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Volatile Constituents of *Ogiri-Igbo* Condiment Produced by Traditional Alkaline Fermentation of Castor Oil Bean (*Ricinus communis*)

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Abstract
This study was undertaken to determine and compare the volatile profiles of *ogiri-igbo* using 2% NaCl and 2% lime. Volatile components of traditionally fermented castor oil bean condiments (*ogiri-igbo*) were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS). Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST) having more than 62,000 patterns. The three samples include fermented castor oil bean condiments (*ogiri-igbo*) with 2% NaCl, 2% lime and without 2% NaCl/lime. A total of 150 volatiles were identified in the *ogiri* samples. Esters, terpenes, alcohols, acids, ketones, pyrazine, amines, amides and other additional compounds were established and authenticated. However, esters, terpenes, alcohols, acids, ketones, pyrazine, amines and amides accounted for over 50% of the volatile compounds produced in the traditionally fermented castor oil bean condiments. The predominant volatile compounds identified in 2% NaCl *ogiri-igbo* were esters (14%), ketones (14%), amides (12%) and terpenes (10%). Esters (14%) and terpenes (12%) were more abundant in *ogiri-igbo* without NaCl/lime. However, 2% lime *ogiri-igbo* had 10% amines and amide each as the predominant volatile compounds. Most of the identified volatile compounds contribute significantly to the flavour and taste of fermented castor oil bean condiments (*ogiri-igbo*).

Keywords: Volatile constituents, castor oil bean, ogiri-igbo

Introduction

Ogiri is a traditionally fermented food condiment which is widely consumed in eastern and some southern part of Nigeria as protein-rich meat substitute. *A* product of alkaline fermentation of some oil seeds and legumes such as castor oil seed (*Ricinus communis*), African oil bean (*Pentaclethra macrophylla*), African mesquite seed (*Prosopis africana*), melon seeds (*Citrullus vulgaris , Colocynthis vulgaris*), fluted pumpkin seed (*Telferia occidentalis*) etc. Traditionally, these condiments are commonly known as *ogiri-igbo* (castor oil bean), *ogiri – ugba* (African oil bean), *ogiri-okpeye or ogiri-okpei* (African mesquite seed), *ogiri-egusi* (melon), *ogiri-ugu* (fluted pumpkin seed). *Ogiri-igbo* is a traditional condiment manufactured by fermenting castor oil bean seeds (*Ricinus communis*). It is a product of alkaline fermentation of castor oil bean (*Ricinus communis*) which is similar in sensory attributes to African locust bean (*Parkia biglobosa*) daddawa (Omafuvbe, 2006). The fermented castor oil bean (*ogiri-igbo*) condiment is used to impart flavour and enhance meatiness in soups, sauces, and other prepared dishes.

Ogiri-Igbo like other forms of *ogiri* is a solid state fermentation product known for its characteristic ammoniacal flavor that impacts on the taste and flavour of the local Nigerian dishes (Omafuvbe *et al*., 2004, Ezekwe *et al*., 2020). It is a viable source of plant protein and health aiding phytochemicals (Ahaotu *et al*., 2020, Ezekwe *et al*., 2020). Ammoniacal odour has been found to be a common odour of most fermented leguminous products as a result of protein degradation (Onyenekwe *et al*., 2014) however each fermented product has unique characteristic odour that makes it possible for one product to be differentiated from another apart from the texture and colour. This study was therefore designed to identify the volatile constituents produced by traditionally fermented castor oil bean seeds (*ogiri-igbo*).

Materials and Methods

The castor oil bean, lime and salt (sodium chloride) used for this project work were purchased from Ahia Afor Lokpaukwu in Umunneochi L.G.A. of Abia State.

Production of fermented castor oil bean seed (ogiri-igbo) samples

The method described by Ojimelukwe *et al.* (2011) with slight modifications was used. The castor oil seeds were sorted and boiled at 100^0C for three hours. The seeds were dehulled, drained and rinsed in clean water. They were boiled again for one hour and allowed to cool. After cooling, the mash was divided into three parts. To one part was added 2% of lime juice, to the second part was added 2% salt while the third portion served as the control; with no salt or lime. They were then wrapped with enough banana leaves and packed in clean container to ferment for four days. After fermentation, the seeds were ground into paste and oven-dried at 40^0C to obtain the three different *ogiri-igbo* samples.

Volatile compounds determination of ogiri-Igbo

Sample preparation

The samples were first extracted using spectroscopic pure ethanol under a cold extractor. The extracted components were immediately used for GCMS analysis.

