatment (soaking, parboiling and drying). This rice is specially relished due to its characteristics flavour that develops during soaking as a result of fermentation activities of some microorganisms (Adeniran *et al.,* 2012).

As *Ofada* rice is common to the Southwest, so also the variety of rice called *Caroline* is common in the northern part of Nigeria. The unprocessed grain of Caroline is white, smooth longer grain and relished because of its taste. Therefore, it is important to identify those components that contribute to aroma and taste of these Nigerian rice. A lot of researches have been conducted on determination of aromatic components of rough and cooked rice (Fukuda *et al.,* 2014; Bryant and McClung, 2011; Champagne, 2008; Yajima *et al,* 1978) with variations in their results. However, little or no work has been done on determination of aromatic components of Nigerian rice. Variations in the results of the researchers may be due to method adopted. Genetics plays a large role in the determination of rice grain attributes and non genetic factors also contribute substantially to the physical, chemical, and sensory attributes of rice (Bryant and McClung, 2011). Careful examination of research findings have lent strength to the influence of rough rice handling and processing on the quality of Nigerian rice. This study was undertaken to determine the volatile aromatic components of cooked *Ofada* and Caroline rice respectively.

MATERIALS AND METHODS

Rice samples:

The two rice varieties were obtained at different locations. *Ofada* rice (*OS-6*) was purchased at farmgate, Mokolokin, Ogun state (South-West, Nigeria) and *Caroline* rice was purchased in a local market at Gashua, Yobe state (North-East).

Rice processing:

The paddy was cleaned by winnowing to remove the chaffs and immature paddy. Paddy (2 kg) was soaked in cold water at ambient temperature (28 ±2 °C) typically for 5 days to hydrate the kernels. The water was changed daily to prevent off flavour due to fermentation. The soaked paddy were parboiled at 120 °C at constant pressure using digital autoclave for 15 mins. The parboiled paddy was tempered for 30 mins to cool and air dried in oven at 40 °C. The rice samples were milled (hulling and debranning) in grantex cono disc milling machine.

Extraction of volatile compounds of rice:

The method of Liyanaarachchi *et al.,* (2014) was used in the extraction of the volatile components. 250 g sample of milled rice was grinded using laboratory scale hammer mill and sieved manually with 60 µg mesh. 5 g was taken as a representative sample and the volatile components were extracted with 10 ml of methylene chloride (CH_2Cl_2), which was kept in water bath at 70 °C for 5 h. The solvent extraction process was carried out at lower temperature to reduce loss and deterioration of the desired products. The solvent extraction was done repeatedly for three times. The extract was dried over anhydrous sodium sulphate and concentrated to 1.0 ml using rotary evaporator at 7.5 rpm.

Gas Chromatography- Mass Spectrometry Analysis

The volatile compounds of parboiled milled rice were analysed by Gas Chromatography-Mass Spectrometry (GC-MS, Shimadzu GCMS-QP2010PLUS) equipped with OI 4660 "Eclipse" Purge and Trap Concentrator with an OI 4551A autosampler (Sakamoto *et al.,* 2010).

A temperature profile of 35-220 °C was utilized, and the advanced flow control (AFC) was programmed to provide a constant linear velocity of 35 cm/sec. A total analysis time of 12.5 mins gives a GC cycle time that corresponds approximately to the cycle time of the purge and trap concentrator. The mass spectrometer was scanned in the full-scan mode from m/z 36 to 260 in 0.5 sec scan intervals. The volatile components of unknown samples were identified by computer through searching and matching NIST and Weily MS libraries. The volatile components were quantified using peak area normalization. The detector (electron multiplier) was adjusted to give adequate response at the lowest calibration level and avoid saturation at the highest calibration level.

RESULTS AND DISCUSSION

The components in the parboiled *Caroline* and *OS 6* variety of *Ofada* rice are presented in tables 1 and 2 respectively while the classifications of the compounds and aroma characteristics are shown in tables 3 and 4. The classification of components in Caroline rice are: acids, alcohols, aldehydes, esters, alkenes, ketones, and amine while Ofada rice components include- acids, alcohols, aldehydes, esters, alkanes, alkynes, ketones, phenol and others. The results showed high percentages of acids 53.50% and 50.00% for Caroline and *Ofada* rice respectively. Caroline contained more of alcohols (13.95%), alkenes (6.98%), aldehydes (2%), esters (11.63%), ketones (6.98) and amines (2.33%); while alcohols (10%), aldehydes (10 %), esters (12.5 %), alkanes (2.5%), alkynes (2.5%), ketones (2.5%), phenol (2.5%), and others (2.5%) were found in Ofada rice. 2-acetyl-1-pyrroline (2-AP), has been reported as one of the important volatiles in rice flavour and commonly recorded in aromatic rice (Liyanaarachchi *et al.,* 2014; Champagne, 2008; Maraval *et al.,* 2008 and Bergman *et al.*, 2000). However, this was not detected in any of the varieties. Due to the highly unstable nature of 2-acetyl-1-pyrroline (2-AP), it is scarcely used commercially in flavour formulations. It is clear that other compounds are responsible for cherished taste and flavour of Nigeria rice.

