IDENTIFICATION AND CHARACTERISATION OF FLAVOURANTS ASSOCIATED WITH OFADA RICE (*Oryza sativa* LINNAEUS) PADDY PRE-TREATMENTS

BY

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BSc, MSc Food technology (IBADAN)

A thesis in the Department of Food Technology submitted to the Faculty of Technology in partial fulfilment of the requirements for the Degree of

DOCTOR OF PHILOSOPHY

of the

UNIVERSITY OF IBADAN

August 2021

CERTIFICATION

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DEDICATION

This work is dedicated to God, the giver, and sustainer of life. And to the ever-loving memory of my father, Pa Olagoke Osunrinade.

ACKNOWLEDGEMENTS

I am grateful to God for the opportunity to begin and complete this work in sound health and mind. I am extremely indebted to my supervisor, Professor R. Akinoso for teaching me the art of research and being a father indeed through his gentleness, motivation, and versatility in knowledge. I am grateful sir. I also appreciate outstanding scholars like Prof O. C. Aworh, Prof G. O. Adegoke, Prof K. O. Falade, Uzo-Peters, Prof A. A. Olapade, Dr O. Ezekiel, Dr Arueya, DrDr. I. Uzo-Peters, Dr F. Adeboyejo, and Dr. A. Oladunjoye of the Department of Food Technology for their immense contribution toward the success of this work. Dr. A. O. T. Ashafa of the University of Free state, Qwaqwa campus, South Africa would forever be remembered and appreciated for hosting me in his laboratory to carry out a sizeable part of this work. I also appreciate Dr. Fatai Balogun and Dr. Ramganesh for the laboratory assistance granted to me at the Department of Plant Science laboratory, University of Free State and Department of Molecular biology, UNISA, South Africa. The unforgetTable input and encouragement of Prof J. O. Babalola (UI), Prof A. B. Saba (UI), Prof J. O. Olaomi (UNISA), Prof. A. L. Toriola (Tshwane Unitech, Pretoria), Dr A. A. Oyagbemi (UI), Mr. K. O. Jimoh, Dr (Mrs) A. O. Alabi (TOPS) and Mr. J. O. Babalola (TOPS) are well appreciated.

My ever caring mother, Mrs. Elizabeth Osunrinade gave her all to the success of this work, I am grateful ma. To all my brothers (Oluyinka, Olugbenga, Olukunle and Olufunso Osunrinade, Abiola, Olutope and Banji Onifade) and wife, your labour and sacrifices to see me attain this height will forever linger on my mind. Thank you. Olubukola Ifeoluwa Osunrinade who has been a caring niece aided this work in no little way. My enduring and lovely wife, Olubukola D. Osunrinade nee Onifade, and my sons Oladele and Iseoluwa Osunrinade who believed so much in me, greatly pushed me to this success, God bless you. To Rev (Mrs) F.J. Saba who gave significant support, I am grateful.

I received impeccable encouragement during this work from Pst S.B. Ipinsokan, Dr. U. C. Onuoha, Mr. Adeleke Ishola, and all members of the Saviour's House of Glory Ministries, thank you all.

ABSTRACT

Ofada rice(*Oryza sativa*, variety OS-6**)** is an indigenous rice variety highly sought mostly for its flavour. However, there are subjective noticeable variations in these flavour attributes which sometimes cast doubt on the rice quality. Profiling of rice flavour compounds and their formation during processing is sparsely documented. Hence, this study was to identify and characterise flavour compounds in Ofada rice as affected by paddy pre-treatment process.

The D-optimal mixture design of steeping time (1, 3 and 5 days), initial steeping temperature (30, 65 and 100^{o}C), parboiling temperature (80, 100 and 120^{o}C) and drying temperature (30, 50 and 70^{o}C) as pre-treatment variables were adopted to condition Ofada rice paddy sourced from Ogun State Agricultural Development Program (OGADEP). The pre-treated 25 samples were processed into parboiled rice using standard procedures. The pH and total Titratable Acidity (TTA) of the fermenting steep water were determined, and bacterial type identified by standard methods. Proximate composition, amylose, Free Fatty Acid (FFA), flavonoid, Total Phenolic Content (TPC), Total Antioxidant Capacity (TAC), Ferric Reducing Antioxidant Power (FRAP) and 1,1-diphenyl-2-picrylhydrazyl radical scavenging assay (DPPH) of the processed rice were determined by standard procedures. Amino and organic acids in processed rice were analysed by liquid chromatography-mass spectrometry,while rice extracts from solvent extraction method were analysed using gas chromatography-mass spectrometry.Parboiled rice was cooked and evaluated using 25-member panelists. Data were analysed using descriptive statistics and ANOVA at $\alpha_{0.05}$.

The pH and TTAranged from 4.9 to 6.9, and 0.1 to 0.4 mg/mL, respectively. Predominant bacteria in the fermenting steep water were proteobacteria (*Acinetobacter*, *Azotobacter*, *Pseudomonas*, *Enterobacter* and *Citrobacter*) and firmicutes(*Lactobacillus, Bacillus, Paenibacillus, Brevibacillus* and *Aneurinibacillus*). Moisture, ash, fat, protein, crude fibre, carbohydrate, amylose contents and FFA of the rice were 4.2-10.2, 0.2-0.7, 0.6-1.7, 7.4-9.5, 0.6-1.7, 78.3-84.6, 17.4-22.3, and 0.7-3.8%, respectively. Flavonoid (0.6-1.5 mgQuercetin/g), TPC (25.4-62.6 mg GAE/g), TAC (0.5-3.9 mgGAE/g), FRAP (19.0-33.4 mgTrolox/g) and DPPH (277.8-1372.7 µg/mL) were obtained. Glutamic acid (8.5–26.1%), alanine (7.4-23.6%), proline (2.4-18.8%), asparagine (2.2-4.8%), aspartic acid (1.3–4.1%), threonine (1.2-3.6%), methionine(0.6-3.2%), serine (0.8-2.1%) and glycine (0.6-1.9%) were the major amino acids detected. Organic acids identified were 2-aminobutyric, nicotinic, pantothenic, pyruvic, lactic, citric, succinic and fumaric acids. The samples extracts contained 30 alcohols, 28 aldehydes, 50 hydrocarbons, 13 nitrogenous compounds, 36 ketones, 33 esters, 15heterocyclics, and 12 phenols. Aroma impacting compounds [pentanoic acid (green), 2-methyl-butanoic acid (cheese-like), butanoic acid (rancid), 1-octanol (metallic), decanal (citrusy), hexanal (fatty), 2-octenal (nutty), 4-methyl-2-heptanone (spicy) and 2-methoxy-4-vinylphenol (clove-like)] were detected mostly among samples steeped for five days.Only initial steeping temperature and duration significantly influenced composition of the compounds.Rice produced from paddy steeped for a day at 30^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 50^{o}C was most acceptable to the panelists.

Flavourants identified in Ofada rice predominantly contained fatty, metallic, clove-like, citrusy, nutty and green flavour notes which varied with steeping treatments. Ofada paddy rice should not be steeped beyond a day for flavourants acceptability.

Keywords: Ofada rice, Paddy steeping, Rice processing, Flavourants

Word count: 493

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CHAPTER 1

INTRODUCTION

1.1 Background

Rice variety and differences in processing methods are among the most important factors that influence rice quality and acceptability. Although rice is produced in most ecological zones of Nigeria, there are differences in the adopted processing methods (Sanni *et al.*, 2005). Rice grain quality is a complex characteristic having many components as yardsticks for its acceptability (Rabiei, 2004). Rice flavour is one of the factors that influence preference for either local or foreign rice consumption in Nigeria. Rice is a major product of people's diet in many countries, the eating and cooking quality is of utmost consideration in the export market and consumer acceptance (Abesekara, 2008). Abeywickrama *et al.* (2010) opined that achievement of the better intrinsic quality of rice may be a challenge in the next decade and also an important factor in consumer acceptability.

It is a well-accepted fact that rice is a popular staple food, consumed by a majority of people in the world (Singh *et al.*, 2005; Cai *et al.*, 2011; Thomas *et al.*, 2013). The global demand for rice as food is about 477.77 million tonnes with Nigeria still among the top three rice importing countries (Statista, 2019). Abulude (2004) identified *Oryza sativa* and *Oryza glabberima* as the two rice varieties mostly processed in Nigeria. In general, the Japanese prefer short-grain, sticky rice that is usually used in making sushi. Conversely, in India, Pakistan, and the Middle East, Basmati rice is well-liked due to its fragrance and its elongated, dry grains when cooked (Suwannaporn and Linnemann, 2007). In Nigeria, an affinity for Ofada rice is primarily due to its unique flavour, but consumers are sometimes put off by the inconsistencies in flavour and the off flavour present. This could be attributed mainly to the processing method adopted (Adeniran *et al.*, 2012). Hot water soaking of rice paddy was reported to have eliminated undesireable fermented odour in milled rice (Anuonye *et al.,* 2016). All over the world, rice varieties with aroma quality known as aromatic or fragrant rice have earned a reputation and wide popularity.

The aroma of both aromatic and non-aromatic rice cultivars consists of a complex mixture of odour-active compounds. Aromatic rice has a natural nutty, popcorn flavour and accounts for 86% of imports into the United States (USDA Economic Research Service, 2010). Jasmine, Basmati, and ‘‘Della’’ type aromatic cultivars are distinguished by their grain shape, cooked rice texture, and flavour (Bergman, Bhattacharya and Ohtsubo, 2004), Genetics, growing conditions, and post-harvest handling are factors that have been shown to affect the aroma and flavour of rice (Champagne, 2008). Rice volatiles has been intensively studied as they are important aspects of consumer acceptance (Widjaja, Craske, and Wootton, 1996; Lam and Proctor, 2003; Wongpornchai, Dumri, Jongkaewwattana and Siri, 2004; Yoshihashi *et al.*, 2005).

Volatile compounds in rice have been studied especially due to their typical flavour (Liyanaarachchi*et al.*, 2014). Volatiles in rice could have both positive and negative influences on taste. A major focus has been on 2-acetyl-1-pyrroline (2-AP), the primary volatile compound in aromatic rice. However, lipid oxidation products have also been identified, which are known to have a negative impact on rice acceptability (Champagne, 2008). Lipid oxidation products are compounds that may become more prevalent in rice flavour with the length of storage time or due to poor post-harvest handling. The desireable characteristic taste of rice could be linked to flavour and fragrance components such as 2AP and aromatic alcohols, while the negative effect could be off-flavours of hexanal and 2-pentylfuran amongst others.

Heinio (2003) reported that the processed grain flavour is related to the specific grain composition.The presence of certain aldehydes, alcohols, and ketone which are compounds known to be flavour-active, are among the determinants of perceived flavour in grains. During processing, there are important flavour precursors which include fatty acids, amino acids, and phenolic compounds. These precursors have been reported to be responsible for flavour formation (Heiniö, 2003). Small peptides, free amino acids, sugars, phenolic compounds, and volatile compounds have been identified as markers that significantly influence the perceived flavour in the rice. Lipids have been found to indirectly influence rice flavour due to their oxidation which leads to the production of hydroxy fatty acids or hydrolytic oxidation of lipids which are lipase-catalysed to produce free fatty acids.

The flavour of cereal products is largely influenced by acids (amino and organic), as well as phenolic components that are non-volatile (Dimberg *et al.*, 1996; Peterson, 2001). Ferulic acid, which is a phenolic acid that influences flavour is abundantly present in cereals. Whereas, acids such as p-coumaric, sinapic and caffeic are necessary for flavour development (Weidner *et al.*, 1999; Andreasen *et al.*, 2000). Dimberg *et al.,*(1996) noted that despite the low percentage of free phenolic acids in cereals, their presence might have a significant effect on flavour perception.The influence of free amino acids on perceived flavour could be related to their percentages or activity as flavour precursors. For example, when the Maillard reaction takes place at high temperatures, compounds such as pyrroles, furfurals, and pyrazines are flavour active, and they are produced from amino acids (Heiniö, 2003).

Variation of flavours in food has been ascribed to the occurrence of volatile compounds in the product headspace. The molecules of the flavour compounds are sensed by the nasal cavity which has the olfactory receptors. Evaluation of volatile compounds in rice involves the process of collection, concentration, separation, and quantification. For collection and concentration of rice volatiles, methods such as purge and trap, direct solvent extraction, steam distillation-solvent extraction, solid-phase microextraction (SPME), and static headspace have being used (Reineccius, 2006). Although, some of these methods are time-consuming, which require many steps in the preparation of samples, and thus may not be appropriate to analyse large numbers of samples. Efficient identification and characterisation of these volatile compounds require adequate extraction and separation techniques. The choice of extraction technique has been identified to be paramount in volatile compounds recovered in the grain samples.

Studies on thecomposition of the rice volatile fraction identified a large number of components and defining several key-aroma compounds (Bryant and McClung, 2011; Champagne, 2008; Widjaja *et al.*,1996a; Yang *et al.*, 2008b; Zeng, 2008). These include saturated and unsaturated aldehydes, alcohols, and cyclic compounds; in particular hexanal, 1-octen-3-ol and 2-pentylfuran are markers of both quality and ageing, while 2-acetyl pyrroline (2-AP) is one of the aroma quality markers for aromatic rice (Buttery *et al.*,1988; Widjaja *et al.*, 1996a; Mahatheeranont *et al.*, 2001;

Champagne, 2008; Laguerre *et al.*, 2007). Additional information on the releaseof key-aroma compounds was also obtained from quantitation and its dependence on grain shape and chemical composition. In some Italian rice cultivars investigated, the ratios of Heptanal/1-octen-3-ol and heptanal/octanal were defined as characterising the aroma quality indices of the rice samples (Alessandra *et al.*, 2015).

However, using experimental design or other kind of method to incorporate the numerous conditions of pretreatment and processing is critical in identifying variations in flavour componentof rice. Response surface methodology (RSM) has been severally used as an experimental design tool to effectively incorporate all test conditions and also describe the contribution of each test conditions (Meilgaard *et al.*, 1999). The use of RSM enables the generation of composite conditions that enhances the interpretation of results.

1.2 Problem statement

The sensory characteristics of the rice grain is a factor of major consideration in acceptability due to the method of its consumption which could be with or without seasoning. Yau and Liu (1999) noted that slight changes in sensory characteristics, which includes aroma can influence consumers desireability and acceptability. Ofada rice is specially relished because of its characteristics flavour that develops during steeping as a result of the fermentative activities of some microorganisms (Adeniran *et al.*, 2012). The major criteria for preference among rice consumers have been reported to be aroma and flavour (Del Mundo and Juliano, 1981). Local rice in Nigeria are often preferred to the imported rice due to their flavour and nutritional value. However observable inconsistency in processing methods has made local rice flavour quality to be unpredictable. Although rice has been the subject of much investigation in the area of basic production in Nigeria where several studies on the effect of processing condition on rice quality has been carried out. However, profiling of flavour compounds responsible for variation in acceptability of Ofada rice has not beenwell documented. Thorough identification and characterisation of the volatile components in Ofada rice and assessment of the impact of the processing condition on flavour component are required to improve the quality of Ofada rice.

1.3 Justification

There has been much focus on optimising the processing condition of Ofada rice. But most of this work have not taken into cognizance the contributory effect it has on its unique flavour. There is a need to ascertain factors that could be responsible for the relished Ofada rice flavour and also identify compounds responsible for the off flavour which often determines the level of consumer's acceptance. According to NCRI and WARDA (2007), it is easy to obtain high quality grains if appropriate steps are followed from harvesting to marketing. The acceptability of local rice in Nigeria is largely dependent on the standardisation of the flavour components. Consistent rice flavour could be obtained with proper research in studying the processing effect of steeping, parboiling/steaming, and drying.

1.4 Aim and objectives

This research was aimed at identifying and characterising flavour compounds associated with Ofada rice. Specific objectives include:

a) To study bacteria growth dynamics during steeping of Ofada rice paddy

b) To determine the relationship between antioxidant properties and Ofada flavour characteristics at varying steeping, parboiling, and drying conditions.

c) To determine the effect of rice paddy pre-treatment on amino and organic acids.

d) To determine the effect of rice paddy pre-treatment on aroma compounds using gas chromatography mass spectrometry.

e) To determine the effect of paddy pre-treatment on sensory characteristics of Ofada rice.

CHAPTER 2
LITERATURE REVIEW

2.1 Importance of rice

Rice is cultivated widely in most nations of the world. This made rice to be among the most important cereals of the world (Adeniran *et al*., 2012). As noted by Itani *et al.* (2002), the world's number one human food crop in ranking is rice. Over three billion; which is more than half of the world's population have rice as staple food (Central and Reeves, 2002; Ebuehi and Oyewole, 2007). Rice is a cereal belonging to the *Poaceae* or tufted grass family (Kassali *et al*., 2010); contains carbohydrates and proteins as the major constituents while lipids, minerals, sugars and free amino acids (FAAs) are present in low amounts (Verma and Srivastav, 2017). Oko *et al*. (2012) reported that rice grain is made up of 75-80 % starch and about 7-10 % protein. Sotelo *et al*. (1994) obtained the range of rice fat to be between 0.50 to 2.23 % while the ash content is between 0.55 to 0.78 %. Generally, milled rice has been reported to be rich in dietary energy, low in fat content, a veriTable source of thiamine, riboflavin and niacin together with a protein with high digestability compared to other cereals (Yousaf, 1992).