Analysis

GC-MS analysis was carried out on a GC Clarus 500 Perkin Elmer system comprising of a AOC-20i auto-sampler and gas chromatograph interfaced to a mass spectrometer (GC-MS) instrument employing the following conditions: column Elite-1 fused silica capillary column (30 x 0.25 mm ID x 1 µM df, composed of 100 % dimethylpoly diloxane), operating in electron impact mode at 70 eV; helium (99.999 %) was used as carrier gas at a constant flow of 1 ml /min and an injection volume of 0.5 µI was employed (split ratio of 10:1) injector temperature 250 ºC; ion-source temperature of 280 ºC. The oven temperature was programmed from 110 ºC (isothermal for 2 min), with an increase of 10 ºC/min, to 200 ºC, then 5 C/min to 280 ºC, ending with a 9 min isothermal at 280 ºC. Mass spectra were taken at 70 eV; a scan interval of 0.5 seconds and fragments from 40 to 450 Da. Total GC running time was 36 min.

Interpretation

Interpretation of mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST) Abuja, having more than 62,000 patterns. The spectrum of the unknown component was compared with the spectrum of the known components stored in the NIST library. The name, molecular weight and structure of the components of the test materials were ascertained. The concentrations of the identified compounds were determined through area and height normalization.

Results and Discussion

Table 1 shows the percent composition of the different volatile compounds identified in the traditional fermented castor oil bean (*ogiri-Igbo*) samples. A total of 50 volatile constituents were identified in each of the traditionally fermented castor oil bean samples (with 2% NaCl, 2% lime and without 2% NaCl/lime). The esters and ketones were the dominant compounds in the 2% NaCl *ogiri-igbo* sample while esters, terpenes and alcohols were dominant in the fermented castor oil bean sample without 2%NaCl/lime. The amides and amines were the only dominant compounds in the 2%lime *ogiri*. Azokpota *et al*. (2008) reported pyrazines as the dominant constituent group in *afitin, iru* and *sonru* (fermented soybean food condiments). This is also contrary to the findings of Ojinnaka and Ojimelukwe (2013) who found acids as the dominant volatile compounds in bacillus fermented castor oil bean condiment. Volatile compounds have been shown to be partly responsible for the aroma of fermented foods (Zhao *et al*., 2011, Ezeocha *et al*., 2022). This can be supported by the finding of Azokpota *et al*. (2010) and Zannou *et al*. (2018) who reported that product obtained within 48h of fermentation are richer in volatile compounds than 24h of fermentation

Table 2 shows the identified compounds in 2%NaCl *ogiri-igbo*. The esters present in the 2%NaCl *ogiri-igbo* sample were Bicyclo[3.1.0]hexan-3-ol, 4-methylene-1-(1-methylehyl)-, acetate, 10,12-Tricosadiynoic acid, methyl ester, [1,1'4-Bicyclohexyl]-4carboxylic acid, 4'-propyl-, 4-pentylcyclohexyl ester but they occurred at different retention times. [1,1'4-Bicyclohexyl]-4carboxylic acid, 4'-propyl-, 4-pentylcyclohexyl ester occurred at retention times of 7.361, 12.104, 25.924, 24.029 secs and at percentage area normalized at 0.49, 0.75, 0.22, 2.09%. 10,12-Tricosadiynoic acid, methyl ester was identified at retention time of 7.005, 11.748 secs and relative peak area of 1.05 and 0.43%. Esters have been reported as the major volatile compounds in most African fermented seasonings and are likely to be the product of reactions between microbial acidic and alcoholic metabolites which have been associated with nice flavour (Leejeerajumnean *et al*., 2001). Ezekwe *et al*. (2020) in their work on qualitative phytochemical and GC-MS analysis of fermented castor seed (*Ogiri* Igbo), reported 9,12-Octadecadienoic acid (Z,Z)-methyl ester as the most abundant in their study. The acetates of higher alcohols and the ethyl ester of fatty acids had been suggested to be the most desirable compounds in miso products to enhance the aroma of the finished products and are responsible for the fruity tinge of freshly prepare miso (Giri *et al*., 2010). Ezeocha *et al*. (2022) in their study on the evaluation of indigenous *Okpeye (Prosopis africana)* processing conditions and its effect on the quality of the fermented seasoning, reported that aroma compounds such as esters and alcohol were more abundant in the fermented samples than in the raw samples. Esters have also been reported to be responsible for quality sensory properties of various fermented foods (Perestrelo *et al*., 2006).