Table 1: Volatile components of *Caroline* rice

Chemical name	Retention time (min)	Retention Index
Cyclohexene	5.742	1018
1,7- Octodiene	6.842	940
Nitroisobylglycerol	12.95	1444
1,3-propanediol, 2 ethyl-2-(hydroxymethy	13.05	1261
D-glucopyranoside	13.9	3139
Propylene carbonate	13.95	875
Phthalic acid	14.5	2286
2-Acetylbenzoic acid	15.41	1499
3,3-Dimethyl-1-1(2-carboxyphenyl)triene	15.72	1582
6-Amino-1,2,3,4-tetrahydroindan-5-7-dione	16.5	1989
Phthalic acid, monoethyl ester	16.32	1629
Methyl 14- methylpentadecanoate	18.03	1814
Decanoic acid	18.47	1282
Methyl 6-methyl heptanoate	18.63	1019
Hexadecanoic acid	18.78	1914
Tridecanoic acid	19.97	1580
Palmitic acid	19.05	1968
Eicosanoic acid	19.45	2366
Heptadecanoic acid(margarinic)	19.75	2067
Octadecanoic acid (stearic acid)	19.85	2167
Pentadecanoic acid	20.04	1869
11-Octadecenoic acid	20.433	2085
Cyclopropaneoctanoic octanoic acid	20.84	1941
6-Octadecenoic acid	21.1	2085
15-Tetracosenoic acid	21.433	2682
10-Octadecenoic acid	21.64	2085
Oleic acid	22.23	2175
E-9-Tetradecenoic acid	22.325	1777
E-2-Octadecadecen-1-ol	22.55	2061
Pentanoic acid	22.72	1769
Hexadecenoic acid	23.89	1976
Octadecenyl aldehyde	24.617	2007
1,3-propanediol	25.31	1934
Allylpentadecyl ester (oxalic acid	25.88	2334
Allyl hexadecyl ester (oxalic acid)	26.01	2433
Glycerol-1-palmitate	26.12	2482
Butyldiethylene glycolacetate	26.75	1334
Butylamine	26.874	1334
3-Cyclopropyl-4-hydroxy-4,5,5-trimethyl-oxazolidin-2-one	27.08	1485
Palmitic acid beta monoglyceride	27.33	2498
9-Tetradecenal	27.675	1609
Cis-11-Hexadecenal	27.86	1808
Cyclopentadecanone	28.2	2031
Linoleic acid chloride	28.06	2139

Table 2: Volatile components of *Ofada* rice

Chemical name	Retention time (min)	Retention index
Acetic acid	1.54	819
Acetaldehyde	1.67	651
Glycolaldehyde	3.27	1074
Phthalic acid(di-(1-hexen-5-yl) ester	14.28	2286
Monoethyl ester (benzoic acid)	14.396	1629
Diethyl ester	14.6	1639
Acetyl benzoic acid(2-acetyl)	14.692	1499
6-Amino-1,2,3,4-Tetrahydroindan-5,7-dione	14.921	1989
Methyl caprinate	16.82	1282
Tridecanoic acid	17.46	1580
Hexadecanoic acid	18.21	1914
Pentadecanoic acid	18.642	1814
Hexanoic acid	18.891	1019
Palmitic acid	19.775	1968
Nonadecanoic acid	19.93	2266
n- Decanoic acid (caprinic acid)	20.74	1372
11-Octadecenoic cid	21.13	2082
Cyclopropanepentanoic acid	21.433	2140
6-Octadecenoic acid	21.68	2085
9-Dodecenoic acid	21.73	1489
Cyclooropanedodecanoic acid	21.901	2538
Oleic acid (cis-oleic acid	22.367	2175
Hexadecenoic acid	22.56	1976
Z-8-Methyl-9-tetradecenoic acid	22.738	1813
Erucic acid	23.45	2572
Octadecenyl aldehyde	24.28	2007
Undecylenic aldehyde	24.77	1293
E.9-Tetradecenal	25.05	1609
3,11-Tetradecadien-1-ol	25.617	1672
E-2-Octadecadecen-1-ol	25.84	2061
Glycerol-1-monopalmitate	26.12	2482
Hexadecanoic acid(Hexadecanoyl glycerol)	26.58	2498
Pentadecanoic (ethylpentanoate)	26.96	2399
Decanoic acid(octyl decacoate)	27.125	1978
Glycerol linolenate(9,12,15- Octadecatrienoic acid)	27.3	2705
Cyclohexane-1,2-diol	27.683	1739
Cyclohexanone	27.683	1468
Hexadecadien-1-ol	27.83	1870
1-Tetradecyne	29.34	1411
Carbonicdihydrazines	2.06	0
Indole	19.68	2074
2-benzothiazole	21.44	1829
Hydrazine	1.833	0