Rice is consumed mostly as intact grains after the removal of the hulls, bran and germ. Its mode of preparation which could be boiled or ground and eating with stew or soup makes its consumption to be important at the level of the household. Although consumer's preferences vary from region to region, the majority of consumers prefer well milled or white rice, that contain little or no bran on the endosperm (Lyon *et al*., 1999). Rice for consumption is in the form of white or milled rice produced by a combination ofdehulling and milling processes purposely for hull and bran layers removal of the rough rice kernel (paddy).

The importance of rice in food security, uses at social events and its nutritional tendencies enhances employment andincome, hence it helps in poverty alleviation

and thus making rice to be an economic crop (Marshell and Wadsworth, 1993; USAID, 2009). Rice economic importance is evident at the levels of production, processing, marketing, food vending and importation/exportation (USAID, 2009). Although rice is grown in almost all the ecological zone in Nigeria, however, it has been reported that demand for rice has always been far above the level of domestic production (Ologbon *et al*., 2012). This could be due to the fact that rice production in Nigeria is mostly by the small-medium scale processor. Being a major cereal grain, evaluating the nutritional and cooking qualities of rice has been given highest priority (Tan *et al*., 1999; FAO, 2004; Jiang *et al*., 2005; Dong *et al*., 2007). This is due to cushioning effect it has for under-nutrition and severe hunger among many Nigerian households; as it is commonly eaten in many localities and processed into different forms. Consumers' preference varies based on the type of rice and their origin (Azabagaoglum *et al*., 2009; Musa *et al*., 2011).

2.2 Rice variety and characteristics

Rice has a great economic importance as it serve as a source of income and a major staple food in most Nigerian homes. There are several varieties of rice and each variety varies in their cooking and sensory characteristics. Sanni *et al*., (2005) reported that rice grown in different ecology zones varies in their properties. About 25 species of the *Oryza* genus have been reported to be cultivated in both tropical and sub-tropical part of Asia, Africa, China, Northern Australia and South America. *Oryza Sativa* and *Oryza glabberima* are the most commonly cultivated rice varieties in Nigeria (Abulude, 2004). The WARDA's hybrids rice is also gaining acceptance among rice farmers in Nigeria (Adekoyeni, 2014). The sub species of Oryza sativa included indica (long grain rice), japonica (short grain rice high in amylopectin) and javanica (broad grain grown in the tropics) (Juliano, 1993). However, the javanica sub species are off-shoot of japonica rice (IRRI, 2007). These sub species vary in their cooking and processing properties (Bao and Bergman, 2004). Hizukuri *et al*.,(1989) observed the slow nature of retrogradation of short and wide japonica rice which is also reported to be sticky and cook soft.

Variation in the percentage of starch in rice species majorly distinguishes their characteristics. Milled rice was reported to have about 90% starch. And this has made rice varieties to be classified base on their amylose content as high (25 - 30 %),

intermediate (20 - 25 %), low (10 - 20 %), very low (2 - 9 %) and waxy (1 - 2 %)(Bao and Bergman, 2004). It has been reported that variations in composition and cooking quality of rice is mainly dependent on the genetic as well as surrounding environmental factors where they are grown (Giri and Vijaya, 2000; Singh *et al*., 2005).

There are numerous physicochemical properties, which confers variation in grain quality, cooking behaviour and cooked rice texture on the rice (Bocevska *et al.*, 2009; Moongngarm *et al*., 2010). For example, eating and cooking qualities of rice is dependent on amylose content. Whereas, factors such as gel consistency and gelatinisation temperature could vary base on the variety (Juliano, 1972; Bhattacharjee *et al.,* 2011).While appearance quality is usually represented by the grain size and translucency of the endosperm (Tan *et al*., 1999). It has been reported that soft and sticky cooked texture are associated with low amylose rice while the high amylose rice have hard and flaky texture (Juliano, 1985).

Rice grain size and shape have a direct effect on the marketability and commercial success of improved rice cultivars. South asian, middle east and near east asian countries consumers preferred long and slender grain cultivars (indica type). However, in indica rice consuming countries, there is preference for long grain rice with intermediate gelatinisation temperature. The preference is due to it non sticky, soft and fluffy after cooking properties, whereas short and bold grain cultivars (japonica type) produce sticky and clumped grains after cooking (Hossain *et al*., 2009). Aroma, hardness and roughness depended on temperature, and are variety specific which in turn affects the sensory properties of cooked rice. Genetic variability for such traits exists within the rice gene pool, with remarkable differences between two major subspecies viz. indica and japonica. Quality assessment of rice entails a combination of sensory tests and physicochemical determinations which is determined by physical properties, cooking quality, gelatinisation temperature and chemical composition of cooked rice (Zhou *et al*., 2002).

2.3 Rice Production and Processing in Nigeria

Sowunmi *et al*., (2014) observed that demand for rice had risen steadily while domestic production increased at a much slower rate despite the forecast increase in

demand caused by the expected increase in population by over 50 percent between the years 2000 and 2020. Being the highest importer of rice in Africa, Nigeria rice production and processing is a major part of government strategy to overcome food shortage and reduce importation (Sowunmi *et al.,* 2014).The Nigerian National Rice Development Strategy (NRDS) in 2009 targeted to produce 12.8 million tonnes of paddy rice in 2018 from the 3.4 million tonnes production reported in 2007. Rice being one of the major staple in Nigeria, with 25kg per person per capita consumption annually is produced in all ecological zones in Nigeria with different processing methods (Sanni *et al*., 2005). In Nigeria, the cultivated species of rice are *Oryza glaberrima Steud* and *Oryza sativa L*. (Adeyemi *et al*., 1986; Abulude, 2004).

Breeding efforts led to the introduction of several varieties of *Oryza sativa L.,* among which was OS 6 released as FARO 11.Other varieties released through breeding include ITA 150 and NERICA 1 named as FARO 46 and FARO 55 respectively. All these varieties are in cultivation in the South West region of Nigeria. Some other 14 varieties released by WARDA and NCRI are NERICA 1, NERICA 3, NERICA 5, NERICA 8, WAB 450-1-B-P-180-HB, WAB 189-B-B-B-8-HB, WAB33-25, WAB 450-24-3-2-P18-HB, ITA 150, ITA 301, ITA 117, ITA 321, OS 6 and WAB 706-3-4-K4-KB. Seven varieties (NERICA 1 (FARO 55), WAB 189-B-B-B-8-HB (FARO54), ITA 150 (FARO 46), ITA 301 (FARO 48), ITA 117 (FARO 47), ITA 321(FARO 53) and OS 6 (FARO 11)) had since been released for commercial cultivation (NCRI and WARDA, 2007).

Upland rain-fed, lowland rain-fed and irrigated production is used for both local and improved domestic rice production in Nigeria (Daramola, 2005). And this accounted for 97% of rice production in Nigeria. Gboko, Abakaliki, Mokwa and Ofada rice are the major local varieties grown in Nigeria. Faro series, Nerica 8 and ITA series are the three improved domestic varieties grown along the local counterparts (Sowunmi *et al.*, 2014).

Ofada rice variety OS6 as reported by NCRI and WARDA (2007) isamong the earliest released *Ofada* rice. A short grain robust rice was used for the description of the variety. Generally, rice cultivated and processed in the South-West geopolitical zone of Nigeria is often refered to as Ofada (Adekoyeni *et al.*, 2012; NCRI and ARC,

2005; Longtau, 2003). However, production among several households in Obafemi Owode LGA in Ogun state, Nigeria made the community to be the largest producer of Ofada rice (NCRI and ARC, 2005). The popularity of this rice variety has put the name of the local government on the African map of rice producers (Ologbon *et al*., 2012). Due to its unique taste and aroma, Ofada rice is more popular than other local varieties in Nigeria. Longtau (2003) affirmed that the first area of Asian rice cultivation was Abeokuta, Ogun State. It later spread to Lagos area in Epe and Okitipupa; from there it moved to Ogoja and Abakaliki provinces after the Second World War. It then spread across the sahara and to northern Nigeria via the oases and the Trans-Saharan trade.

In Nigeria, considering that different varieties of rice are produced in differenet geo-political zone, demand for imported rice is still on the rising mostly because of variation in the processing methods that impact negatively on its quality (Ebuehi and Oyewole, 2007). However, consumption of Ofada rice and other local rice is largely depended on the natural and local taste of the rice (Eme and Nathan, 2007). Sowunmi *et al*., (2014) in a study of the consumer perception of Ofada rice affirmed the dependence of consumers' acceptance of Ofada rice on its quality.

Daramola (2005) opined that the major challenge in rice processing in Nigeria could be attributed to inefficient processing technology with respect to steeping, parboiling and drying method; which gives local rice its undesirable characteristics. Quality defects such as off flavours, presence of stones, uneven grains, and non-uniformity in quality even with the same variety is a product of variation in the processing conditions. Bhattahcarya and Subbarrao (1996) reported the undesirable odour imparted on rice due to its paddy steeping method. Efficiency of rice parboiling and drying has been reported to be a determinant of the technical performance of milling which in turn would affect the milled rice quality.

Otegbayo *et al*., (2001) reported inadequacies in Ofada rice processing which are mostly based on crude post-harvest practices by local processors. Eme and Nathan (2007) reported south west Nigeria as the prevalent Ofada rice producer and consumer. However, improvement and standardisation of paddy processing to

produce consistent high quality rice would accord Ofada rice an international recognition.

Paddy rice processing could be classified into non-hydrothermal (non-parboil) and hydrothermal (Parboil) methods (Patindol *et al*., 2008). Non-hydrothermal method is by direct milling of paddy rice for the production of milled rice and this will require equilibrating paddy moisture to between 18 - 22 %. This is often achieved by artificial heated air or with the natural sunshine in places where the suiTable drying technology is not available (Daniels *et al*., 1998). On the other hand, parboiling of rice paddy which is majorly done to improve rice quality is known as the majorly consumed processed rice in Nigeria (Otegbayo *et al*., 2001). Its market in the industrialized nations has also been on a yearly increase (Patindol *et al*., 2008). "Ofada" rice is processed traditionally by parboiling method that involves three stages of moisture treatment (steeping, steaming and drying) as presented in Figure 2.2.

Four critical stages are involved in parboiling operation of rice; these are steeping, steaming, drying and milling. Hot, warm or cold water could be used for paddy steeping, and the duration of steeping is usually dependent on the cultural practises of the specific production cluster area (Adekoyeni, 2014). While paddy steeping could be done for eight days is some production area, it can also be done just for some hours in other areas. This leads to the swelling of the grain which is achieved through the penetration of water to the endosperm of paddy which forms hydrates by hydrogen bonding. The process of parboiling hardens the kernel and hence allowing for optimised milling yield. In addition to kernel hardening, rice parboiling also improve the nutritional content of the rice and thus conferring comparatively better health benefits.

Steeping parameters such as steeping time, temperature and moisture content after steeping was reported by Igbeka *et al*., (1991) to influence physical properties of rice such as translucency, colour and deformity in grains. In the work of Ayelogun and Adegboyega (2000), it was observed that steeping rice paddy for too long leads to unacceptable colour, taste, and odour of rice due to fermentation processes. Advances in technology has created more sophisticated method of rice parboiling which includes dry-heat parboiling and pressure parboiling (Bello *et al*., 2004).

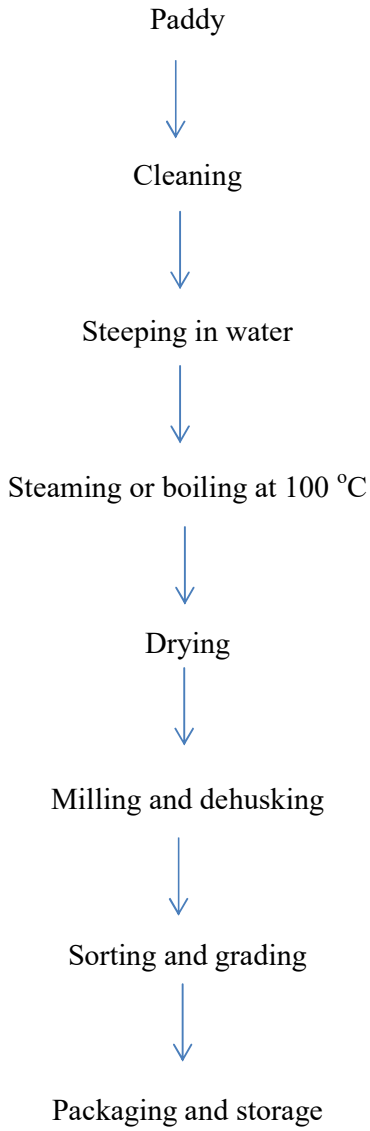

Figure 2.1 : Traditional method of rice prosessing

Source: Vikrant K., Jaivir S., Neelash C., Suresh C., Vivak K. and Yadav M. (2018) Process of paddy parboiling and their effects on rice" A Review. *Journal of Pharmacognosy and Phytochemistry*. SP1: 1727-1734

Cherati *et al*., (2012) stated that parboiling enhances milling yields, nutritional value and resistance of rice to insects and mould. Otegbayo (2001) reported that steeping contributes mainly to organoleptic, physical and nutritional changes in rice. Bacteria, yeast and moulds being mainly responsible for the organoleptic properties. Production of flavour compounds and aroma are made possible through fermentation processes carried out by yeasts and lactic acid bacteria (Omemu, 2007). The presence of fermented odour in Ofada rice as reported by Anuonye *et al.,* (2016) was successfully removed by adopting hot water steeping method. In the work of Adeniran *et al.* (2012), use of starter cultures reduced the steeping time of "Ofada" rice and it also enhanced the nutrient composition and improved the sensory quality of the rice. This showed the role being played by microorganism in defining the Ofada rice flavour.

Adeniran *et al.,*(2012) noted that Ofada rice is specially relished because of its characteristics flavour that develops during steeping as a result of the fermentative activities of some microorganisms. It has been reported that aside the changes in composition and distribution of nutrients in the rice kernel, Steeping also causes the leaching of rice constituents into the steep water (Otegbayo *et al*., 2001; Ibukun, 2008; Sareepuang *et al*., 2008). This leads to decrease in starch content of rice due to leaching of starch granules (Sareepuang *et al.*, 2008). Hot water steeping has also been reported to make rice undergoes gelatinisation which alters its physical, chemical, nutritional, rheological and viscosity properties (Sareepuang *et al.*, 2008; Mir and Bosco, 2013).

Development in drying method over the traditional sun drying method of drying is the use of mechanical driers (IDRC, 1976). Sun drying method aside being labour intensive, could lead to delay in drying due to its dependent on the prevalent weather condition. Contamination with foreign materials, exposure to bird and uneven drying has been associated with the traditional drying methods (Bhattacharya and Alli, 1990). Drying temperature is of major importance in the use of mechanical driers, because forcing heated air through parboiled rice could also lead to losses of quality. As such, high quality rice are produced when drying is carried out at low drying temperature (Adeyemi *et al.*, 1986).

2.4 Rice Flavour

The fragrance of rice grain and the flavour of cooked rice are important quality factors that influence consumer acceptability. Rice (*Oryza sativa L.*) is enjoyed by many people as a staple food because of its flavour and texture. The demand for fragrant rice varieties has increased in recent years in countries that consume rice as a major food. This is evident in the willingness of consumers to pay a higher price for it (Liyanaarachchi*et al*., 2014). In order to increase the choice of rice available in Nigeria, effective study to characterise flavour associated with processing variation is paramount. According to Laing and Jinks (1996), food flavour is commonly defined as being the sensation emanating from the combination of signals produced due to sensing smell, taste, and irritating stimuli from a food product.

Generally, a combination of taste and aroma characteristics are refered to as flavour. The sense of taste and gustation impressions perceived by taste bud stimuli are sweetness, saltiness, bitterness,sournessand umami (delicious or savoury). Whereas, perception of the stimulation of the olfactory epithelium by volatiles(odourants) through the retronasal or the orthonasal is refered to as Aroma or odour (Roudot-Algaron, 1996; Hummel, 2008; Pszczola, 2004). As such, little variations in sensory characteristics mostly aroma influence consumers judgement about the desireability and unacceptability of rice (Yau and Liu, 1999).Therefore, consumers rated aroma and taste as an important determinant of acceptance during rice consumption (Del Mundo and Juliano, 1981).

Unlike other grain that passes through numerous processes before comsuption, rice is cooked and consumed as a whole grain. This makes consumers to desire aromatic rice compared to non-aromatic rice. Bryant and McClung (2011) reported consumer preference for scented rice due to their distinctive aroma and flavour. Several reports on the volatile compounds of rice have been studied due to their typical flavour and odour. Aromatic rice has a natural nutty, popcorn flavour and accounts for 86% of imports into the United States (USDA Economic Research Service, 2010). Jasmine rice from Thailand and basmati rice from Pakistan and India have been identified as the main sources of aromatic imports. However, Aside variation in flavor due to processing, breeding process of researchers have develop aromatic rice cultivars that

can be grown and favourably compete with Jasmine and basmati rice (Bergman, Bhattacharya and Ohtsubo, 2004).

Studies have demonstrated that consumers can discern sensory attributes that distinguish different types of aromatic rice cultivars during preference tests (Fitzgerald and Hall, 2008; Fitzgerald, McCouch, and Hall, 2009). 2-acetyl-1-pyrroline (2-AP)has been reported to be a primary determinant of flavour in aromatic rice (Buttery, Ling and Juliano, 1982). It is the only flavour component that breeders have the ability to manipulate its production in rice. Genetics, growing conditions, and post-harvest handling are factors which have been shown to affect the aroma and flavour of rice (Champagne, 2008).