Ketones were among the dominant compounds identified and quantified in the 2%NaCl *ogiri-igbo*

sample. They include 10-Undecen-4-one,2,2,6,6-tetramethyl, 2-Acetylcyclopentanone and 2-Cyclopenten-1-one,3,4-dimethyl, 2-Acetylcyclopentanone. These compounds occurred at different retention times and different peak. Ketones are usually derived from lipid and amino acid degradation during microbial fermentation and have a high impact on food odour (Owens *et al*., 1997). Akanni *et al*. (2018) identified aldehydes, acids, and ketones as key volatile compounds in *Bacillus* alkaline fermented bambara groundnut into dawadawa- type African food condiment. Onyenekwe *et al*. (2014) also identified ketones in their work on identification and quantification of headspace volatile constituents of okpehe, fermented *Prosopis africana* seeds and reported that ketones may contribute to the odour of Okpehe. Amides were also identified in 2%NaCl *ogiri-igbo* (N-Benzylacrylamide, Propanamide and Phenylacetamide, N-ethyl-N-(3-methylphenyl)-).

Table 3 showed only amines and amides as the dominant compounds in 2% lime *ogiri* sample. The identified amines were 2-Propyn-1-amine, N,N-di-2-propynyl-, Methoxyamine, TMS derivative and Isobutylamine while the amides were N-Benzylformamide and N-Benzylformamide. The results shown in Table 4 shows the major compounds identified in fermented castor oil bean (*ogiri-igbo*) without 2% NaCl/lime addition as esters, terpenes and alcohols. The esters identified were Bicycle[3.1.0]hexan-3-ol,4-methylene-1-(1-methylethyl)-, acetate, 10,12-Tricosadiynoic acid, methyl ester, [1,1'Bicyclohexyl]-4-carboxylic acid,4'-propyl-,4 pentylcyclohexyl ester –, Fumaric acid,2,2-dichloroethyl decyl ester and Ethyl 4-{[(3-cyano-4,6-dimethylpyridin-2-yl)sulfanyl]methyl}-5-methyl-1,2-oxazole-3-carboxylate. Esters, mainly formed by esterification of carboxylic acids and alcohols were reported to determine the characteristic pleasant aromatic notes (Klesk and Qian, 2003). The importance of ester contributions toward food aroma is undisputed with the fact that esters with low carbon atoms are highly volatile at ambient temperatures and the perception thresholds are ten times lower than their alcohol precursors (Nogueira *et al*., 2005).

Terpenes identified in this sample include Alpha.-phellandrene, Linalool, (Z,Z)-alpha.-Farnesene, gamma.-Muurolene, Germacrene D and Trans-.beta.-Ocimene. Alpha.-phellandrene was identified at 7.12% concentration at a retention time of 5.20secs while linalool 3.33% at retention time of 5.80 secs. Germacrene D was identified at 2.21% at a retention time of 27.66 secs. Terpenes contribute to the aroma of some food condiments and spices. Terpenes have been reported as the main compound in *P.guineense* responsible for their characteristic flavour (Jirovetz *et al*., 2002). Owolabi *et al.* (2013) reported linalool as the major oil responsible for the characteristic flavor in fruit berries of *P.guineense.*

Alcohols were among the major compounds identified in the sample fermented without the addition of salt and lime (Table 4). The alcohols identified were 2H-Pyran-3-ol, tetrahydro-2-(1,7-nonadiene-3,5-diynyl)-,11-Fluoroundecan-1-ol,TMS derivative, 4,4-Dimethyl-cyclohex-2-en-1-ol, 4-Cyclononen-1-ol and 4-Cyclononen-1-ol. It has been reported that alcohols contribute to the flavour of okpehe, fermented *Prosopis africana* seeds condiment (Onyenekwe *et al*., 2014). Alkanols present in the condiment help prevent spoilage since they are known to act as antifungal and prevent food spoilage (Onyenekwe *et al*., 2012, Onyenekwe *et al*., 2014). Alcohols contribute to the flavour of the condiments. This is consistent with previous work where alcohol has been reported in fermented *Prosopis Africana, okpee* , (Onyenekwe *et al*., 2014, Ezeocha *et al*., 2022) as important contributor of flavour.

Acids identified in the sample fermented without salt and lime addition (Table 4) were Aminocaproic acid, Z-7-Hexadecenoic acid and 5-Hydroxyanthranilic acid. They occurred at different retention times. The highest concentration of the acids was found in 5-Hydroxyanthranilic acid (5.99%) at retention time of 31.342 secs. Hexadecanoic acid is a common saturated fatty acid found in plants. n-Hexadecanoic acids were also identified in all the samples in the study of the volatile compounds and amino acid profile in *bacillus* fermented castor oil bean condiment (Ojinnaka and Ojimelukwe, 2013). Some of these acids are used in various industries as flavouring agents. Some organic acids have been determined as major aroma compounds in Korean soysauces and barley bran sauces (Steinhaus and Schieberle, 2007). Amides were also among the least identified in the sample fermented without the addition of salt and lime (Propanamide and N-Benzylacrylamide).The amides identified could be precursors of pyrazines which has been related to sensory attributes of legumes (Lee and Ahn, 2009).