Table 3: Classification of volatile components of *Caroline* rice

Caroline	Aroma characteristics
Acids	
Phthalic acid	
2-acetylbenzoic acid	Roasty
Decanoic acid	Sweaty
Tridecanoic acid	Waxy, woody
Hexadecanoic acid (palmitic acid)	Soapy, waxy
Eicosanoic acid (arachidic)	
Heptadecanoic acid	
Octadecanoic acid (stearic)	
Pentadecanoic acid	
11-Octadecanoic acid	
6-Octadecenoic acid	
Cyclopropaneoctanoic acid	
15-Tetracosenoic acid	
Oleic acid	Fatty
9-Tetradecenoic acid	
10-Octadecenoic acid	
Pentanoic acid	
Hexadecenoic acid	Waxy
Oxalic acid (allyl pentadecylester)	
Oxalic acid (allyl hexadecylester)	
Hexadecanoic acid (palmitic acid)	
Palmitic acid beta monoglyceride	
Alcohols	
Nitroisobutyl glycerol	
1,3-Propanediol	
Sucrose	
E-2-Octadecadecen-1-ol	
2-Dodecyl-1-3-propanediol	
Ethanol (Butyl diethylene glycol acetate)	
Aldehydes	
9-Tetradecenal	
Cis-11-Hexadecenal	
Esters	
Propylene carbonate	
Monoethyl ester	Fruity
Methyl 4-methyl penta decanoate	
15-Methyl-methylester	
9-Octadecenal	Citrus, sweet
Alkenes	
1,7- Octodiene	
3,3-Dimethyl-1-(2-carboxyphenyl)triene	
Cyclohexene	Floral black currant
Ketones	
6-Amino-1,2,3,4-tetrahydro indan-5,7-dione	
3-Cyclopropyl-4-hydroxyl-4,5-trimethyl-oxazolidin-2-one	
Cyclopentadecanone	Musk
Amines	
Acetylamine (N-acetyl-1-cyano-2-methyl)	

Table 4: Classification of volatile components of *Ofada* rice

Ofada	**Aroma characteristics**
Acids	
Phthalic acids	
Acetyl benzoic acids(2-acetyl)	Roasty
Decanioc acid	Sweaty
Tridecanoic acid	Goaty
Hexadecanoic acid	
Pentadecanoic acid	
Hexanoic acid	Cheesy, sweaty, waxy
Palmitic acid	Waxy
Nonadecanoic acid	
Caprinic acid	Burnt hair
11-octadecanoic acid	
Cyclopropanpentanoic acid	
6-Octadecenoic acid	
9-Dodecenoic acid	
Cyclopropandodecanoic acid	
Oleic acid	
Hexadecenoic acid	
Z-8-Methyl-9-tetradecenoic acid	
Erucic acid	
9,12,15-Octadecatrienoic acid	
Alcohols	
3,11-Tetradecadien-1-ol	
E-2-Octadecadecen-1-ol	Sweet fruity
Hexadecanoyl glycerol	
Hexadecadien-1-ol	
Aldehydes	
9-Octaldecenal	Citrus, sweet
E-9-Tetradecenal	Blood smell, citrus
Glycolaldehyde	
Acetaldehydes	Ethereal, fruity
Esters	
Monoethyester	Fruity
Diethyl phthalate	
Glycerol-1-monopalmitate	
Ethylpentanoate	Fruity
Octyl decanoate	Floral smell
Alkanes	
Cyclohexanes	
Alkynes	
1-Tetradecyne	
Ketones	
6-Amino-1,2,3,4-tetrahydroindan-5,7,-dione	
Phenol	
Cyclohexanone	
Others	
Carbonidihydrazines	
Indole	
2-benzothiazole	
Hydrazine	