There are several studies that have evaluated a number of rice cultivars from different genetic backgrounds and the results showed a significant qualitative and quantitative variation in flavor compounds (Bergman *et al*., 2000; Laguerre *et al.*, 2007; Yang *et al*., 2008a; Zeng *et al.*, 2008). Rice storage duration and variation in storage condition have been reported to lead to depletion of desirable aroma compounds and production of undesirable aroma compounds (Widjaja, Craske, and Wootton, 1996a; Suzuki *et al*., 1999; Zhou, Robards, Helliwell, and Blanchard, 2002; Wongpornchai, Dumri, Jongkaewwattana, and Siri, 2004; Tulyathan, Srisupattarawanich, and Suwanagul, 2008). Although, in the study of rice aroma, major focus has been on 2-AP, however, lipid oxidation products have also been identified to be significant in rice aroma as they have negative impact on acceptability (Champagne, 2008).

For more than thirty years, numerous research has been carried out to establishhow genetic, pre-harvest (environment, cultural methods) and post-harvest (Steeping, steaming, drying, milling, storage and cooking method) conditions influences the cooked rice aroma and flavor.Some of these conditions have been assessed to know their effects on volatile compounds detected in rice.The numerous studies were centered on knowing important compounds that will enable rice processors to monitor and control the pre-harvest and postharvest operations such that desired rice flavor characteristcs can be obtained (Champagne, 2009).

Studies on rice flavor reported the dependence of changes in rice flavours to the presence of volatile compounds which can be attributed to preharvest and postharvest operations. The use of concentration or aroma value (AV) in determining the influence of volatiles has been reported. Preference or descriptive sensory evaluation has also been used by some reseachers together with volatile analysis. Aside the use of 2-acetyl-1-pyrroline (popcorn aroma) as a marker compound, no other volatile compound has been reported to be adequate for monitoring and control of pre-harvest and post-harvest conditions that influences aroma and flavor (Champagne, 2008).

2.5 Aromatic Rice

Rice can be classified as aromatic and non-aromatic rice on the basis of aroma. Aromatic rice emits a special aroma with cooking and while eating. Example of internationally known aromatic rice are Jasmine rice from Thailand, Della from USA, IAC 600 from Japan and basmati rice from Pakistan and India, (Bisne and Sarawgi, 2008; Bryant and McClung, 2011). Studies have demonstrated that consumers can discern sensory attributes that distinguish these types of aromatic rice cultivars in preference tests (Fitzgerald and Hall, 2008; Fitzgerald, McCouch, and Hall, 2009). Traditionally, many varieties of aromatic and non-aromatic rice are grown by farmers in all areas. The aromatic rice varieties are sold at a premium price in local and international markets because of their superior grain quality and pleasant aroma (Nayak *et al*., 2003; Verma and Srivastav, 2015). Aroma is rated the highest desired trait followed by taste and elongation after cooking by Indian consumers (Bhattacharjee *et al.*, 2002). At a time Asian consumersliving in the United States considered appearance and aroma as the most essential acceptance factors of cooked rice (Meullenet *et al.*, 2000).

The volatile aroma component of aromatic rice is a popcorn-like flavour compound (Buttery *et al.*, 1983). This is considered as the single most critical quality trait in rice preferred by many consumers (Suwansri *et al.*, 2002). This flavour compound is stemming primarily from its 2-acetyl-1-pyrroline content (Bhattacharjee *et al.*, 2002), which generally plays a role in consumer acceptability of rice (Bergman *et al.*, 2000). This aromatic compound also contributes to the ‘roasted aroma’ reported in different food products viz. crusts of wheat and rye breads (Schieberle and Grosch, 1985), wetted ground pearl millets (Seitz *et al.*, 1994), popcorn (Schieberle, 1991), cooked

beef etc. In aromatic rice, flavour is seen as one of the most significant factors in market business, which distinguishes aromatic rice from ordinary rice (Givianrad, 2012).

In the world market of rice, basmati and Thai Jasmine are the two major rice types containing 2AP. However, Yasumatsu *et al*., (1996) had found volatile carbonyl compounds such as acetaldehyde, propanol, 2-butanone, pentanal and hexanol in fragrant rice varieties. More than 100 volatile components have been identified in cooked rice (Yajima *et al*., 1979; Tsugitha *et al*., 1980). Suwandal and Kuruluthuda are popular fragrant traditional rice varieties among commercial growers of rice in Sri lanka. The volatiles that contributed to rice variety aroma are aldehydes, ketones, alcohols and heterocyclic compounds (Liyanaarachchi *et al*., 2014). In the work of Yang *et al*., (2008) on the characterisation of aromatic volatiles in black rice, 2-AP, guaiacol, indole, and *p*-xylene largely influenced the difference between the aroma in cooked black and white rice. However, 2-AP and guaiacol were major contributors to the unique character of black rice based on odor thresholds, relative concentrations and olfactometry.

2.6 Component of Rice Flavour

In general, the genotype, environment and the interactions of this factors causes significant variation in the grain chemical composition. These factors may significantly influence the final flavour of rice directly or indirectly. For example, amino acids which determine the protein content of rice is largely dependent on the nitrogen content of the soil. Peculiarity of harvesting and post harvesting operations together with other grain treatment before serves as a major influence to grain composition. The amount of protein and fat has been reported to be determined mostly by the production season.

The amount of protein, carbohydrate or starch, fat, ash and fibre found in a cereal grain is largely dependent on the grain variety. Welch(1995) reported that oat is composed of approximately 11% protein, 5% fat and 59% carbohydrate content whereas rye had 12% protein, 3% fat and 63% carbohydrate content (Shewry and Bechtel, 2001).

As presented in Figure 2.2, free amino acids, sugars, small peptides, volatile and phenolic compounds of a grain may have significant effect on the perceived flavour in food. Also, the indirect effect of lipids on flavour could be linked to both oxidation and lipid catalysed hydrolytic oxidation of lipids to produce hydroxy fatty acids and free fatty acids respectively.

Variation in perceived flavour due to the composition of a grain could be observed in two ways. The presence of flavour-active component which include alcohols, ketones and aldehyde in the grain matrix is a first major effect of grain composition (Hansen, 1995).On the other hand, the presence of flavourprecursors (fatty acids, amino acids and phenolic compounds) which are required for flavour production during processing is the second way through which perceived flavour in grains varies (Hansen, 1995).

2.6.1 Volatiles

The volatile profile of a rice variety is important not only to use in rice breeding programmes, but also to assure the quality of whole grain or grain products in the market. Most of the volatiles that are produced through metabolic pathways are dependent on the variety, agronomic practices, storage conditions and post-harvest handling among other factors. Therefore, volatile profiles may have the potential to mark the identity of the variety and to interpret the quality of rice. Volatile profiles of rice varieties is analysed so that they can be used in breeding programmes for grain quality improvement and also in quality assurance studies. Difference in the rice volatile profiles has been a major factor for differentiating composition of aromatic rice from non-aromatic rice. (Liyanaarachchi *et al*., 2014).

Rice aroma and flavor are determined by the presence of numerous volatile and non-volatile compounds. However, among the over 200 volatiles detected in rice, only a few has been reported to be responsible for cooked rice aroma and flavour (Champagne, 2008). One major characteristic flavour from aromatic rice has been linked to the presence of 2-AP (popcorn aroma), whereas, there are no other flavour compound that is definitive to give a particular flavour characteristics with respect to fragrant rice but rather a synergistic effect of numerous volatiles.

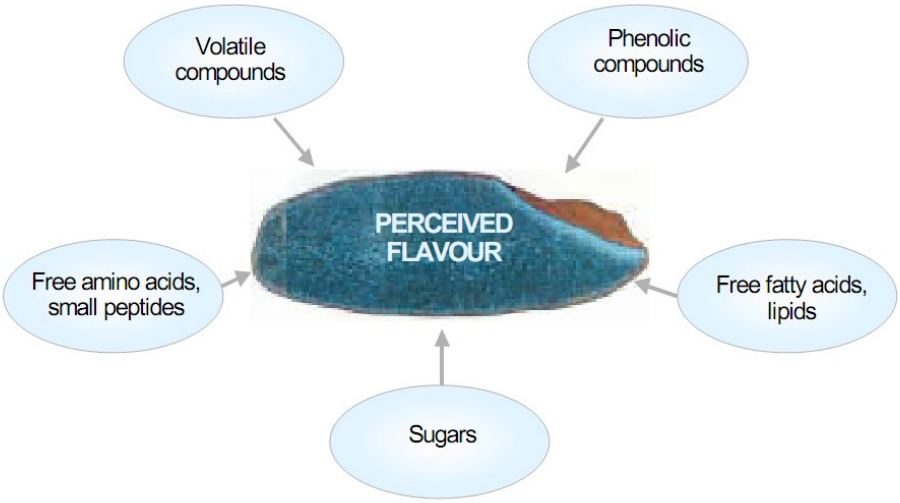

Figure 2.2.Components of grain determining perceived flavour

In human, perception of rice aroma is through the hair like millions of tiny cilia which serves as a covering for the epithelium and located at the highest point of the nasal cavity. Flavour detection occurs when volatile compounds move through the nasal passage (Meilgaard *et al*., 1999). The volatile profile of rice has been explored by several investigators, some of whom have also determined a corresponding aroma using gas chromatography (GC)/mass spectroscopy (MS) olfactometry for most of the compounds detected (Jezussek, Juliano, and Schieberle, 2002; Widjaja, Craske, and Wootton, 1996; Yang, Shewfelt, Lee and Keys, 2008; Zeng *et al*., 2008). As a result, more than 200 volatile compounds have been identified in rice by different reseachers (Bergman *et al*., 2000; Champagne, 2008; Widjaja *et al*., 1996a, 1996b; Yajima, Yani, Nakamura, Sakakibara, and Hayashi, 1979; Zeng *et al*., 2008), some of which, e.g. 2-AP, 2-acetyl-pyrrole, a-pyrrolidone, and pyridine have been identified as enhancing the consumer acceptability of rice, while other compounds, e.g. lipid oxidation products, such as hexanal, acetic acid, and pentanoic acid, can have a negative influence on acceptability. Fragrant rice known with the popcorn aroma was associated with the presence of 2-AP as the marker volatile compound(Buttery *et al*., 1983).

Lorieux *et al*., (1996) and Bradbury *et al.,*(2005) reported the unique potential in the gene of fragrant rice to store 2-AP. In the work of Hussain *et al.,* (1987) on differentiating the rice volatiles; a non-fragrant rice variety and aromatic basmati rice, it was reported that hexanol, pentadecan-2-one, and 2-pentylfuran were absent in non-fragrant rice but present in the basmati rice. In another work to differentiate between aromatic and non-aromatic rice by Lorieux *et al.* (1996), it was reported that 2-AP, 6, 10, 14-trimethyl-pentadecan-2-one,pentanol, (E)-hept-2-enal,hexanol, benzaldehyde, octanal, pentadecan-2-one, and hexadecanol were the nine compounds that serves as variation markers.The study of Widjaja *et al.,* (1996) reported oxidation products as markers and noted that non-aromatic rice has higher percentage of n-hexanal, l-octen-3-ol, (E)-2-heptenal, 4-vinylphenol,(E)-2-octenal, *n*-nonanal, (E)-2, (E)-4-decadienal,4-vinylguaiacol and 2-pentylfurancompared to aromatic rices.

Tava and Bocchi (1999) only detected 2-AP and lipid oxidation products as markers for differences in fragrant rice samples.Aside 2-AP and lipid oxidation products, there is no other particular volatile or non-volatile compound that has been reported to be

responsible for the rice flavour characteristics. The volatile compounds in fragrant rice, which provide the characteristic aroma and flavour have been studied by a number of researchers and more than 100 volatile components have been identified in cooked rice (Yajima *et al*., 1979; Tsugita *et al*., 1980; Tsugita *et al*., 1983). Yasumatsu *et al.*(1996) found volatile carbonyl compounds such as acetaldehyde, propanol, 2-butanone, pentanal and hexanol in fragrant rice varieties and Buttery *et al*., (1983) had previously identified 2-acetyl-1-pyrroline (2AP) as the principal fragrant compound associated with aromatic rice. The compound 2AP is present in many folds in *Pandanus* species and sometimes the rice is cooked with a piece of *Pandanuslatifolius* (Rampe) leaf.

2.6.2 Amino Acids

Although rice quality in terms of nutrition is valuable for its protein content and the balance of essential amino acids, however, free amino acids (FAAs) may be responsible for the perceived flavour or act as precursors for flavour production. FAA profile has been successfully used for discrimination of variety and origin of natural foods in food authentication (Kamara *et al.*, 2010; Maro *et al*., 2011; Cometto *et al.*, 2003). In higher plants, amino acids serve as precursors for secondary metabolism. The free amino acids also act as precursor or substrate for several processes. For example acrylamide, which is a carcenogenic compound formed during heating has been discussed in literature to be related to the presence of asparagine (Curtis *et al*., 2010; Postles *et al*., 2016).

Scientific findings revealed that, even though found as minor constituents, FAAs together with soluble sugars play a significant role in deciding the organoleptic properties of food (Kamara *et al.*, 2010; Kasumyan, 2016). Roudot-Algaron (1996) reported the variations of taste profile of some free amino acids as shown in Table 2.1. Branched-chain amino acids (BCAAs), such as Leucine, Isoleucine and valine were reported by Mukai *et al.,*(2007) to be bitter extremely, and their perceived odour is unpleasant. Also among the bitter amino acids are tryptophan, phenylalanine and tyrosine. When compared to its crystal forms, proline, serine and cysteine in solution looses their bitterness (Asao *et al*., 1987). However, Roudot-Algaron (1996) and Asao *et al.,* (1987) observed the sweetness of some bitter L-amino acids D-enantiomers.

Table 2.1: Taste perception and thresholds of amino acids in solutions

Amino acid	Taster Profile	Taste thresholds (mg/mL) (Kato *et al*., 1989)
Valine	slightly sweet,Flat to bitter	0.4 (bitter)
Leucine	Flat to bitter	1.9 (bitter)
Isoleucine	Flat to bitter	0.9 (bitter)
Threonine	May be bitter, sour or fatty, flat to sweet,	2.6 (sweet)
Aspartic acid	Flat, sour, slightly bitter	0.0 3(sour);1 (Umami)
Glutamic acid	may be meaty,Salty, bitter	0.05 (sour); 0.3 (umami)
Lysine	Flat, mineral,complex	0.5 (sweet and bitter)
Lysine monohydrichloride	Bittter, complex, salt, sweet	-

Source: Roudot-Algaron, F. (1996). The taste of amino acids, peptides and proteins: example of tasty peptides in casein hydrolysates. *Lait* 76: 313-348.

The reaction offree sugar with small peptides or amino acids at high temperature leads to the formation of heterocyclic pyrazines, furans, pyrroles, and sulphur-containing compounds that are aroma active. Amino acids can only be perceived by gustative nerve due to their non-volatility nature. However, their catabolism leads to the production of several aldehydes, alcohols, esters and acids which are volatiles that can be perceived through the olfactory receptors (Jinap and Hajeb, 2010).

During fermentation, microorganisms break down amino acid to produce different volatiles which also include amines, pyrazines and pyridines (Licthenthaler *et al*., 1997). One of the pathways through which this occurs is the Erhlich's pathway (Figure 2.3) which involves both transamination to an alpha keto acid and a decarboxylation step to produce aldehydes, alcohols and acids (Table 2.2). It has been shown in many models that the deamination/transamination step is very often a limiting step in amino acid catabolism and specially for lactic acid bacteria in dairy products (Yvon *et al.*, 1998).

Maloney *et al*., (2010) noted that aldehydes are also products of amino acids through the anabolic pathway during its synthesis. Many of the aldehydes have fruity or malty flavour (for example isobutanal, 2 methyl butanal, 3-methy butanal) or flowery taste (for example benzene acetaldehyde). Although most of the aldehydes produced are further transformed to their alcohol and acids. The acids obtained are easily esterified and permit the synthesis of series of esters with the alcohols produced by the same metabolism or from the one issued from other pathways.

Several methods have been reported for determination of amino acids in food including rice (Saikusa *et al*., 1994; Kamara *et al*., 2010; Sasaki *et al.,* 2013; Liyanaarachchi *et al*., 2018). viz. gas chromatography (GC)–MS (Calder, Garden, Anderson and Lobley, 1999; Wood, Khan and Moskal, 2006), capillary electrophoresis– mass spectroscopy (CE)–MS (Poinsot, Gavard, Feurer and Couderc, 2010; Soga and Heiger, 2000; Soga, Kakazu, Robert, Tomita andNishioka, 2004), liquid chromatography–mass spectroscopy (LC–MS) (Armstrong, Jonscher and Reisdorph, 2007; Piraud *et al*., 2005) and many other complicated methods.