As shown from the three identification tables, pyrazines were absent in the samples fermented with 2% salt and 2% lime but subsequently appeared only in the sample fermented without the addition of salt and lime (Table 4). The identified pyrazine was 2-[5-(4-Methoxyphenyl)-1H-1,2,4-triazol-3-yl]pyrazine. Ezekwe *et al*.(2020) also reported a pyrazine compound, 2-t-Butyl-3,6-dimethyl pyrazine (0.35%) as the least abundant in their study using fermented castor oil bean seed. On the contrary, Onyenekwe *et al.* (2012) reported pyrazines as the most dominant flavor constituents of daddawa (fermented locust bean seeds) and the major components identified therein being 2, 5 – dimethyl pyrazine, tetramethyl pyrazine and trimethyl pyrazine. Dajanta *et al*.(2011) reported pyrazines as the dominant volatile compound in thua nao, a Thai fermented soy product. Pyrazines has been related to the sensory attributes of soy sauce (Lee and Ahn, 2009). From this study pyrazines were identified only in the sample fermented without the addition of salt and lime.

Conclusion

Many volatile compounds were identified in traditionally fermented castor oil bean (*ogiri-igbo*)

samples. Esters, terpenes, alcohols and amides were identified as key volatile compounds in the three fermented castor oil bean samples and these compounds contribute mainly to the total flavour profile of these condiments. More research on characterization of the aroma-active consitutents of the *ogiri-igbo* is therefore encouraged.

References

Ahaotu. N., Nnennaya,I., Chidi, J., Agunwa, I.E. and Chinelo, K. (2020). Antinutritional and phytochemical composition of fermented condiment (*ogiri*) made from Sandbox (Huracrepitan) Seed. *European Academic Research*, 8(4):1871-1883

Akanni, G.B., De Kock, L., Naudé, Y. and Buys, E.M. (2018). Volatile compounds produced by Bacillus species alkaline fermentation of bambara groundnut (*Vigna subterranean* (L.) Verdc) into a dawadawa-type African food condiment using headspace solid-phase microextraction and GC × GC–TOFMS. *International Journal of Food Properties,* 21(1): 930- 942

Azokpota, P., Hounhouigan, D.J., Annan, N.T., Odjo, T., Nago, M.C. and Jakobsen, M. (2010). Volatile compounds profile and sensory evaluation of Beninese condiments produced by inocula of *Bacillus subtilis*. *Journal of the Science of Food and Agriculture*, 90: 438-44.

Azokpota, P., Hounhouigan, D.J., Annan, N.T., Nago, M.C. and Jakobsen, M. (2008). Diversity of volatile compounds of *afitin*, *iru* and *sonru*, three fermented food condiments from Benin. *World Journal of Microbiology and Biotechnology,* 24:879-85.

Dajanta, K., Apichartsrangkoon, A., Chukeatirote, E. and Frazier, R.A. (2011). Free amino acid profiles of thua nao, a Thai fermented soybean. *Food Chemistry,* 125(2), 342-347.

Ezekwe, A.S., Rizwan, A.A., Karimah, M.R. and Ewa, O. (2020). Qualitative phytochemical and GC-MS analysis of fermented castor seed (*Ogiri* Igbo). *GSC Biological and Pharmaceutical Sciences,* 13(02): 284-289

Ezeocha, C.V., Ugwuja, J.L. and Onyeabor, C.S. (2022). Evaluation of Indigenous *Okpeye (Prosopis africana)* Processing Conditions in Nsukka L.G.A, Enugu, Nigeria and Its Effect on the Quality of the Fermented Seasoning. *Nigerian Agricultural Journal*, 53(1). 191 - 199

Giri, A., Osako, K. and Ohshima, T. (2010). Identification and characterisation of headspace volatiles of fish miso, a Japanese fish meat based fermented paste, with special emphasis on effect of fish species and meat washing. *Food Chemistry*, 120: 621–631

Jirovetz, L., Buchbauer, G., Ngassoum, M.B. and Geissler, M. (2002). Aromacompound analysis of *Piper nigrum* and *Piper guineense* essential oils from Cameroon using solid-phase microextraction–gas chromatography, solid-phase microextraction–gas chromatography–mass spectrometry and olfactometry. *Journal of Chromatography,* 976:265-275.