The presence of acetic (organic acid), acetaldehydes (aldehydes) and glycoaldehydes contributed to aromatic nature of foods. Acids such as 2-acetylbenzoic, decanoic acid, tridecanoic acid, hexanoic, and caprinic may contribute to the aroma of the rice. 2-acetylbenzoic has good flavour and is documented to be responsible for the aroma of cocoa and tridecanoic to the aroma of goat milk (Sie-farth and Buettner, 2014). Phthalic acid is an aromatic dicarboxylic acid with low toxicity level. Decanoic acid is sweaty, and formed from oxidation of primary alcohol-decanol and used in the manufacture of esters for artificial fruit flavours and perfumes. Hexedecanoic is a common saturated fatty acid found in plant. It adds to texture and mouth feels of processed foods. Pentadecanoic is a fatty acid of exogenous origin and usually found in milk fat from cow (Wolk *et al.,* 2001). According to Qian and Reineccius (2002), hexanoic acid odour was described as fatty, cheesy and like that of goat milk. Methyl oleic acid and n-hexadecanoic acids are parts of major aroma compounds in Korean soy sauces and barley bran souses (Choi *et al.,* 2007). The different characteristics odour, flavour, and contribution to taste of these acids may be responsible for aroma of Nigerian rice.

There are more aldehydes in *Ofada* rice compared to *Caroline*. Benzothiazole is notable for slightly sweet aroma (Bryant and McClung, 2011) while octaldecenal, tetradecenal, and acetaldehyde are found responsible for certain fruity and blood flavour. The aromatic nature of octaldecenal is found in cooked meat. Aldehydes are considered to enhance flavour quality, which is mainly associated with sweat fruit, nutty, and caramel like odour (Fors, 1983: Xiao *et al.,* 2016).

By reaction process, certain amino acids (serine and cysteine) degrade to produce acetaldehyde and glycolaldehyde that can undergo aldol condensation to produce furan after cyclization and dehydration steps (Hofmann and Schieberle, 2002). Indole is known with sweet, floral and burnt flavour characteristics. Alcohol such as E-2-Octadecadecen-1-ol is a bioactive phytochemical component and taste sweet fruity (Raza *et al.,* 2013). Other groups of compounds such as ketones, esters and hydrocarbons present also have their individual flavour which is contributed to the aroma of the parboiled rice.

Lipid oxidation products and unsaturated fatty acids were identified in the parboiled rice which is known to have a negative impact on acceptability (Champagne, 2008). Production of unsaturated fatty acids such as oleic acid, palmitic acid and decenoic acid may be due to fermentation (Suomalainen and Lehtonen, 1978). The processing operation of Nigerian local rice usually involve soaking of paddy for 3-7 days and such may lead to the production of unsaturated fatty acids. Oleic acid is a monounsaturated fatty acid but odourless and colourless. It is known that some fatty acids inhibit -oxidation in mammals. The unsaturated fatty acids may become more prevalent with the length of storage time or due to poor post-harvest handling (Bryant and McClung, 2011).

Ojinnaka and Ojimelukwe (2013) reported that in addition to functional impartation of fruity floral characteristics by esters, their presence can also diminish or mask the sharpness of free fatty acids derived notes. Mostly, these esters are formed by esterification between the short chain free fatty acids and the alcohols (Qin and Ding, 2007). Alcohols, phenols, and amines can give a pleasant aroma like 2-acetyl-1-pyrroline (2-AP). Increase in sucrose concentration, increase aroma volatility (Cavarrubias-Cervantes *et al.,* 2004) may affect stability of volatile aroma in Caroline variety. The compound (1,3- propanediol) and its favourable properties have long been known; also production of 1,3- propanediol from microbial fermentation of glycerol has been described more than 120 years ago (Sauer *et al.,* 2008). Soaking of rice paddy for 5–7 days during processing may result to conversion of glycerol into 1,3- propanediol.

CONCLUSIONS

Organic acids, aldehydes, ketones, amine, alcohols, hydrocarbons and esters are found to be the major constituents that provide aromatic attributes of *Ofada* and *Caroline* varieties of Nigerian rice. Volatile compound in different combination and matrices yield different flavour and such produce distinct aromatic perception responsible for these rice varieties. However, 2-acetyl-1-pyrroline (2-AP), which is a common compound in most aromatic rice, was not detected in any of the varieties.

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