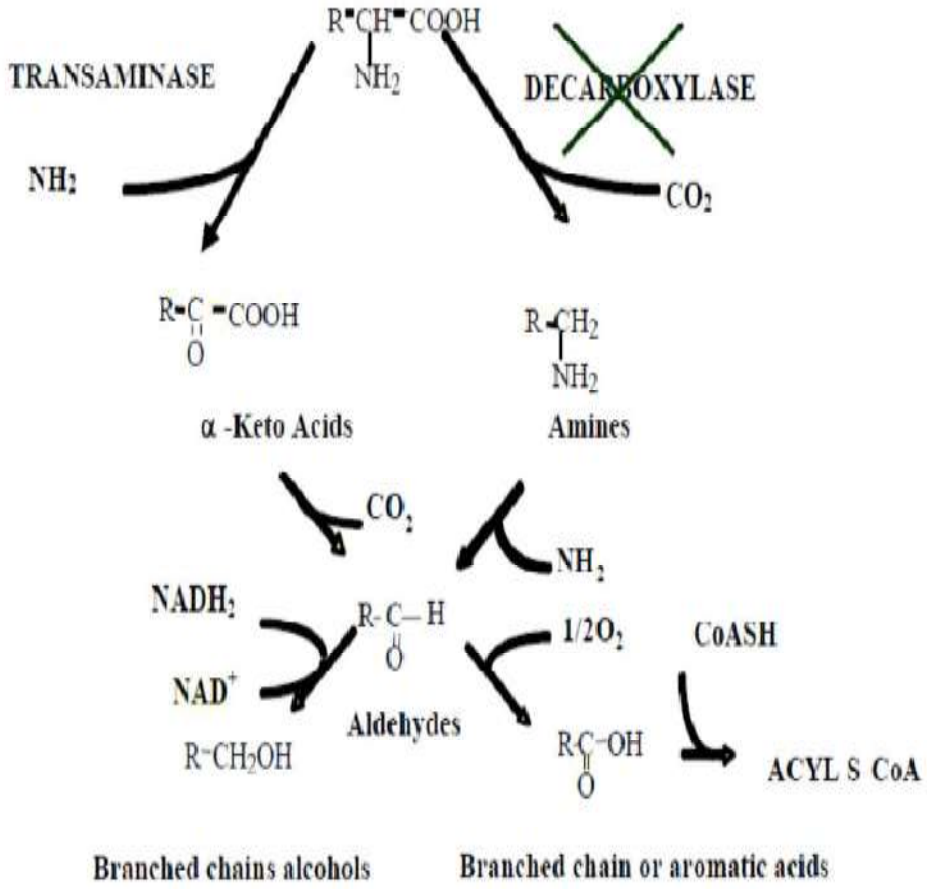

Figure 2.3: The Ehrlich-Neubeur pathway

Source: Spinnler H. E. 2011. Flavours from amino acids.UMR de Génie et Microbiologie des Procédés Alimentaires, AgroParisTech / INRA, 78850 Thiverval-Grignon.DOI: 10.1201/b11187-7

Table 2.2: Volatile compounds from the Ehrlich-Neubuer pathway

Amino acid	Aldehyde	Alcohols	Acids
Leu	3-Methyl butanal	3-Methyl butanol	3-methyl butanoic acid or isovaleric acid
Ileu	2-Methyl butanal	2-Methyl butanol	2-Methyl butanoic acid
Val	Isobutanal	Isobutanol	Isobutanoic acid
Ala	Acetalldehyde	Ethanol	Acetic acid
Meth	Methional or 3-methyl thiopropionaldehyde	MEthional or 3-methylthio-propyl alcohol	3-methyl thio-propionic acid
Met	Methylthio-ethanal	2-methylthioethanol	Methyl thio acetic acid
Cys	MErcapto-ethanal	2-mercapto ethanol	Mercaptoacetic acid
Phe	Phenyl acetaldehyde	2-phenyl ethanol	Phenyl acetic acid
Phe	Benzaldehyde	Benzyl alcohol	Benzoic acid
Glu	4-oxo butyric acid	4- hydroxyl butyric acid or butyrolactone	Butyric diacid
Tyr	(4-hydroxyphenyl) acetaldehyde	2-(4-hydroxyl phenyl) ethanol or tyrosol	(4-hydroxyl phenyl) acetic acid
Try	Indyl acetaldehyde	2-indyl ethanol	Indyl acetic acid

Source : Spinnler(2011)

Spinnler H. E. 2011. Flavours from amino acids.UMR de Génie et Microbiologie des Procédés Alimentaires, AgroParisTech / INRA, 78850 Thiverval-Grignon.DOI: 10.1201/b11187-7

However, analysis of amino acids have been commonly performed by precolumn derivatization with orthophthaldialdehyde and/or 9-fluorenylmethyl chloroformate which is followed by reversed phase high-performance liquid chromatography and fluorescence detection (Blankenship, Krivanek, Ackermann and Cardin, 1989; Einarsson, Josefsson, and Lagerkvis, 1983). GC requires derivatization of amino acids, which has been effectively performed with chloroformates (Husek, 1991; Husek and Sweeley, 1991).

Owing to the high polarity, low volatility and the absence of specific chromophores for ultraviolet (UV) or fluorescence detection, amino acid analysis generally requires a derivatization step which improves the separation and the sensitivity of detection. Pre-column derivatization using either orthophthaldialdehyde (OPA) (Henderson *et al*., 2000; Schwarz *et al.*, 2005; Igor *et al.*, 2013; Liyanaarachchi *et al*., 2018) or 9-fluorenylmethyl chloroformate (FMOC) (Henderson *et al*., 2000; Jambor and Perl, 2009; Schwarz *et al.*, 2005, Igor *et al.*, 2013) reagents are the most common derivatizations found in literature associated with amino acid analysis. However, these techniques generally encounter complexities related to derivatization such as incomplete derivatization, derivative instability, reagent interference, long preparation times, lack of analyte specificity and the hazard associated with the use of potentially toxic derivatizing reagents (Francesca *et al*., 2013; Thiele *et al*., 2012).

Furthermore, the use of costly reagents and buffers, lenghty run times and decreased reproducibility are among the other drawbacks associated with derivatization. Although this method demonstrates good resolution and sensitivity, it requires manual derivatization procedure. It is a laborious method and fails to determine some amino acids (Thiele *et al*., 2008). Several approaches have been made for underivatized FAA analysis using MS/MS detection combining either hydrophilic interaction liquid chromatography (HILIC) (Gao *et al.*, 2016; Konieczna *et al*., 2018, Guo *et al*., 2013) or reversed phase liquid chromatography (RPLC) (Nimbalkar *et al*., 2012, Ozcan and Senyuva, 2006; How *et al*., 2014; Cocuron *et al*., 2017). Studies have reported the use of HILIC for successful separation of the whole spectrum of underivatized FAAs (Gao *et al*, 2016; Konieczna *et al*., 2018, Guo *et al*. 2013).

However, in comparison to HILIC, relatively a lesser number of underivatized FAAs have been reported in relation to RPLC, as the chromatographic methods described

have failed to separate the complete profile of amino acids on RPLC (Nimbalkar *et al*., 2012, Ozcan and Senyuva, 2006; How *et al*., 2014; Cocuron *et al*., 2017). Reversed phase HPLC is a commonly used technique to analyse plant metabolites (Pai, Pawar, Nimbalkar and Dixit, 2011). However, polar low relative molecular mass compounds such as hydrophilic amino acids are not adequately retained and therefore alternatives are required.

LC-MS/MS technique proffers an efficient analysis of amino acids without derivatization, this has been established by numerous research work among others (Schwarz *et al.*, 2005; Ozcan and Senyuva, 2006; Thiele *et al*., 2008; Igor *et al.*, 2013; Guo *et al*. 2013; How *et al*., 2014; Gao *et al*, 2016; Cocuron *et al*., 2017; Konieczna *et al*., 2018). The major advantage of LC–MS technique over other known methods is that amino acids can be analysed without derivatization. Therefore, the analytical limitations inherent with derivatization are eliminated in the underivatized LC-MS/MS detection with improved selectivity.

2.6.3 Phenolic Compounds

Phenolic compounds are derivatives of tyrosine or phenylalanine identified as plant secondary metabolites. Structurally, they are made up of aromatic group having one or more hydroxyl functional groups attached to the aromatic ring (Shahidi and Naczk, 2003). Simple phenolic acids are derivatives of cinnamic or benzoic acid while tannins and flavonoids belong to the complex family. Different flavour perceptions emanate from phenolic and polyphenolic compounds. This is in the range of the sweet chalcones and some glycosylated flavonoids to bitter and astringent tannin compounds. Phenolics can be closely linked with the nutritional and sensory quality of fresh and processed plant foods. However, Dimberg *et al*., (1996) and Peterson (2001) reported that cereals flavour is contributed to by the presence of non-volatile phenolic compounds. Naturally occuring phenolic compounds in spices and herbs usually serve the dual purpose as food preservatives and flavourants (Soto-Vaca *et al*., 2012).

In cereals, among the most abundant phenolic acid that influences flavour is ferulic acid.Although,phenolics including p-coumaric, sinapic, and caffeic acids present in small amount are also flavour active in property (Andreasen *et al*., 2000; Weidner *et*

al., 1999). For example, Dimberg *et al*., (1996) observed that though the percentage of free phenolic acids in cereals may be small (10- 90 mg/kg), however they have a strong influence onthe perceived flavour in oat. The perception of bitter taste in cereal products has been linked to the presence of phenolic compounds such assyringic and pinoresinol acid (Heiniö *et al*., 2008). Flavour activeness of phenolic is dependent on its nature of availability. For example, free phenolics are perceived in food due to ease of adherance to the taste receptors whereas this is not so when in bound form. Though, ferulic acid in rye is in a bound form, however it is not flavour active; despite the fact that it is the most abundant phenolic acid present in rye. The plant type, food processing operations and storage conditions were identified to determine the concentration and type of phenolic compound in foods (Schroeter *et al*., 2010)

Research reports have established the benefical effects of phenolic compounds' antioxidants in cereal foods and humans (Fardet, 2010; Bondia-Pons *et al*., 2009). Higher concentrations of phenolic compounds are found in the bran which is located at the outer layer of grains (Decker *et al*., 2002; Shewry and Bechtel, 2001). Nutrient loss together with the production of undesireable flavour and colour during fruit and vegetable processing is attributable to the enzymatic browning reaction of phenolic compounds in the presence of polyphenoloxidase. In some specific foods, colour and flavour development during processing and preservation is solely by the oxidative changes of polyphenols.

The desirable and undesireable taste, and aroma of cereals is sometime directly determined byits phenolic compounds. p-cresol, 2- isopropylphenol, 3,4-dimethylphenol, carvacrol, thymol, 3-isopropylphenol and 4- isdpropylphenol were observed by Ho (1992) to give the characteristics sheep-mutton aromas in ovine fats. Flavourants in beef have been attributed to the presence of cresols, especially w-cresol. 4-vinylguaiacol produced during storage and processing of citrus products is among the main contributor of major detrimental off-flavors (Ho, 1992).The formation of the compound (4-vinylguaiacol) is from ferulic acid following the release of ferulic acid from bound forms.

Among the factors that determines the phenolic and polyphenolic concentration in plants are moisture, growing conditionsand attack by plant pathogens. Also,

conditions of storage, processing, extent of ripeness, germination significantly influence phenolic concentration (Soto-Vaca *et al*., 2012). Postharvest conditions and storage affect the stability of flavonoids and phenolic acids in cereals in a wide-range of reactions. Phenolic compounds in plant foods are in abundant, and therefore, a significant quantity is consumed in our daily diet. They are closely connected with the nutritional and flavour quality of fresh and processed plant foods.

One of the food preservative uses of antioxidants is in its addition to fats and oils, and sometimes foods that are fat base in nature. This is to inhibit the production of compounds of undesireable flavours from the oxidation of lipids (Namiki, 1990). Most natural antioxidants are phenolic in nature. As such, the natural use of food materials that are rich in phenolic compounds as additive to perform antioxidative functions on the host material. These has led to the developments in its use over the years in the food industry (Ho, 1992).

More generally, polyphenolic compounds are known to impact astringent or bitter flavours. And as such much of the research interest on the compounds were focused on its flavour properties (Table 2.3). Flavour imparted by phenolic compounds is of major importance in isolated proteins, wine and beer, as well as vegeTables. The presence of this flavour compound could be both desirable and undesireable. Although the desireability or otherwise is largely dependent onthe concentration of the compound of interest and the type of food.

The extent of polyemerization of phenolic compounds affects the concentration and duration of their bitter or taste responses. Although eugenol and vanillin which belong to the simple molecule group have a strong and characteristic flavour. However, bitter and astringent flavours are generally associated with polyphenolic compounds. The work of Callemien and Collin (2009) reported the numerous impact of phenolic compounds on beer, and other papers have also discussed their importance on wine flavour (Waterhouse, 2002; Cheynier *et al*., 2006; Monagas *et al*., 2005).

Table 2.3 Phenolic Compounds flavours and their occurence in Foods

Class	Examples	Occurence	Flavour
Benzoic acid	Hydroxybenzoic protocatechuic vanillic syringic	Beer Raspberries Acai Acai	Bitter Astringent Astringent Bitter
Hydroxy-cinnamic acid	Synaptic Ferulic cinnamic	Cereals Apple Coffee cinnamon	Bitter-sweet Astringent Cinnamon
Stillbenes	Resveratrol	Grapes Peanut	Bitter Astringent
Flavan-3-ols	(*)-catechin (-)-Epicatechin	Tea, Chocolate Tea Chocolate	Bitter Astringent Bitter Astringent
Isoflavones	Genistein Glycitein Daidzein	soy	Bitter
Chalcone	Neoshesperidin	Citrus	Sweet when hydrogenated to dihydrochalcone
Flavone	Tangeritin Nobiletin	Orange orange	Bitter Bitter
Flavonol	Quercetin	Wine, Tea Endive	Bitter
Flavanone	Naringin	Orange Juice Grapefruit Juice Citrus peel	Bitter

Source: Soto-Vaca A., Ashley G., Jack N. L., Zhimin X., and Finley J. W. (2012).Evolution of Phenolic Compounds from Color and Flavor Problems to Health Benefits. *J. Agric. Food Chem*. 60: 6658−6677.

Phenolic compounds contribute to food flavors in multiple ways. In addition, and because of their antioxidant properties, they participate in the lipid oxidation process that takes place in many foods upon processing. In these foods they play an important part in the inhibition of the production of lipid-derived off-flavors (Plaza *et al*., 2014; Gobert *et al*., 2014; Contini *et al*., 2014). Furthermore, because some phenols are able to react with carbonyl compounds, they are able to modify the flavor generating reactions in which these carbonyl compounds are involved. This occurs, for example, in the flavor and off-flavor development via Maillard chemistry (Kokkinidou and Peterson, 2014; Tressl *et al*., 1977; Totlani and Peterson, 2005).

Moreover, phenolic compounds are able to scavenge lipid-derived carbonyl compounds that significantly impart flavor of foods (Hidalgo and Zamora, 2014). In addition to all of these roles, phenolic compounds can also be transformed either enzymatically or nonenzymatically into quinones, and quinones are able to degrade amino acids with major consequences in the flavor of foods (Rizzi, 2008).

2.6.4 Fatty Acid

Rice lipids are generally classed as starch lipids which are associated with the starch granules and non-starch lipids, from other cellular components. Lipid content of rice ranges from 1 % to 4 % (Kennedy and Burlingame, 2003; Mano *et al*., 1999). The free fatty acids in rice are mainly palmitic, stearic, oleic and linoleic acid (Zhou *et al*., 2002a). Volatile formation from lipids results from lipolysis, lipid oxidation and decomposition. Lipase produces free fatty acids that may undergo oxidation. Hydroperoxides formed with lipid oxidation readily decompose yielding a variety of products with varying molecular weights and odor thresholds. Decomposition products include aldehydes, lactones, ketones, furanones, alcohols, acids and hydrocarbons many of which impact flavor (Nawar, 1996).

During storage, the activity of lipase and lipoxygenase increases, resulting in an enhanced production of the volatile compounds, particularly hexanal which contributes to off-flavor (Zhou *et al.,* 2002). The 2-alkanone is produced from saturated fatty acids in substantially larger quantities during thermal oxidation. Lipids are also important to the flavor of foods, because they increase the binding of

lipophilic flavor compounds. Lipids can moderate both flavor release and perception (Reineccius, 2006).

Another major causes of off-flavour and bitterness in food is rancidity. This is due to hydrolysis of lipids, and subsequent fatty acid oxidation. The occurrence of hydrolysis of lipid could be chemical reactions or enzymatic base. This depended upon the conditions of processing. Stability of whole grain flavours could be controlled by impeding the formation of free fatty acid through this processes. This established the fact that flavour stability of food materials is significantly influenced by the hydrolysis of lipids. Although the presence of carbohydrate or protein degradation product may cause some changes in food flavour, but lipid hydrolysis has the most significant impact on flavour stability of wholegrain flavour(Doblado-Maldonado, Pike, Sweley, and Rose, 2012; Heiniö, Lehtinen, Oksman-Caldentey, and Poutanen, 2002). This is also true of flour during storage, where sensory property is negatively affected by the lipolysis. The flour is thus perceived as rancid with a bitter note. Rice with FFA levels above 0.1% might adversely affect beer flavor (Lam *et al*., 2001). Oil off-flavors resulting from high FFA levels are due to unsaturated FFA oxidation to conjugated diene (CD) hydroperoxides, which then decompose to form volatile off-flavors (Galliard, 1989).

2.7 Rice Processing and Effect on Flavour Components

Change in rice flavour has been reported to be dependent on pre- and post-harvest treatments. This change has been associated to changes that are evident in the composition of the volatile constituents under diverse conditions (Borompichaichartkul *et al*., 2007). As such proper pre and post harvest activities are key to maintaining the required flavour quality for rice, and this will also influence the rice yield significantly. Cultural pre and post harvest activities during rice processing is largely dependent on the geographical location. Variations in methods with respect to temperature of paddy steeping, drying, steaming, storage moisture content, storage conditions and duration are important factors that determine the acceptability of rice flavour.