Klesk, K. and Qian, M. (2003). Preliminary aroma comparison of Marion (*Rubus spp. Hyb*) and Evergreen (*R. laciniatus* L.) Blackberries by dynamic headspace/OSME Technique (RH Marion and Evergreen Volatiles). *Journal of Food Science, 68*: 697-700

Lee, S.J. and Ahn, B. (2009). Comparison of volatile components in fermented soybean pastes using simultaneous distillation and extraction (SDE) with sensory characterization. *Food Chemistry,* 114: 600-609

Leejeerajumnean, A., Duckham, S.C., Owens, J.D. and Ames, J.M.(2001). Volatile compounds in *Bacillus* fermented soybeans. *Journal of the Science of Food and Agriculture. 81*(5), 525-529.

Nogueira, M.C.L, Lubachevsky, G. and Rankin, S.A. (2005). A study of the volatile composition of Minas cheese. *Lebensm-Wiss. Technologie, 38*, 555-563.

Ojinnaka, M.C. and Ojimelukwe, P.C. (2013). Study of the Volatile Compounds and Amino Acid Profile in *Bacillus* Fermented Castor Oil Bean Condiment. *Journal of Food Research,* 2(1): 191 - 203

Ojimelukwe, P.C., Okechi, A. and Ojinnaka MC. (2011). Physicochemical characteristics of fermenting castor seeds containing lime and NaCl as additives. *African Journal of Food Science, 5*(14):754-760.

Omafuvbe, B,O. (2006). Effect of salt on the fermentation of soybean (*Glycine max.)* into daddawa using *Bacillus subtilis* as starter culture. *African Journal of Biotechnol*ogy, *5*(10):1001-1005.

Omafuvbe, B.O., Olumuyiwa, S.F., Osuntogu, B.A. and Adewusi, R.A. (2004). Chemical and biochemical changes in African locust bean (*Parkia biglobosa*) and melon (*Citrullus vulgaris*) seeds during fermentation to condiments. *Pakistan Journal of Nutrition*, 3(3):140-145.

Onyenekwe, P.C., Odeh, C. and Enemali, M.O. (2014). Identification and quantification of headspace volatile constituents of okpehe, fermented *Prosopis africana* seeds. *International Food Research Journal*, 21(3): 1193-1197

Onyenekwe, P.C., Odeh, C. and Nweze, C.C. (2012). Volatile constituents of *ogiri,* soybean daddawa and locust bean daddawa, three fermented Nigerian food flavour enhancers. *Electronic Journal of Environment, Agriculture and Food Chemistry,* 11(1):15-22.

Owens, J.D., Allagheny, N., Kipping, G. and Ames, J.M. (1997). Formation of volatile compounds during *Bacillus subtilis* fermentation of soya beans. *Journal of the Science of Food and Agriculture, 74*: 132-140.

Owolabi, M.S., Lawal, A.O., Ogunwande, I.A., Hauser, R.M. and Setzer, W.N. (2013). Aroma chemical composition of Piper guineense Schumach. & Thonn. From Lagos, Nigeria: a new chemotype. *American Journal of Essential Oils and Natural Products,* 1:37-40.

Perestrelo, R., Fernandes, A., Albuquerque, F.F., Marques, J.C. and Camara, J.S. (2006). Analyticalcharacterization of the aroma of Tinta Negramole red wine; identification of the main odorants compounds. *Analytica chemical Acta.* 563(1-2): 154-164.

Steinhaus, P., and Schieberle, P.(2007). Characterization of the key aroma compounds in soy sauce using approaches of molecular sensory science. *Journal of Agriculture and Food Chemistry,* 55: 6262-6269.

Zannuo, O., Chabi, I.B.and Koca, I. (2018). Some traditional fermented foods from African locust beans (*Parkia biglobosa*): production and volatile compounds. *Global Scientific Journal,* 6(9): 673 - 701

Zhao, J., Dai, X., Liu, X., Zhang, H., Tang, J. and Chen, W. (2011). Comparison of aroma compounds in naturally fermented and inoculated Chinese soyabean pastes by GCMS and GCOl factometry analysis. *Food Control*, 22(6): 1008-1013.