A solution adopted in post harvest handling of wet paddy was the introduction of high temperature drying by the rice industry in Thailand (Soponronnarit, 1995). However,

the quality of processed rice is dependent on drying temperature. High temperatures may lead to losses and changes in some components of rice. The work of Sunthonvit *et al*., (2005) on the influence of high-temperature fluidized bed drying operation on volatile compounds in rice (Thai jasmine) reported that increasing temperature of drying from 100 to 150^{o}C might lead to increase in the quantity of 2-AP in rice. Although this is in contrast at low temperatures according to a study by Wongpornchai *et al*. (2004) who found out that the amount of 2AP in Thai jasmine rice decreases as the temperature of drying increased from 30 to 70^{o}C. However, these variations in experimental output could be linked to the different drying techniques adopted by the researchers. In the work of Borompichaichartkul *et al.,*(2007) who combined 30 min tempering between the pass or ambient air drying and drying at various high temperatures (115–150^{o}C) reported its significant effect on the composition of volatile compounds and thus making the quality of thai jasmine rice to be acceptable commercially.

According to Sunthonvit *et al*., (2005) in a study which explored the effect of drying on thai fragrant rice reported that volatiles (aldehydes, ketones, alcohol and heterocyclics compounds) are generated, evaporated and degraded during thermal processes. This is however dependent on the flavour compound and as such could enhance or degrade flavour quality of rice. It was also reported that hexanal obtained in rice samples could be due to the thermal decomposition of linoleate and hydroperoxides (Houston, 1972). Formation of alcohol in thai rice samples is also attributed to the decomposition of fatty acids by lipoxygenase activity. However, the reaction is catalysed by heating. The presence of 1-Octen-3-ol was linked to the action of heat on hydroperoxide of linoleic acid (Hsieh *et al*., 1994).

Formation of heterocyclics in food is mainly through non-enzymatic browning reaction or maillard reaction. High processing temperature of processing was identified as a factor that could be responsible for increase in the heterocyclics that influence rice aroma (Sunthonvit *et al*., 2005). Maillard reaction which is known to be temperature dependent has been indicated in the formation of 2AP in cooked rice (Maga, 1984). The effect of temperature was reported to give oat its desired flavour and also inactivation of lipolytic enzymes in oats (Molteberg *et al.* 1996). This was also corroborated by Sides *et al*., (2001) who reported that the cereal like flavour in

oat could be associated to the heat treatments. Parker *et al*., (2000) reported the enhancement of Maillard reaction by high temperature during extrusion and described the product as a toasted and cereal like flavour.

2.8 Principle of Flavour perception

Flavour comprises perception received from a food material in the mouth through the chemical senses (Meilgaard *et al*., 1999). Olfactory and gustatory perceptions are two major identifier of a given flavour compound. The olfactory perception is defined by volatile compounds identified by the nasal cavity while gustatory perception is defined as the soluble substances identified in the mouth as taste. However,chemical feeling perception is of major importance because factors such as spice heat, astringency, bite and cooling are know to stimulate the nerve ends. Therefore taste perception; odour perception and trigeminal nerve response ascribe a description to a particular flavour in food (Lawless and Heymann, 1999).

Over the years, consumers have been able to define four basic tastes in food product which include sweet, salty, sour and bitter. However other tastes such as umami and metallic have evolved in both definition and acceptance. It is worthy of note that researchers varies in their opinion on the definition of this basic taste in food due to the technicality in the synesrgistic effect in their interactions (Breslin, 1996; Brannan *et al*., 2001; Keast and Breslin, 2002). Flavour compounds have their unique taste when found alone, however the concentration of the taste solutions is a major factor in the taste interactions (Keast and Breslin 2002). For example, Breslin (1996) noted that moderate concentrations of salts and acids enhance each others flavour, while they suppress themselves at high concentrations.

Headspace of food products is occupied with diversity of flavours which is due to presence of volatile compounds which are easily perceived at the nasal cavity through the olfactory receptors. This variation is facilitated by smell. Transduction mechanism was reported to be responsible for olfactory perception of flavour (Bell, 1996). Flavour release has been reported to be complex due to the mode of interactions that are in diversity (Plug and Haring, 1994; Taylor 1996; Laing and Jinks, 1996; Taylor and Linforth 1996; Buettner and Schieberle, 2000; Dattatreya *et al*. 2002; Linforth *et al*. 2002). Numerous works have been done on volatile compounds in food, however the influence of food-bound or semi-volatile compounds requires focus, since they

may also contribute significantly in the understanding of flavour in food (Dattatreya *et al*., 2002).

Human asseessment of flavour compounds is controlled by the flavour compound present and their availability to the senses with respect to time together with the mechanism of perception. The nasal cavity serves as the route of transduction where the aroma-binding proteins transfers the odorous compounds across. The membrane-bound receptors recognise this odorant and translate them to chemical information into nervous activity. Among numerous factors responsible for flavour release as reported by Guichard (2002) are some dependent factors such as amount and composition of saliva, shear-force of chewing, breathing rate and volume. While some other factors are dependent on the nature and interaction of food components.

Aside individual variation in odour perception, understanding of the importance of odour threshold is key to understanding flavour. According to Meilgaard *et al*., (1999), sensory capacities have limits and this is refered to as odour thresholds. Variation inodour threshold is characterised by detection, recognition, difference and terminal thresholds. These thresholds are determinants for aroma perception of food by a consumer. For example, Van Gemert (1999) reported 0.0003 - 20.6 mg/m^3 in air while it was 0.00008-0.03 mg/m^3 in water for dimethyl sulphide (DMS) odour threshold. The work of Noble (1996) described the relationship that existed between taste and aroma. It was reported that the apparent intensity of taste may lead to increase in the aroma while the apparent intensity of aroma might also lead to increase in taste. Complexity in the factors that determines flavour perception also makes flavour description of a food to be a herculean task even with the use of standardised method during sensory evaluation. Variation in senses of flavour due to individual experiences and knowledge is a factor of consideration in understanding flavour perception and standardisation.

2.9 Microbial dynamics during food processing

Microbial and enzymatic processing of food which is an influence of food fermentation confers desirable characteristics on food. Characteristics which ranged from prolonged shelf-life, improved safety, attractive flavour, nutritional enrichment, to promotion of health (Holzafpel, 1997) are related to microbial activities in food. Microbial biosynthesis is a major route for flavour formation in food (Krings and

Berger, 1998; Vandamme and Soetaert, 2002). During microbial growth on cereal grains, Harris *et al*., (1986) noted that microorganisms produce a wide variety of specific metabolites and volatiles which includes acids, alcohols, ketones, enzymes, antibiotics, alkanes, and terpenes. Major fermentation microorganisms in food include lactic acid bacteria (LAB), moulds and yeasts (Okada *et al*. 2006; Olstorpe *et al*. 2008).

The complexity in food fermentation processes have been reported by researchers (Giraffa,2004). This could be due to the fact that agricultural products of animal or vegeTable origin are fermented by either the indigenous microflora or an added starter culture to improve or obtain shelf life, nutritional value, health benefit, flavour or texture. As shown in Figure 2.4,variation in compounds produced by the action of different microorganisms differs. For example diacetyl is reported to be produced by lactic acid bacteria while 2,3-butanedione and acetaldehyde are produced by milk cultures of *Streptococcus lactis, S. cremoris* and *S diacetilactis* (Ibragimova, 1980**)**. Also 3-octanone, 1-octen-3-ol, 1-octanol and 3-methyl-1-butanol, 2-methyl- 1-propanol, 3-methylfuran, ethyl acetate, 2-methyl-isoborneol, and geosmin are metabolites that are produced by fungi (Kaminski *et al* 1975; Abramson *et al* 1980; Tuma *et al* 1989; Schnurer *et al* 1999).

Factors like temperature, pH, inoculums, size, type of cereal and propagation time determines the type of microorganisms that is found in association with each type of fermentation (Achi and Ukwuru, 2015). As such, fermenting organisms acting on a particular substrate at a particular time is dependent on the factors mentioned. Physiology, metabolism and genetic properties of microorganisms are also major determinants of the dynamics of fermentation processes (Ibragimova, 1980). Therefore the knowledge of microorganism, subtrates and conditions of growth are of major importance in order to control the production of metabolites (Giraffa, 2004). Microbial diversity, growth cycle, taxonomic identity, quantitative changes, and spatial distribution of microbial species are key factors to be understood at every stage of food fermentation (Fleet, 1999). This is to ensure effective management of the growth and activities of microorganisms in fermented foods.

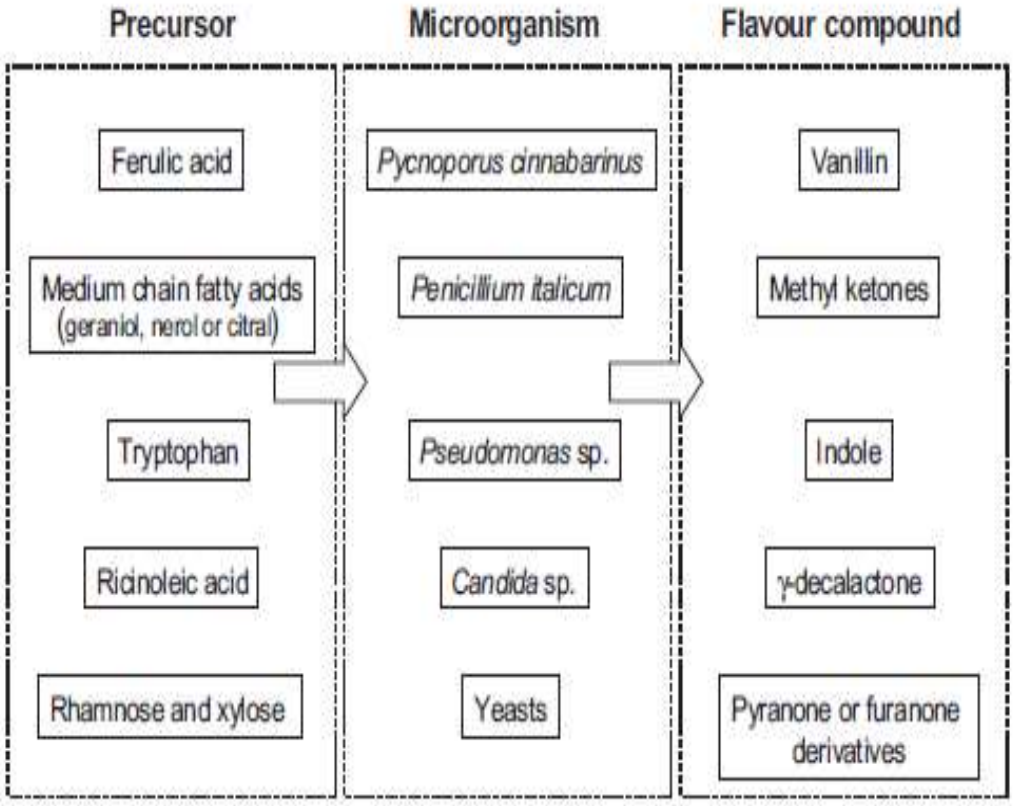

Figure 2.4 Microbial activities on precursors in the production of flavour compounds
Source: Longo, M.A. and Sanromán, M.A..2006. Production of Food Aroma Compounds.*Food Technol. Biotechnol. 44(*3): 335–353

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In the work of Ayelogun and Adegboyega (2000), it was reported that steeping rice paddy for too long leads to unacceptable colour, taste, and odour of rice due to fermentation processes. Otegbayo (2001) reported that steeping contributes mainly to organoleptic, physical and nutritional changes in rice. Bacteria, yeast and moulds being mainly responsible for the organoleptic properties. Production of flavour compounds and aroma are made possible through fermentation processes carried out by yeasts and lactic acid bacteria (Omemu, 2007). The presence of fermented odour in Ofada rice as reported by Anuonye *et al.* (2016), was successfully removed by adopting hot water steeping method. In the work of Adeniran *et al.* (2012), use of starter cultures reduced the steeping time of "Ofada" rice and it also enhanced the nutrient composition and improved the sensory quality of the rice. This showed the role being played by microorganism in defining the Ofada rice flavour.

Adeniran *et al.,*(2012) noted that Ofada rice is specially relished because of its characteristics flavour that develops during steeping as a result of the fermentative activities of some microorganisms. It has been reported that aside the changes in composition and distribution of nutrients in the rice kernel, Steeping also causes the leaching of rice constituents into the steep water (Otegbayo *et al*., 2001; Ibukun, 2008; Sareepuang *et al*., 2008).

Understanding the relationship that exist among microbial growth, activity of individual microorganisms, product quality, safety are among factors highlighted by Fleet (1999) that aids the understanding of microbial activities. Research works has reported the synergistic mode of microbial activities and dynamics in several fermentation studies (Kantachote *et al*., 2008; Adeniran *et al*., 2012; Adelekan and Nwadiuto, 2011*)*. For example, Kantachote *et al*., (2008) in a study on fermentation dynamics of wild forest noni (*Morinda coreia* Ham) reported initial increase of lactic acid bacteria for the first 7 d of fermentation and its dominance for 21 d before yeast took over the dominance in the fermentation media. In the study, *Lactobacillus plantarum* and *Saccharomyces cerevisiae* were identified as the dominant lactic acid bacteria and yeast respectively.

According to Kantachote *et al*., (2008) *Lactobacillus pentosus* was only detected on the 21st day of fermentation while it was also reported that *Saccharomyces cerevisiae* was originally the dominant yeast but was later replaced by *Pichia membranifaciens* followed by *Pichia anomala*. All this variations in the fermenting microbes were reported to be responsible for the changes in the pH and acidity of products. Thus, reflecting numerous complexities associated with the formation of metabolites that gives the products its characteristics.

Adelekan and Nwadiuto (2012) in a bacteria dynamic study during production of "iru" (locust bean) through the fermentation of african locust bean reported the dominance of *Bacillus anthracis* relatives, *Bacillus cereus and Bacillus* sp. *a*t 0 h, 24 h and 48 h of fermentation respectively. In the same work the presence of *Enterobacter*sp. was found only after 72 h of feremtation. Each stage of fermentation has been reported to possees differences in microbial community and this lieu is responsible for the characteristic aroma and texture of the food product. In the same vein, during "Ogi" fermentation it was reported that as the population of the filamentous mould decreases, the LAB population increases within 2 d (Omemu, 2011). This variation is significant in the production of metabolites as they are regulated by microbial activity and this leads to the modification of the composition of the product. The work of Vieira-Dalode (2007) on identifying microoganisms responsible for *gowe* production from sorghum reported a mixed fermentation of sorghum which was dominated by *Lactobacillus fermentum*, *Weissella confusa*, *Pediococcus acidilactici*, *Kluyveromyces marxianus*, *Pichia anomala*, *Candida krusei.*

In a study that characterised bacteria and yeasts responsible for the paddy rice fermentation by Adeniran *et al*. (2012), it was reported that the dominant lactic acid bacteria found to be responsible for the fermentation of "Ofada" rice were *Leuconostoc mesenteroides* and *Lactobacillus amylophillus*, while *Saccharomyces cerevisiae* and *Saccharomyces uvarum* were the dominant yeasts in the fermenting steep water. Seven species of LAB detected during Ofada rice fermentation includes *Lactobacillus maltaromicus, Lactobacillus alimentarius, Lactobacillus kefir, Pediococcus acidophilus, Lactobacillus amylophillus and Lactobacillus farciminis* and *Leuconostoc mesenteroides*. Whereas six species of yeasts detected were

Saccharomycodes sinensis, Saccharomyces uvarum, Pichia angusta, Candida mesenterica,Torulaspora delbrueckii, S. cerevisae.

Adeniran *et al*., (2012) observed that the onset of paddy rice steeping (0 h), the yeasts that were present were *Candida mesenterica, Saccharomyces cerevisiae* and *Torulospora delbrueckii* while the bacteria were *Lactobacillus maltaromicus*, *Lactobacillus kefir, Lactobacillus amylophillus* and *Pediococcus halophillus.* At the 24-h mark of steeping, *Leuconostoc mesenteroies, Pediococcus halophillus* and *Saccharomyces uvarum* were found in the steep-water in addition to the former isolates, while *Lactobacillus alimentarius* disappeared. By 48 h, *Lactobacillus farciminis, Saccharomycodes sinensis* and *Pichia angust*a appeared in the water and the latter two remained till the end of the fermentation while *Lactobacillus maltaromicus*, *Lactobacillus kefir*, *Candida mesenterica*, *Torulospora delbrueckii* and *Pedicoccus halophilus* disappeared.

Variation in the microbial composition as the steeping duration increases was attributed to changes in pH and acidity (Adeniran *et al*., 2012). The knowledge of this dynamics have been used to compose starter culture used during Ofada rice steeping in order to improve its sensory attribute. The use of lactic acid bacteria and yeasts during Ofada rice steeping have been reported to have reduced steeping duration and also improve the nutritional profile of Ofada rice (Adeniran *et al*., 2012).

Identification and characterisation of microbes that determines the characteristic properties of foods during fermentation process is of great importance. This is to allow control of the process through selection of appropriate conditions for the process, and so as to be able to use the right starter culture to improve the technology of processing. Cocolin and Ercolini (2009) noted increased interest in identifying microbial ecology due to advances made in molecular biology which includes advent of polymerase chain reaction and DNA sequencing. These methods help to identify microbial populations; including those that grow in laboratory media, those that did not grow, as well as those that are both active and dormant in the community.

This modern method of understanding microbial dynamics comprises the use of molecular methods on isolated strains during the fermentation for their identification

and characterisation (culture-dependent methods), as well as direct application of molecular biology techniques in order to profile the microbial diversity without the need of cultivation (culture-independent methods). Their application in the field of the microbial ecology of fermented foods has been reviewed (Cocolin and Ercolini, 2009). Errors associated with cultivation of microorganism in culure media are avoided when methods that are not dependent on culturing are used. When using the culture independent methods, factors such as microbial growth, culture media enrichment does not affect the identification of microorganism. This is because the process involves the use of DNA and/or RNA gotten from the sample under consideration.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials and Equipment

Ofada rice paddy (OS-6) was purchased from the Ogun State Agricultural Development Program (OGADEP) Idi aba, Abeokuta. Equipment such as calibrated water boiler, pressure cooker, forced air-draft oven and pH meter were used at the Food Processing and Food Chemistry laboratory of the Department of Food Technology, University of Ibadan for paddy rice processing. Phenolic extractions and analysis were done in the Department of Plant Science, University of the Free State, Qwaqwa Campus, South Africa. Identification of aroma compounds with Gas Chromatography-Mass Spectrometry (GCMS) and determination of primary metabolites was done with Liquid chromatography-Mass spectrometry at the University of South Africa, Florida Campus. Metabolomics to determine the succession of bacteria during the steeping of paddy rice was done at the Department of Molecular Biology, University of South Africa, Florida Campus. All chemicals used at different stages were of laboratory standard.

3.2 Experimental Design for Paddy Rice Processing

The traditional processing methods of Ofada rice involvingsteeping, parboiling, drying and milling were thoroughly studied with the local processors. Variation of pretreatment conditions was made using initial steeping temperature, steeping duration, parboiling temperature and drying temperature as critical processing controls points. Literature also showed these pretreatment conditions to be critical to flavour and off-flavour development in Ofada rice. The initial steeping temperature used were 30^{o}C, 65^{o}C and 100^{o}C. The choice of these temperatures was made base on the report of Anuonye *et al.,* (2016) on the effect of initial soaking temperature on rice flavour acceptability. Steeping duration of one, three and five days were adopted as reported by Adeniran *et al.,* (2012) to be the local practice of rice processing households in the southwest part of Nigeria.

Paddy parboiling was done at 80, 100 and 120^{o}C, while rice paddy drying was carried out at 30, 50 and 70^{o}C. The choice of parboiling and drying temperature were made based on the optimisation of Ofada rice processing conditions (Adekoyeni *et al.,* 2015).

To incorporate all traditional processing method variables, D-optimal Response Surface methodology was adopted for the design of the experiment (Montgomery, 2004). The variable constraints are as shown in Table 3.1. while the randomised experimental design is as shown in Table 3.2. The traditional processing sequence in which the experimental design variables were imposed is as shown in Figure 3.1.

3.3 Preparation of sample

At the reception of the paddy, mixing and cleaning were done to ensure randomness and removal of immature paddy together with unwanted materials before processing. Prior to steeping, paddy rice was thoroughly washed three times and floating paddy was removed to eliminate any other immature paddy. The paddy rice was well-drained with a screen mesh of 0.05µm and the wash water was allowed to be dried before further processing. Cleaned Ofada paddy (4 kg each) were weighed into nine different sealable containers. While five litres of water at 30^{o}C, 65^{o}C and 100 ^{o}C were added and sealed to commence fermentation of paddy rice.

After one, three and five days of steeping, samples were taken for parboiling/steaming operations inside a temperature calibrated stainless steel water boiler. After parboiling at 80, 100 and 120^{o}C for 20 minutes, paddy was allowed to drain before the commencement of drying operation at 30, 50 and 70^{o}C in a forced air-draft oven. A local rice milling machine was used for the milling operation of dried rice paddy. Immediately after milling, processed rice was packaged in Ziploc brand laminated polyethylene and individual placed in an air-tight container, and kept under refrigeration before further analysis.

Table 3.1. Design Variables and Variation for Ofada Rice Processing

Parameters	Variation
Steeping duration	1, 3 and 5 days
Initial steeping temperature	30, 65 and 100^{o}C
Parboiling temperature	80, 100 and 120^{o}C
Drying temperature	30, 50 and 70^{o}C

Table 3.2. Randomised Experimental Design for Variables

S/N	Initial steeping temperature (^{o}C)	Steeping duration (Days)	Parboiling temperature (^{o}C)	Drying temperature (^{o}C)
1	100	5	80	30
2	100	1	120	50
3	30	5	120	30
4	30	1	120	30
5	30	1	120	30
6	100	5	120	70
7	65	2	100	50
8	65	3	120	50
9	30	1	80	70
10	100	3	100	50
11	100	5	120	70
12	30	5	80	70
13	100	1	80	30
14	30	3	80	30
15	30	1	80	70
16	30	3	100	50
17	100	3	120	30
18	100	1	80	30
19	30	3	120	70
20	65	3	80	50
21	100	1	100	70
22	65	5	100	50
23	65	1	120	70
24	30	5	120	30
25	100	3	80	70

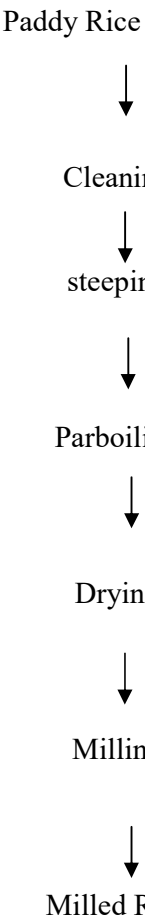


Figure 3.1.Milled rice processing from paddy

Source: Adekoyeni, O.O., Sogunle, K.A. and Fagbemi, S.A. (2015). Effect of storage and processing parameters on protein and amylose of Ofada rice grain. *FUDMA J. Agric & Agric. Tech*.1(1): 21 – 30.

3.4 Sample collection for microbial analysis

Using the generated experimental design data (Table 3.2), variation in the initial steeping temperature and steeping time was explored to know fermenting microorganisms and also determine their succession. Ofada rice Paddy (4 kg) was steeped in water at initial steeping temperatures of 30, 65 and 100^{o}C. Steep water samples were collected at 24 h interval for 5 d. The pH was determined before being stored at -86^{o}C in an ultra-low temperature freezer for subsequent microbial determination.

3.5 Determination of pH of steep water

The AOAC (2000) method was used for pH determination. This was done at every 24 h interval when the steep water was collected. pH of the steep water was measured using a digital pH meter (Bante instrument, PHS.25CW microprocessor).

3.6 Determination of titraTable acidity of steep water

The method of Edema and Sanni (2008) was used in determining the titraTable acidity of fermentingsteep water. The method involved diluting ten millilitres of the steep-water in 90 mL of sterile distilled water. The diluted steeped water (25 mL) was then titrated against 1 M NaOH with 10 % phenolphthalein (0.5% in 5% ethanol) as an indicator. The titratable acidity content was calculated using equation 3.1.

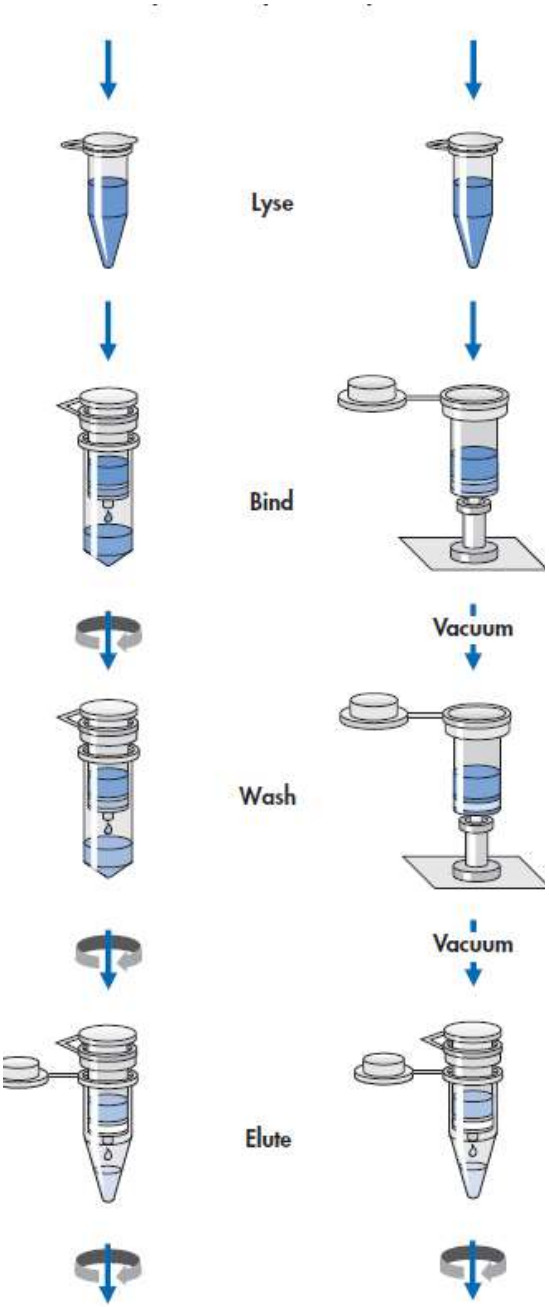
$$\text{Lactic acid mg mL} = \frac{\text{Average Titre value X 90.08}}{\text{Volume of sample}} \qquad 3.1$$

Where 90.08 is the lactic acid equivalent factor.

3.7 Characterisation of fermenting microorganisms

Nucleic acids were extracted from 10 mL of concentrated steep water samples using manufacturer instruction of QIAamp Kit (AllPrep PowerViral DNA/RNA Kit) as shown in Figure 3.2. Eluates were stored in aliquots at -80 ^{o}C for subsequent amplification by polymerase chain reaction. Real-time PCRs were carried out using a Bio-Rad Mini Opticon system with 5 µL of the extracted genome used. A disposable optical 48-well PCR plate in a 25-µL mixture was used for the reaction.

steeping water sample



Pure Nucleic acid

Figure 3.2: QIAamp™ DNA extraction Procedure

Source:Qiagen (2010). QIAamp Viral RNA Mini Handbook.Available at www.qiagen.com.

The reaction mixture was heated for 2 min at 50°C and then at 95°C for 10 min; activation was followed by a 40-cycle, two-step process, each cycle consisting of denaturation at 95°C for 15 s and annealing at 60°C for 1 min. The raw forward and reverse ABI files were aligned and assembled into a single consensus sequence using the Molecular Evolutionary Genetics Analysis (MEGA) software, version 6.0. NCBI BLAST (Basic Local Alignment Search Tool) http://blast.ncbi.nlm.nih.gov/Blast.cgi was used to evaluate the relatedness of sequences which finally determined bacteria present as described by the method of Iaconelli *et al*., (2017).

3.8 Determination of antioxidant properties

3.8.1 Preparation of rice sample for antioxidant properties

Samples were prepared for antioxidant properties according to the method of Walter et al. (2011). Five grams of Ofada rice sample were ground to a particle size suitable for the extraction by Cyclone sample mill (Cole-Parmer, USA) and 50 mL of 80% methanol (v/v) was used for extraction for 20 h at ambient temperature with shaker being used for agitation of the mixture. The mixture was placed on a shaker to enhance extraction before being filtered by Whatman-40 filter paper. The extracts were kept at –20°C until further analysis.

3.8.2 Total phenolic content of Ofada rice

The Folin-Ciocalteu assay as reported by Qiu *et al.,* (2010) was used to determine the Total Phenolic Content (TPC) of rice. This was done with 2.5 mL of Folin-Ciocalteu reagent and 2 mL of 7% (w/v) sodium carbonate added to 0.5 mL of rice extracts. This was done in triplicate. The mixture was allowed to react for 90 min. A spectrophotometer (JENWAY, 7305 by lasec) was used to measure the absorbance at 765 nm after it has earlier been blanked with methanol. Different concentration of gallic acid was prepared by using serial dilution method to achieve a decreasing concentration. Then various absorbances at 765 nm of the different concentration were used to contsruct the standard curve. A derived equation from the standard curve was adopted to determine the concentration of the samples under analysis. The TPC of rice extracts were reported as the mean of the triplicate analysis. The calculation of the TPC was in gallic acid equivalent in mg per 100 g (mgGAE/100g).

3.8.3 Ferric-reducing antioxidant power of Ofada rice

The method described by Chu *et al*., (2000) for the determination of ferric-reducing antioxidant power (FRAP) adopted for rice extract. Acetate buffer was prepared by mixture of 1.6 g of sodium acetate and 8 mL of acetic acid and this was made up to 500 mL with a pH of 3.6. TPTZ was dissolved in 40 nM HCl before being used for the preparation of the FRAP reagent. The FRAP reagent was prepared with acetate buffer, TPTZ and $FeCl_3.6H_2O$ in the ratio 10:1:1 respectively. 300 µL of sample and 700 µL of water were added with 2.85 mL of FRAP reagent. Different dilutions of Trolox (200 µg/mL) were also prepared and were mixed as was done for rice extract with FRAP reagent. The mixture was kept safely at 50 °C for 20 min after which the resulting colour change was measured with a spectrophotometer (JENWAY, 7305 by lasec) set to an absorbance of 700 nm. A trolox calibration curve was constructed and the linear equation generated was used to determine the FRAP of rice extract in mg Trolox/g.

3.8.4 Total Flavonoid content (TFC) of Ofada rice

The method of Dewanto *et al*., (2002) was used in determining the TFC of Ofada rice. 0.25 mL of rice extract and standard solution of quercetin in triplicate were diluted with 1.25 mL of distilled water after which 75 µL of $NaNO_3$ Solution (5%) was added and incubated at ambient temperature for 6 min. After the incubation was the addition of 150 µL of 10% $AlCl_3$ and again incubated for another 5 min. 0.5 mL of 1 M sodium hydroxide solution was added and was diluted with distilled water to make up 3 mL. A spectrophotometer (JENWAY, Model 7305) was used to measure the absorbance at 510 nm. A calibration curve was constructed and the equation obtained was used to obtain the concentration of TFC in rice samples in mgQuercetin/g.

3.8.5 Total antioxidant capacity of Ofada rice

The total antioxidant capacity of Ofada rice extracts was evaluated using the method of Banerjee *et al.,* (2005). Phosphomolybdenum reagent was prepared by the mixture of 3.3 mL sulphuric acid, 335 mg sodium phosphate, and 78.4 mg ammonium molybdate in 100 mL of distilled water. Rice extract (0.1 mL) was added to the phosphomolybdenum reagent and placed in a water bath operating at 95^{o}C. Boiling of the mixture was done for 90 min after which the absorbance of the resulting mixture was read at 695 nm. This was also donefor blank reagent made up of an appropriate

volume of the same solvent in place of the sample. A standard curve was constructed using a varying concentration of gallic acid in place of the sample. The equation obtained was used in calculating the total antioxidant capacity of Ofada rice extract and the results were presented in mgGAE/g.

3.8.6 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging assay for Ofada rice extract

The DPPH radical-scavenging assay decribed by Turkoglu *et al*., (2007) was adopted for Ofada rice extract. The ability of the Ofada rice extract to decolourise the purple colour of the methanol solution of DPPH was used as a measure of their antioxidant activity. The DPPH (0.004 %) was prepared by mixing 4 mg of DPPH in 100 mL of methanol. Ofada rice extract (100 µL) was added to 300 µL of DPPH. Negative control of DPPH solution was used and a blank containing methanol instead of the rice extract was also prepared. The absorbance of the colour changes was read at 516 nm after being kept for 30 min with a microplate reader (BIO RAD, Model 680, Japan). Absorbance was used to calculate the percentage inhibition rate of the rice extract according to equation 3.2.

$$Percentage\ inhibition = \frac{Ac - Ae}{Ac} \times 100\% \qquad 3.2$$

Where Ac = Absorbance of control

Ae = Absorbance of extract

Gallic acid standard curve was constructed and the equation generated was used to determine the concentration of rice extract that gives 50% inhibition of DPPH radical in mg/mL.

3.9 Determination of primary metabolites in Ofada rice

3.9.1 Extraction for determination of primary metabolites

The method of Nimbalkar *et al*., (2012) was adopted in the determination of primary metabolites in Ofada rice. Milled rice was first ground to powdery form after which 1 g was weighed and added to 5 mL of 0.1% (v/v) formic acid in 20% (v/v) methanol in glassware. The mixture was vortexed for appropriate mixing and reaction for 5 min.

The mixture was then centrifuged at 4^{o}C at 10,000 rpm in 10 min. The obtained supernatant after centrifugation was filtered with a 0.2 µm nylon membrane filter. 10 µL of the filtrate was put in a glass vial and subsequently used for primary metabolites analysis.

3.9.2 LC-MS determination of primary metabolites

Perkin-Elmer Series 200 HPLC system connected to an API 2000 (ABS Sciex) triple quadrupole mass spectrometer equipped with electro spray ionisation (ESI) probe was used for the analysis of primary metabolites in the rice extract. Glass vials containing Ofada rice extracts were placed in the sample holders (chamber). The autosampler attached injected a 10 µL aliquot into the HPLC system. C18 column (Perkin-Elmer, 220 mm × 4.6 mm × 5 mm) was used for separation. The mobile phase was made upof water/methanol / acetonitrile (50:50:50). Acetonitrile and methanol/water (50:50) 0.1% (v/v) formic acid with flow rate 1 mL/min. The voltage of the ion source was 5500 V while the temperature of operation used was 500^{o}C. Also nebulizer gas operated at 207 kPa while the heater gas was at 379 kPa. The positive mode was adopted for the estimation and this was done by multiple reaction monitoring (MRM) using a scan duration of 50 min.

3.10 Determination of chemical properties of Ofada rice

Chemical properties determined were moisture, protein, carbohydrate, fat, ash, crude fibre, energy, amylose and free fatty acids. The chemical properties, amylose and free fatty acid were obtained in percentage while energy was in kCal/100g.