Table 1: Percent composition of different volatile compounds identified in 2% NaCl *Ogiri*, 2 % lime *Ogiri* and *Ogiri* without Nacl/lime (%)

S/N	Compounds	2% NaCl *Ogiri*	2% lime *Ogiri*	*Ogiri* without Nacl/lime
1	Esters	14	4	14
2	Terpenes	10	-	12
3	Alcohols	6	2	10
4	Acids	6	4	6
5	Ketones	14	2	6
6	Pyrazine	-	-	2
7	Amines	-	10	2
8	Amides	12	10	4
9	Other compounds	38	68	44

Table 2: Identified volatiles in *Ogiri* with 2% Nacl (%)

S/N	Compounds	Retention time (secs)	Relative Peak Area (%) *Ogiri* (with 2% Nacl)
	Ester		
1	10,12-Tricosadiynoic acid, methyl ester	7.005	1.05
2	[1,1'4-Bicyclohexyl]-4carboxylic acid, 4'-propyl-, 4-pentylcyclohexyl ester	7.361	0.49
3	Bicyclo[3.1.0]hexan-3-ol, 4-methylene-1-(1-methylehyl)-, acetate	8.565	8.52
4	10,12-Tricosadiynoic acid, methyl ester	11.748	0.43
5	[1,1'Bicyclohexyl]-4-carboxylic acid, 4'-propyl-,4-pentylcyclohexyl ester	12.104	0.75
6	[1,1'Bicyclohexyl]-4-carboxylic acid, 4'-propyl-,4-pentylcyclohexyl ester	25.924	0.22
7	[1,1'Bicyclohexyl]-4-carboxylic acid, 4'-propyl-,4-pentylcyclohexyl ester	24.029	2.09
	Terpenes		
8	.alpha.-Phellandrene	6.765	1.08
9	Linalool	7.860	6.00
10	(Z,Z)-apha.-Farnesene	13.283	1.44
11	.gamma.-Muurolene	13.841	6.20
12	Germacrene D	17.445	2.21
	Alcohol		
13	Bicyclo[4.1.0]heptan-3-ol,4,7,7-trimethyl-,(1.alpha.,3.alpha., 4.alpha., 6.alpha.)-	8.458	0.14
14	2H-Pyran-3-ol, tetrahydro-2-(1,7-nonadiene-3,5-diynyl)-	12.544	6.35
15	Cyclododecanol,1-aminomethyl-	29.109	0.41
	Acid		
16	Hexanoic acid,6-hydroxy	5.166	0.45
17	Hexanoic acid, 6-hydroxy-propanamide	10.886	0.35
18	Z-7-Hexadecenoic acid	16.413	0.31
	Ketone		
19	10-Undecen-4-one,2,2,6,6-tetramethyl	5.016	0.98
20	2-Acetylcyclopentanone	5.806	1.09
21	2-Cyclopenten-1-one,3,4-dimethyl	8.307	0.92
22	2-Acetylcyclopentanone	8.661	1.46
23	2-Acetylcyclopentanone	8.984	12.11
24	10-Undecen-4-one,2,2,6,6-tetramethyl-	9.646	1.14
25	10-Undecen-4-one,2,2,6,6-tetramethyl-	10.035	0.19
	Amides		
26	N-Benzylacrylamide	7.143	1.06
27	Propanamide	7.892	0.65
28	N-Benzylacrylamide	11.982	0.16
29	Phenylacetamide, N-ethyl-N-(3-methylphenyl)-	23.309	0.35
30	N-Benzylformamide	25.676	2.11
31	N-Benzylformamide	27.496	0.32
	Others		
32	Imidazo[4,5-e] [1,4] diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro	5.998	0.37
33	1,8-Cyclopentadecadiyne	6.211	0.12

34	Propane, 1-isocyano-	6.617	0.45
35	Hexane, 1-chloro-5-methyl-	6.798	2.57
36	Benzene, 1-methyl-3-(1-methylethyl)-	7.451	7.09
37	Cyclohexene,1-methyl-4-(1-methylethenyl)-, (S)	7.499	4.22
38	4-chloro-3-nitro-2H-pyrazole	8.110	0.11
39	1,1'-Bicyclopropyl	8.217	0.21
40	2-(1,2,3,4-Tetrazol-1-yl)acetonitrile	8.369	0.24
41	p-Xylene	8.507	1.34
42	Phenol,2,3,4,6-tetramethyl-	8.713	1.24
43	Cyclohexene, 2-ethynyl-1,3,3-trimethyl	8.843	5.09
44	2-Hexanal,2-ethyl	11.019	2.00
45	Hexane, 1-chloro-5-methyl-	11.312	0.21
46	Naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-(IS-cis)-	14.198	8.34
47	Benzo[b]naphtho[1,2-e] [1,4]dioxin-6a(6H)-ol,5,12a-dihydro-	25.533	0.45
48	(Cyanocyclohexyl) carbamate	19.015	4.09
49	1,5-Hexadiyne	22.754	0.17
50	Thiosemicarbazide,4-(1-adamantylcarbonyl)-	26.833	0.66