3.10.1 Determination of acid value and free fatty acidsof Ofada rice

The Acid Value (AV) and the Free Fatty Acid (FFA) were analysed using the method 940.28 of the AOAC (2000). Sample (0.2 g) of solvent extracted oil was mixed with 10 mL of ethanol. The resultant mixture was titrated against 0.1 M sodium hydroxide solution using phenolphthalein indicator. The titration was stopped at the disapperance of the pinkish colouration. The equations presented in equations 3.3 and 3.4 were used to estimate the acid value and the percentage of free fatty acid respectively.

$$\text{Acid value} = \frac{56 \text{ x molarity of NaOH x titre value}}{\text{weight of oil}} \qquad 3.3$$

% Free fatty Acid as Oleic acid = 0.503 × Acid Value 3.4

3.10.2 Determination of amylose and amylopectin of Ofada rice

Janaun *et al.,*(2016) method for the determination of amylose content was adopted for rice samples. Milled rice was powdered and a sample of 0.1 g was placed inside a volumetric flask (100 mL). 1.0 mL of ethanol (95%) and 9.0 mL of Sodium hydroxide were poured into the volumetric flask containing the rice sample. This was mixed thoroughly before boiling over shaking water bath (BS-31) for 10 minutes. The resulting solution was allowed to cool, after which it wasmade up to mark of the volumetric flask using distilled water. From the flask, 5 mL was taken and poured into an empty cleaned volumetric flask (100 mL). Acetic acid (1 mL of 1 M) and 2 mL of KI were poured into the solution and its volume was made up to mark of the volumetric flask. The solution was mixed thoroughly and allowed to stand for 20 minutes. Using UV-Vis Spectrophotometer JASCO V-650 at a wavelength of 620 nm, the absorbance of the mixture was read. A standard curve constructed by using various amylose concentrations for the above procedure was used to calculate the amylose concentration of the rice sample. The percentage of amylopectin was obtained by subtracting the percentage of amylose obtained from 100, as presented in equation 3.5.

% Amylopectin = 100 - % Amylose 3.5

3.10.3 Moisture content determination of Ofadarice

AOAC (2000) method of moisture determination was adopted. The weight of the dried crucible was noted after which five grams of processed rice was weighed. The crucible with the sample was then placed in a forced draft air oven for two hours. The weight of the crucible with the rice sample was determined hourly until no significant change in the weight was observed. The percentage moisture content was calculated as percentage loss in weight of the sample. Equation 3.6 was used to calculate the moisture content:

$$\%Moisture\ content = \frac{M1 - M2}{M1 - Mo} \times 100 \quad 3.6$$

Where Mo = weight of empty cruscible before drying

M_1 = weight of crucible and sample before drying

M_2 = weight of crucible and sample after drying

3.10.4 Protein content determination of Ofada rice

The milled rice samples crude protein was determined using the AOAC (2005) micro-Kjeldahl method number 978.04. The rice sample (5 g) was weighed into the Kjeldahl digestion flask. The Kjeldahl catalyst containing 5g K_2SO_4 and 5 mg Se was added followed by addition of 12 mL concentrated H_2SO_4. Digestion of the milled rice was allowed to take place for 4 h. The digest was made up to 100 mL, after which 5 mL of the solution was transferred into the kjeldahl flask and 5 mL of 40% (w/v) NaOH was added. Steam distillation of the mixture was allowed to take place and the liberated NH_3 gas was collected in 10 mL of 2% boric acid. It was then titrated against 0.01 M HCl solution. Percentage nitrogen in the digest was calculated by applying equation 3.7 while the percentage protein was obtained by using equation 3.8

$$Percentage\ Nitrogen = \frac{(Titre\ value - blank) \times 14.007 \times 0.1}{1000 \times Sample\ weight(mg)} \times 100 \quad 3.7$$

$$\text{Percentage Crude Protein} = \%\ \text{Nitrogen} \times 6.25 \quad 3.8$$

3.10.5 Ash content determination of Ofada rice

The ash of milled rice samples was analysed using the AOAC (2015) method no 930.05. Crucibles were washed and pre-dried with a forced air-oven, that was operated at 105^{o}C for 30 minutes. This was cooled in a desiccator and afterwards weighed before 5 g of samples were added and transferred to a preheated muffle furnace operating at 550^{o}C. Ashing was allowed to take place for 4 h before the muffle furnace was cooled to about 100 ^{o}C after which the crucible was transferred into the desiccator to cool to room temperature. It was then weighed and the ash content calculated according to equation 3.9

$$\%\ Ash\ Content = \frac{(Weight\ of\ crucible + ash) - (Weight\ of\ empty\ crucible)}{weight\ of\ sample} \times 100 \quad 3.9$$

3.10.6 Crude fibre content of Ofada rice

The AOAC (2000) method for crude fibre determination was used. Milled rice (2 g) was weighed into the 250 mL conical flask. 1.25% of H_2SO_4 was prepared and added to the sample. The mixture was then boiled under reflux for 30 minutes. The resulting

mixture was filtered and properly rinsed with distilled water while the residue was transferred back into the 250 mL conical flask where 100 mL of 1.25% NaOH was added and again boiled under reflux for another 30 min. The mixture was filtered and the residue was thoroughly rinsed before it was transferred into a crucible and dried in a forced draught oven at 180^{o}C for 3 h. This was then cooled and weighed before being transferred to the muffle furnace for ashing at 550^{o}C for 2 h until it completely ash. It was then cooled and weighed. Equation 3.10 gave the appropriate formula for the calculation of percentage crude fibre.

$$Percentage\ crude\ fibre = \frac{W2 - W3}{W1} \qquad 3.10$$

Where W_1 = weight of sample

W_2 = weight of crucible and sample after drying

W_3 = weight of crucible after ashing

3.10.7 Fat content determination of Ofada rice

The method No 930.09 of AOAC (2015) was used for fat content analysis. Milled rice (2 g) was placed in a fat free extraction thimble and the mouth of the thimble was plugged with defatted cotton wool before being placed in the extraction chamber of the soxhlet apparatus fitted with a reflux condenser on the heating mantle. The rice sample was then extracted with petroleum ether. The weight of the extracted sample was taken after six hours of extraction. The percentage fat content was calculated by using equation 3.11.

$$Percentage\ crude\ fibre = \frac{W1 - W2}{W1} \times 100 \qquad 3.11$$

Where W1 = weight of sample before oil extraction

W2 = weight of sample after oil extraction

3.10.8 Carbohydrate determination of Ofada rice

Milled rice sample carbohydrate content was estimated by difference. Obtained percentages of fat, moisture, protein, crude fibre, ash and moisture contents were deducted from hundred.

3.10.9 Estimation of the energy content of Ofada rice

The Atwater factor as given by FAO (2000) was used to estimate the energy value of milled rice samples. The three basal nutrients which include fat, protein and carbohydrate were used in the process. Percentage protein, carbohydrate and fat calculated were used to multiply their respective Atwater factor and their addition gave the energy value of the Ofada rice sample. The Atwater factor 3.82, 8.37 and 4.16 were used for crude protein, fat and carbohydrate, respectively as used in equation 3.12.

$$E\ (kcal) = (3.82 \times CP) + (8.37 \times CF) + (4.16 \times CHO) \quad 3.12$$

Where E = Energy; CP = Crude protein

CF = Crude fat; CHO = Carbohydrate

3.11 Identification of rice aroma compounds

3.11.1 Extraction of aroma compound

Solvent extraction (SE) of aroma compounds was done using 5 g of ground rice in 10 mL of dichloromethane, which was maintained at 80°C for a duration of 2.5 h in a water bath as reported by Bergman *et al*., (2000). The samples were extracted three times and rinsed with dichloromethane before re-extraction. The extract was dried over anhydrous sodium sulphate and concentrated to 1.0 mL using a rotary evaporator at 7.5 rpm.

3.11.2 Gas chromatographic (GC) analysis of aroma compoundsof Ofada rice

The composition of aroma compounds in the extract was determined by the Shimadzu Gas Chromatography Time of Flight Mass Spectrometer (GC-ToFMS) instrument (LECO Pegasus 4D; LECO corporation, St. Joseph, MI, USA). The chromatography was equipped with a DB-5MS capillary column with dimensions (Length × internal diameter × film thickness) of 30 m × 0.25 mm × 0.25 µm. The method of Zhou *et al*. (1999) was adopted for the GC-MS conditions. The extracted samples in screw cap vials were injected in splitless mode (1 µL). The starting temperature of the GC injector was at 40°C. A temperature increase of 10°C/min was used until the temperature got to 22°C. The total duration of separation of 40 min was used for each rice sample. 1 mL/min was adopted as the flow rate of Argon (the GC carrier gas). The TOF-MS operated with the ions source temperature of 220^{o}C while the mass

range was between 33 and 600. The detector voltage was 1700 V and also having an electron ionization of -70 eV GC analysis was performed three times for each rice sample to check the presence of peaks.

3.11.3 Identification and Quantification of rice aroma compounds

The compounds identification was carried out using mass spectra and retention indices that match the National institute of standards and technology (NIST) and Wiley mass spectra library database. However, the GCToFMS equipment used had been standardized by comparing the retention times of authentic standard of some identified compounds such as hexanal, limonene, 1-pentanol, n-octanol, hexanol, benzyl alcohol, benzaldehyde, octanal, 2-butoxy ethanol, indole and linalool. The method of Liyanaarachchi *et al.*, (2014) was used in quantifying compounds identified. Aroma compounds were approximately calculated by the percentage values obtained from the chromatogram with the assumption that the extraction obtained from CH_2Cl_2 contained a total aroma equivalent to a hundred. The results of the aroma compounds present were reported in percentage

3.12 Sensory evaluation of Ofada rice samples

The milled rice samples of 20g were cooked with the addition of 1g of salt. Sensory evaluations of cooked samples were carried out with 25 panelists who are conversant with parboiled rice sensory parameters. Panelists were instructed to rinse their mouth after every sample while they also take a break of about 5 minutes before assessing the next sample. Preference test using the nine hedonic scale was adopted for the sensory evaluation taking 1 to mean dislike extremely while 9 represented like extremely. Sensory attributes assessed by the panelists were aroma, taste, appearance, mouthfeel and overall acceptability. Panelists were also asked to rank their perception of off-flavour in cooked Ofada samples.

3.13 Statistical analysis

The Data obtained were subjected to descriptive analysis. The data were analyzed by ANOVA using the SPSS statistical program (SPSS13.0, SPSS Inc., New York, USA). Data were reported as means of triplicates. Means were separated by Duncan's multiple range tests to establish significant differences at $p < 0.05$ between samples respectively. Design expert 7.0 was used for the modelling and optimisation of data

obtained. Principal Component Analysis (PCA) of sensory attributes and aroma active compounds detected were performed using PAST version 4.03.

CHAPTER 4
RESULTS AND DISCUSSION

4.1 pH and titratable acidity of paddy rice fermenting steep water

Changes in pH and titratable acidity of the fermenting steep water have been attributed to variation in bacteria colonies responsible for fermentation at the different stages of fermentation (Qianqian *et al.*, 2017). As observed by Holzafpel (1997), changes in the pH could directly be linked to the activity of lactic acid bacteria that produce acids as metabolites. As such different compounds produced by this microorganism could act as precursors in the production of flavour compounds that gave Ofada rice its relish taste and aroma and sometimes the off-flavour compounds.

Physiology, metabolism and genetic properties of microorganisms are also major determinants of the dynamics of fermentation processes (Ibragimova, 1980). Therefore the knowledge of microorganisms, substrates and conditions of growth are of major importance to control the production of metabolites (Giraffa, 2004). Microbial diversity, growth cycle, taxonomic identity, quantitative changes, and spatial distribution of microbial species are key factors to be understood at every stage of food fermentation (Fleet, 1999). This is to ensure effective management of the growth and activities of microorganisms in fermented foods.

The result of pH of fermenting steep water of Ofada rice with different steeping duration and initial steeping temperatures is as presented in Table 4.1. The pH of fermenting steeping water decreased with fermenting days. The highest pH of 6.94 was recorded on day zero with the initial steeping temperature of 100^{o}C, while the lowest of 4.94 was recorded on day five of steeping with 100^{o}C initial steeping temperature. The highest pH recorded in this study was lower compared to that obtained by Adeniran *et al.,*(2012) who reported 7.57 for paddy rice steeped at ambient temperature without inoculation with a starter culture. The difference could be due to the pH of the water used for the steeping operation.

Table 4.1. Changes in pH of fermenting steep water of Ofada rice at varying steeping duration and initial steeping temperature

	Steeping Duration (Days)					
Initial steeping temperature (^{o}C)	0	1	2	3	4	5
30	6.91 ± 0.03^{a}	5.84 ± 0.05^{ab}	5.79 ± 0.04^{c}	5.60 ± 0.00^{c}	5.20 ± 0.00^{b}	5.15 ± 0.00^{b}
65	6.91±0.04^{a}	5.87± 0.00^{b}	5.45 ± 0.02^{b}	5.35 ± 0.00^{b}	5.18 ±0.01^{b}	5.14 ±0.01^{b}
100	6.94 ± 0.00^{a}	5.78 ± 0.01^{a}	5.07 ± 0.03^{a}	5.01 ± 0.02^{a}	4.97 ± 0.02^{a}	4.94 ± 0.00^{a}

Means in the same column withthe same superscript have no significant difference ($p > 0.05$)

There was no significant difference ($p>0.05$) in the pH of fermenting steep water on day zero at the different initial steeping temperatures (30, 65 and 100^{o}C) used in this study. However, there were significant differences in the pH of the fermenting steep water at $p<0.05$ observed after day one of steeping throughout the entire duration of steeping. At 30^{o}C initial steeping temperature, the highest pH of the fermenting steep water was 6.91 while the lowest pH was 5.15 after five days of steeping. For paddy Ofada rice steeped at ambient temperature, Adeniran *et al*., (2012) also reported the highest and lowest pH as 7.00 and 5.84 respectively. At 65^{o}C initial steeping temperature, the highest pH of paddy rice fermenting steep was 6.91 while the lowest was 5.14 after five days of steeping. After a day of steeping, there was a significant difference in pH of paddy rice fermenting steep water between Ofada paddy steeped at 100^{o}C (initial steeping temperature) and other initial steeping temperatures (30^{o}C and 65^{o}C). This could be due to the rapid commencement of fermentation when paddy was steeped at 100^{o}C.

The result of titratable acidity is presented in Table 4.2. The lowest titratable acidity (0.05 mg/mL) was found on day zero of steeping while the highest (0.44 mg/mL) was obtained on day five with an initial steeping temperature of 100^{o}C. These were not in the range of results obtained by Adeniran *et al*., (2012) who reported the lowest and highest value of titratable acidity as 0.54 and 6.31 mg/mL when Ofada paddy rice was steeped at ambient temperature. For 30, 65 and 100^{o}C initial steeping temperature, increases in titratable acidity were observed as the steeping duration increases. The highest titratable acidity of the paddy rice fermenting steep water for 30, 65 and 100^{o}C initial steeping temperature were 0.36, 0.44 and 0.28 mg/mL respectively. Values in this range were reported when starter culture was used during paddy steeping in the work of Adeniran *et al*., (2012); who obtained between 0.20 and 0.48 mg/mL titratable acidity after 48 h of paddy steeping.

A significant difference ($p < 0.5$) was observed in the titratable acidity of treatments with the initial steeping temperature of 30^{o}C, 65^{o}C and 100^{o}C starting from the first day of steeping to the fifth day of steeping. For all treatments, titratable acidity increases as pH decrease in the paddy rice fermenting steep water. This was also reported in the study of Ogi fermentation (Omemu, 2011) and the work of

Table 4.2. Changes in titratable acidity in lactic acid mg/mL of fermenting steep water of Ofada paddy at varying steeping duration and initial steeping temperature

	Steeping Duration (Days)					
Initial steeping temperature (^{o}C)	0	1	2	3	4	5
30	0.05 ± 0.00^{a}	0.06 ± 0.00^{b}	0.14 ± 0.01^{b}	0.22 ± 0.00^{b}	0.28 ± 0.01^{b}	0.36 ± 0.01^{b}
65	0.05 ± 0.00^{a}	0.06 ± 0.00^{a}	0.18 ± 0.00^{b}	0.22 ± 0.00^{b}	0.36 ± 0.01^{c}	0.44 ± 0.01^{c}
100	0.05 ± 0.00^{a}	0.08 ± 0.00^{c}	0.11 ± 0.00^{a}	0.18 ± 0.00^{a}	0.23 ± 0.00^{a}	0.28 ± 0.01^{a}

Means in the same column withthe same superscript have no significantdifference ($p > 0.05$)

Adeniran *et al*., (2012) on the use of starter cultures during paddy rice steeping. According to Efiuvwevwere and Akona (1995) and Zvauya *et al*. (1997), anincrease in acidity and a subsequent decrease in pH when cereals are being fermented could be linked to the actions of yeasts and lactic acid bacteria on free sugars present in the cereal. Factors like temperature, pH, inoculums, size, type of cereal and propagation time determine the type of microorganisms that is found in association with each type of fermentation (Achi and Ukwuru, 2015). As such, fermenting organisms acting on a particular substrate at a particular time is dependent on the factors mentioned.