Table 3: Identified volatiles in *Ogiri* with 2% lime (%)

S/N	Compounds	Retention time (secs)	Relative Peak Area (%) *Ogiri* (with 2% lime)
	Ester		
1	2-Propylphenol, pentafluoropropionate	14.730	5.65
2	[1,1-Bicyclohexyl]-4-carboxylic acid, 4'-propyl-, 4-penylcyclohexyl ester	23.879	3.91
	Alcohol		
3	Cyclododecanol,1-aminomethyl-	28.432	1.57
	Acid		
4	Aminocaproic acid	20.796	1.63
5	Aminocaproic acid	22.009	0.52
	Amine		
6	2-Propyn-1-amine, N,N-di-2-propynyl-	7.971	0.74
7	Methoxyamine, TMS derivative	9.856	1.12
8	Methoxyamine, TMS derivative	10.746	0.84
9	Isobutylamine	13.990	0.55
10	Isobutylamine	14.066	0.74
	Amides		
11	Propanamide	9.764	0.57
12	Propanamide	9.919	0.67
13	Propanamide	10.031	0.62
14	Propanamide	11.221	0.79
15	N-Benzylformamide	24.694	1.12
	Ketone		
16	2-Acetylcyclopentanone	7.106	2.87

	Others		
17	Pyridine	5.495	0.70
18	Pyridine	5.512	0.90
19	Benzofurazan-1-oxide, 6-cyano-	6.605	0.59
20	Benzaldehyde,4-methyl-, oxime,(Z)-	6.736	2.18
21	Imidazol[4,5-e] [1,4] diazepine-5,8-dione, 1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro-	7.388	0.56
22	1,8-Cyclopentadecadiyne	7.582	0.97
23	1,8-Cyclopentadecadiyne	8.006	0.51
24	Tripropylene glycol monopropyl ether, TMS derivative	8.189	2.48
25	o-Xylene	8.362	27.0
26	Aziridine,2,2-dimtethyl-	8.549	4.57
27	Propanal, oxime	8.767	4.86
28	Hexane,1,6-dichloro-	8.869	1.32
29	Aminoacetonitrile	8.909	0.50
30	Aminoacetonitrile	9.341	0.53
31	Aminoacetonitrile	9.552	0.69
32	1H-3a,7-Methanoazulene, octahydro-1,4,9,9-tetramethyl-	9.621	1.54
33	Cyclobutanone,2methyl-2-oxiranyl	9.816	1.25
34	Guanidine, methyl	11.295	0.72
35	4-chloro-3-nitro-2H-pyrazole	12.651	0.65
36	4-chloro-3-nitro-2H-pyrazole	13.035	1.31
37	1,3-Diphenyl-4H-1,2,4-triazoline-5-thione	14.414	0.72
38	1-t-Butyl-4-(adamantly-1) benzene	14.375	0.70
39	Trans-3,4,5-trimethoxy-beta.-methyl-.beta-nitrostyrene	14.547	3.57
40	1,3-Diphenyl-4H-1.2.4-triazoline-5-thione	15.720	1.51
41	Phenylacetamide, N-ethyl-N-(3-methylphenyl)	15.972	0.92
42	Thiazol-2(3H)-one, 4,5-diphenyl	16.498	1.29
43	Imidazol[4,5-e] [1,4]diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro	16.744	1.58
44	Imidazol[4,5-e] [1,4]diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro	17.812	0.14
45	9(10H)-anthracenone,1,4-dimethoxy	17.944	0.56
46	Imidazol [4,5-e] [1,4] diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro-	18.013	0.51
47	Imidazol [4,5-e] [1,4] diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro-	18.670	2.59
48	Imidazol [4,5-e] [1,4] diazepine-5,8-dione,1,4,6,7-tetrahydro-1,4,7-trimethyl-2-nitro-	19.562	0.74
49	Thiosemicarbazide,4-(1-adamantylcarbonyl)-	26.603	0.78
50	N-Benzylformamide	27.009	1.84

Table 4: Identified volatiles in *ogiri* samples (without salt/lime) (%)

S/N	Compounds	Retention time (secs)	Relative Peak Area (%) *Ogiri* (without salt/lime)
	Ester		
1	Bicycle[3.1.0]hexan-3-ol,4-methylene-1-(1-methylethyl)-, acetate	6.329	3.50
2	10,12-Tricosadiynoic acid, methyl ester	13.548	0.68
3	[1,1'Bicyclohexyl]-4-carboxylic acid,4'-propyl-,4-pentylcyclohexyl ester	14.114	0.75

4	Fumaric acid,2,2-dichloroethyl decyl ester	21.961	3.19
5	[1,1'Bicyclohexyl]-4-carboxylic acid, 4'-propyl-4-,4-pentylcyclohexyl ester	22.653	0.29
6	Ethyl 4-{[(3-cyano-4,6-dimethylpyridin-2-yl)sulfanyl]methyl}-5-methyl-1,2-oxazole-3-carboxylate	22.716	0.19
7	[1,1'-Bicyclohexyl]-4-carboxylic acid, 4'-propyl-, 4-pentylcyclohexyl ester	26.024	0.39
	Terpenes		
8	Alpha.-phellandrene	5.200	7.12
9	Linalool	5.809	3.33
10	(Z,Z)-alpha.-Farnesene	14.583	1.44
11	.gamma.-Muurolene	24.314	6.20
12	Germacrene D	27.666	2.21
13	Trans-.beta.-Ocimene	29.004	0.83
	Alcohol		
14	2H-Pyran-3-ol, tetrahydro-2-(1,7-nonadiene-3,5-diynyl)-	14.354	6.35
15	11-Fluoroundecan-1-ol,TMS derivative	20.914	2.30
16	4,4-Dimethyl-cyclohex-2-en-1-ol	22.299	0.47
17	4-Cyclononen-1-ol	29.708	1.28
18	2-Octyl-1-ol	32.129	0.80
	Acid		
19	Aminocaproic acid	22.985	0.25
20	Z-7-Hexadecenoic acid	26.373	0.40
21	5-Hydroxyanthranilic acid	31.342	5.99
	Ketone		
22	2-cyclopenten-1-one,3,4-dimethyl-	6.121	0.51
23	2-Acetylcyclopentanone	6.390	0.66
24	2-Acetylcyclopentanone	6.784	9.93
	Pyrazine		
25	2-[5-(4-Methoxyphenyl)-1H-1,2,4-triazol-3-yl]pyrazine	23.323	0.96
	Amine		
26	Ethanamine,1-(2,4-cyclopentadien-1-ylidene)-N,N-dimethyl	29.653	0.26
	Amide		
27	Propanamide	5.876	0.17
28	N-Benzylacrylamide	13.925	0.26
	Others		
29	Propane,1-isocyano -	5.017	0.74
30	Benzene, 1-methyl-3-(1-methylethyl)	5.376	5.43
31	Cyclohexene,1-methyl-4-(1-methylethenyl)-,(S)-	5.515	2.18
32	4-chloro-3-nitro-2H-pyrazole	5.923	0.23
33	1,1'-Bicyclopropyl	6.037	0.41
34	2-(1,2,3,4-Tetrazol-1-yl) acetonitrile	6.189	0.64
35	Bicyclo[4.1.0]heptan-3-ol,4,7,7-trimethyl-,(1.alpha.,3.alpha.,4.alpha.,6.alpha.)-	6.246	0.30
36	p-xylene	4.987	2.29
37	Phenol,2,3,4,6-tetramethyl	6.521	0.44
38	Cyclohexene,2-ethenyl-1,3,3-trimethyl	6.644	1.73

39	2-Hexenal,2-ethyl-	7.019	2.90
40	Hexane,1-chloro-5-methyl-	12.752	0.47
41	Tricyclo[2.2.1.0(2,6)]heptane,1,7- dimethyl-7-(4-methyl-3-pentenyl)-	15.732	5.91
42	4,8-Dioxatricyclo[5.1.0.0(3,5)]octane,1-methyl-5-(1-methylethyl)-	20.125	1.94
43	Benzenamine,2,6-dichloro	20.582	0.41
44	4,7-Methanoazulene, 1,2,3,4,5,6,7,8-octahydro-1,4,9,9-tetramethyl-[IS-	23.935	2.10
45	Phenylacetamide, N-ethyl-N-(3-methylphenyl)-	24.084	0.27
46	Naphthalene,1,2,3,4,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(IS-cis)-	24.530	8.34
47	Benzo[b]naphtha[1,2-e][1,4]dioxin-6a(6H)-ol,5,12a-dihydro-	24.833	0.30
48	(1-Cyanocyclohexyl) carbamate	28.015	1.19
49	1,5-Hexadiyne	28.754	1.07
50	Propanal,oxime	28.809	0.27