Variation in the microbial composition as the steeping duration increases was attributed to changes in pH and acidity (Adeniran *et al*., 2012). The knowledge of this dynamics is a major tool that could help for starter culture used during Ofada rice steeping in order to improve its sensory attribute. The use of starter culture containing lactic acid bacteria and yeasts during Ofada rice steeping has been reported to have reduced steeping duration and also improve the nutritional profile of Ofada rice (Adeniran *et al*., 2012). All these variations in the fermenting microbes were reported to be responsible for the changes in the pH and acidity of products. Thus, reflecting numerous complexities associated with the formation of metabolites that gives the products their characteristics.

4.2 Bacteria occurrence in fermenting steep water

Identification and characterisation of microbes that determine the characteristic properties of foods during the fermentation process are of great importance. This is to allow control of the process through selection of appropriate conditions for the process, and so as to be able to use the right starter culture to improve the technology of processing. This also enables control of the metabolites production.

With respect to percentage occurrence, proteobacteria had the highest at 30 and 65^{o}C initial steeping temperature while the firmicutes were the highest percentage at 100^{o}C initial steeping temperature. In the fermenting steep water of paddy Ofada rice, present in high percentage were microbial phylum of proteobacteria, Actinobacteria and firmicutes. While minor phylum presents were chloroflexi, Bacteriodetes, Elusimicrobial and acidobacteria (see appendix 4).

At 30^{o}C initial steeping temperature, percentage of Proteobacteria present in the fermentation medium decreases as the no of days of fermentation increases, but for firmicutes, there is increase in the percentage of firmicutes presents as the days of steeping increases. Increase in percentage of bacterioidetes from 0.04 to 1.24 also occurs as the steeping duration increases at initial steeping temperature of 30^{o}C. At 65^{o}C initial steeping temperature, percentage of Proteobacteria (81.90) was highest after five days of paddy rice steeping. While the lowest for proteobacteria was after second day of paddy steeping with 28.18% of proteobacteria present. Actinobacteria showed a decrease in percentage from 27.06% to 0.06% at 65^{o}C initial steeping temperatures. Firmicutes has its highest percentage after the second day of paddy steeping for the 65^{o}C initial steeping temperature treatment.

Adeniran *et al*., (2012) observed that the onset of paddy rice steeping (0 h), the yeasts that were present were *Candida mesenterica, Saccharomycescerevisiae* and *Torulospora delbrueckii* while the bacteria were *Lactobacillus maltaromicus*, *Lactobacillus kefir, Lactobacillusamylophillus* and *Pediococcus halophillus*. At the 24-h mark of steeping, *Leuconostoc mesenteroies, Pediococcushalophillus* and *Saccharomyces uvarum* were found in the steep-water in addition to the former isolates, while *Lactobacillusalimentarius* disappeared. By 48h, *Lactobacillusfarciminis, Saccharomycodes sinensis* and *Pichia angust*a appeared in the water and the latter two remained till the end of the fermentation while *Lactobacillus maltaromicus*, *Lactobacillus kefir*, *Candida mesenterica*, *Torulospora delbrueckii* and *Pedicoccus halophilus* disappeared.

At 100^{o}C initial steeping temperature, proteobacteria, actinobacteria and firmicutes were the only detected phylum in the fermenting medium. Firmicutes have the highest percentage of occurrence of 99.96% after fourth day of fermentation while proteobacteria has its highest percentage of 0.40% after two days of paddy steeping. The lowest percentage (99.58%) of firmicute was observed after two days of paddy steeping while the lowest for proteobacteria was 0.04% after fourth day of paddy steeping. Actinobacteria were only detected after the second and third day of steeping with percentage occurrence of 0.015 and 0.01% respectively. Generally, the higher the percentage of protobacteria, the lower the firmicutes present in the treatments.

Actinobacteria were mostly present at a significant percentage at initial steeping temperature of 65^{o}C after first and second day of paddy steeping. Percentage occurrence of protobacteria decreases as the initial paddy steeping temperature increases, while the percentage occurrence of firmicutes increases as the initial steeping temperature increases from 30 to 100^{o}C.

Variation in the microbial composition as the steeping duration increases was attributed to changes in pH and acidity (Adeniran *et al*., 2012). The knowledge of this dynamics have been used to compose starter culture used during Ofada rice steeping in order to improve its sensory attribute. The use of lactic acid bacteria and yeasts during Ofada rice steeping have been reported to have reduced steeping duration and also improve the nutritional profile of Ofada rice (Adeniran *et al*., 2012).

In a study that characterised bacteria and yeasts responsible for the paddy rice fermentation by Adeniran *et al*. (2012), it was reported that the dominant lactic acid bacteria found to be responsible for the fermentation of "Ofada" rice were *Leuconostoc mesenteroides* and *Lactobacillusamylophillus*, while *Saccharomyces cerevisiae* and *SaccharomycesUvarum* were the dominant yeasts in the fermenting steep water. Seven species of LAB detected during Ofada rice fermentation includes *Lactobacillus maltaromicus, Lactobacillus alimentarius, Lactobacillus kefir, Pediococcus acidophilus,Lactobacillus amylophillus and Lactobacillus farciminis* and *Leuconostoc mesenteroides*. Whereas six species of yeasts detected were *Saccharomycodes sinensis,Saccharomyces uvarum, Pichia angusta, Candida mesenterica,Torulaspora delbrueckii, S. cerevisae.*

4.2.1 Proteobacteria dynamics in paddy rice fermenting steep water

Among all proteobacteria detected, higher percentage of *Acinetobacter*, *Azotobacter*, *Pseudomonas*, *Enterrobacter*and *Citrobacter*were recorded in fermenting steep water. *Acinetobacter*, *Enterobacter*, *Methylobacterium*and *Citrobacter*were not found in the fermenting steep water for the first day of steeping when the initial steeping temperature was 30^{o}C. However, they were found at high percentages when the initial steeping temperature was 65^{o}C. This could be that *Acinetobacter*, *Enterobacter*, *Methylobacterium*and *Citrobacter*are not active at ambient temperature.

The percentage of all proteobacteria found in this work reduced significantly when the initial steeping temperature is 100^{o}C. The high temperature of steeping could have destroyed a greater percentage of the proteobacteria present. At 30^{o}C initial steeping temperature, percentage of *Acinetobacter* increase as the duration of steeping increase up to the fourth day and later decrease on the fifth day of steeping. Whereas, *Enterobacter*, *Azotobacter*, *Methylobacterium*and citrobacter had a decrease in their percentages as the numbers of fermenting days increases irrespective of the initial steeping temperature.

For example, Kantachote *et al*., (2008) in a study on fermentation dynamics of wild forest noni (*Morinda coreia* Ham) reported initial increase of lactic acid bacteria for the first 7 d of fermentation and its dominance for 21 d before yeast took over the dominance in the fermentation media. In the study, *Lactobacillus plantarum* and *Saccharomyces cerevisiae* were identified as the the dominant lactic acid bacteria and yeast respectively.

According to Kantachote *et al*., (2008) *Lactobacilluspentosus* was only detected on the 21st day of fermentation while it was also reported that *Saccharomyces cerevisiae* was originally the dominant yeast but was later replaced by *Pichia membranifaciens* followed by *Pichia anomala*. All this variations in the fermenting microbes were reported to be responsible for the changes in the pH and acidity of products. Thus, reflecting numerous complexities associated with the formation of metabolites that gives the products its characteristics.

At 100^{o}C initial steeping temperature, all the proteobacteria except acinetobacter were not found in the fermenting steep water at a significant percentage throughout the fermenting days. This could be due to the sensitivity of proteobacteria to high temperature. However, acinetobater had a decrease in percentage in the fermenting steep water at 100^{o}C initial steeping temperature as the fermenting duration increases. As the fermentation duration increases, the presence of fermenting microbes which include lactic acid bacteria and yeast led to the removal of enteric microorganism (Kingamkono *et al*., 1995; Olasupo *et al*., 1997).

Adelekan and Nwadiuto (2012) in a bacteria dynamic study during production of "iru" (locust bean) through the fermentation of african locust bean reported the dominance of *Bacillus anthracis* relatives, *Bacillus cereus and Bacillus* sp. *a*t 0 h, 24 h and 48 h of fermentation respectively. In the same work the presence of *Enterobacter*sp. was found only after 72 h of feremtation. Each stage of fermentation has been reported to possees differences in microbial community and this lieu is responsible for the characteristic aroma and texture of the food product. In the same vein, during "Ogi" fermentation, it was reported that as the population of the filamentous mould decreases, the LAB population increases within 2 d(Omemu, 2011). This variation is significant in the production of metabolites as they are regulated by microbial activity and this leads to the modification of the composition of the product. The work of Vieira-Dalode (2007) on identifying microoganisms responsible for *gowe* production from sorghum reported a mixed fermentation of sorghum which was dominated by *Lactobacillusfermentum*, *Weissellaconfusa*, *Pediococcusacidilactici*, *Kluyveromyces marxianus*, *Pichia anomala*, *Candida krusei.*

4.2.2 Actinobacteria dynamics in paddy rice fermenting steep water

When the initial steeping temperatures were 30 and 65^{o}C, all the actinobacteria present in the fermenting steep water decreased in percentage as the number of fermenting days increases. The disappearance of *actinobacteria* could be attributed to the increase in the fermenting microflora as the number of fermenting days increase as was also reported by Adeniran *et al*.,(2012). However, there was an exception for the first day of steeping at initial steeping temperature of 30^{o}C which most of the actinobacteria were not detected. At 100^{o}C initial steeping temperature, it was observed that aside sacharopolyspora (which was found on the first day of steeping) and *Propiniobacteria* (which was detected on the second and fifth day of steeping) there was no other actinobacteria found in the fermenting steep water. This could be attributed to the effect of high temperature in eliminating heat sensitive microbes.

4.2.3 Firmicutes dynamics in paddy rice fermenting steep water

*Lactobacillus, Bacillus, Paenibacillus, Brevibacillus*and *Aneurinibacillus*were the prominent firmicutes in the fermenting steep water of Ofada paddy rice used in this work. Firmicutes were not found in the fermenting steep water for the first day of

steeping when the initial steeping temperature was 30^{o}C. It was also observed in this work that, as the steeping duration increases, the percentage of *Lactobacillus*and *Bacillus*present in the steep water decreases. Paenibacillus had an increase in percentage from first day of steeping to second day before having a continuous decrease until the fifth day of paddy rice steeping. This was also observed for *Brevibacillus*and *Aneurinibacillus*. *Bacillus* has been reported to produce pyrazine (Demyttenaere *et al.*, 2002). *Paenibacillus polymyxa* has been reported to be responsible for the production optically active (*R,R*)-2,3-butanediol from carbohydrates (Ui *et al*., 1983; Marwoto *et al*., 2002).

Greater percentages of *Bacillus*, *Paenibacillus*, *Tumebacillus*, *Brevibacillus*and *Aneurinibacillus*were found in paddy rice fermenting steep water with the initial steeping temperature of 100 ^{o}C. Whereas, greater percentage of *Lactobacillus*and *Leuconostoc*were reported when paddy rice was steeped at initial steeping temperature of 65 ^{o}C. Significance of lactic acid bacteria has been reported to be responsible for cereals fermentation (Edema and Sanni, 2008). This is also confirmed in this work considering the presence of *Lactobacillus*and other *Bacillus*group. Adeniran *et al.,* (2012) in a study to characterize microbial isolates responsible for Ofada rice paddy fermentation reported *Leuconostoc mesenteroides* and *Lactobacillus amylophillus* as the prominent lactic acid bacteria. This could be compared to what was obtained in this work despite the fact that molecular method of microbial identification was used in this work while Adeniran *et al*., (2012) used the culture method.

Omemu *et al.,* (2007) observed that flavour compounds in fermented foods can be attributed to the activity of Lactic acid bacteria (LAB) and yeasts. Flavour development in Ofada rice has been linked to the fermentative activity of microorganism during the steeping period and these has been reported to give Ofada rice its unique relish flavour (Adeniran *et al.*, 2012; Otegbayo *et al.*, 2001).Walsh *et al.,* (2016) work on fermented dairy beverage kefir observed that there is a gene in *Leuconostoc mesenteroides* that carries out the biosynthesis of aromatic amino acid. The work of Walsh and co-workers also correlated *Lactobacillus*spp. with the presence of carboxylic acids, esters and ketones while the presence of *Leuconostoc*spp. was linked to the production of acetic acid and 2,3-butanedione.

Ayelogun and Adegboyega (2000) reported that steeping rice paddy for too long leads to unacceptable colour, taste, and odour of rice due to fermentation processes. Otegbayo (2001) reported that steeping contributes mainly to organoleptic, physical and nutritional changes in rice. Bacteria, yeast and moulds being mainly responsible for the organoleptic properties. Production of flavour compounds and aroma are made possible through fermentation processes carried out by yeasts and lactic acid bacteria (Omemu, 2007). The presence of fermented odour in Ofada rice as reported by Anuonye *et al.,* (2017), was successfully removed by adopting hot water steeping method. In the work of Adeniran *et al.,* (2012), use of starter cultures reduced the steeping time of "Ofada" rice and it also enhanced the nutrient composition and improved the sensory quality of the rice. This showed the role being played by microorganism in defining the Ofada rice flavour.

4.3 Antioxidant properties of Ofada rice

Total phenolic content, total antioxidant capacity, total flavornoid content, ferric reducing antioxidant property (FRAP) and 2,2-di (4-tetradyphenyl)-1-phenylhydrazyl (DPPH) assays revealed antioxidant property of the processed Ofada rice and the result is as presented in Table 4.3

4.3.1 Total phenolic content of Ofada rice

The total phenolic content (TPC) of Ofada rice processed at different processing conditions varied between 25.42 and 62.66 mgGAE/g with an average value of 50 mgGAE/g. The highest value of 62.66 mgGAE/100g of TPC was obtained at first day of steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 50^{o}C drying temperature. Whereas, steeping for five days at 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature gave the lowest value of TPC of 25.42mgGAE/100g. In a study of 133 rice cultivars by Goffman and Bergman (2004), the lowest value obtained for soluble total phenolic content was 69 mg gallic acid eq./100 g in brown rice group while the highest were obtained in the red and purple groups were 213 and 274 mg gallic acid eq./100 g respectively.

Table 4.3. Antioxidant properties of the Ofada rice samples

Run	Steeping duration (Days)	Initial Steeping Temperature(°C)	Parboiling Temperature (°C)	Drying Temperature (°C)	TPC mgGAE/100g	TFC mgQuercetin/g	TAC mgGAE/g	FRAP mgTrolox/g	DPPH I50 (µg/mL)
2	1	30	80	50	58.19^{i}	0.98^{def}	3.38^{ij}	26.89^{fghi}	864.45^{efgh}
6	1	30	100	70	46.18^{gh}	0.73^{abcd}	3.99^{j}	24.53^{bcdef}	706.04^{bcd}
15	1	30	120	30	36.08^{cd}	0.74^{abcd}	1.90^{def}	24.79^{bcdef}	685.74^{bcd}
24	1	30	120	30	57.66^{i}	0.91^{abcdef}	3.27^{ij}	26.79^{efghi}	422.19^{bc}
8	1	65	100	50	51.40^{ij}	0.80^{abcde}	1.78^{cde}	26.42^{defghi}	277.89^{a}
11	1	65	120	70	48.93^{hi}	0.78^{abcde}	1.21^{abcd}	26.27^{defghi}	717.83^{bcd}
18	1	100	80	70	52.46^{ijk}	0.72^{abcd}	0.54^{ab}	20.01^{a}	1372.78^{h}
20	1	100	80	30	46.50^{gh}	0.71^{abc}	0.69^{ab}	24.01^{bc}	665.81^{bc}
25	1	100	80	30	45.84^{gh}	0.75^{abcd}	3.18^{hij}	23.18^{bcde}	1171.15^{ghi}
13	1	100	120	50	62.66^{m}	0.92^{bcdef}	2.16^{defg}	27.82^{ghij}	1224.58^{gh}
16	3	30	100	50	41.34^{ef}	0.70^{abc}	3.27^{ij}	25.88^{cdefgh}	836.24^{cde}
12	3	30	120	70	56.30^{kl}	0.81^{abcde}	3.21^{hij}	28.67^{ij}	615.22^{bc}
7	3	65	100	70	50.11^{hi}	0.75^{abcd}	0.25^{a}	28.90^{ij}	1011.27^{defg}
14	3	65	100	40	55.61^{jkl}	0.89^{abcd}	0.86^{abc}	25.08^{bcdefg}	730.61^{bcd}
5	3	100	100	50	42.38^{efg}	0.66^{ab}	0.50^{ab}	23.63^{bcd}	681.71^{bcd}
9	5	30	80	30	39.39^{de}	1.50^{g}	0.72^{ab}	19.07^{a}	1076.61^{cdefg}
10	5	30	80	30	39.13^{de}	1.42^{g}	2.26^{efgh}	22.62^{b}	910.93^{i}
17	5	30	80	70	33.8oC	1.04^{ef}	0.39^{a}	24.13^{bcdef}	651.93^{bc}
1	5	30	120	50	46.26gh	0.66^{a}	2.88^{ghi}	28.44^{hij}	815.87^{cde}
21	5	65	100	50	25.42^{b}	0.75^{abcd}	1.84^{de}	19.28^{a}	876.01^{cdef}
23	5	100	80	50	33.59^{c}	0.9^{6cdef}	2.81^{fghi}	33.43^{k}	613.11^{bc}
3	5	100	120	70	43.98^{fg}	1.09^{f}	0.33^{a}	32.46^{k}	884.11^{efgh}
4	5	100	120	30	52.63^{ijk}	0.86^{abc}	0.26^{a}	32.31^{k}	586.09^{bc}
19	5	100	120	30	13.92^{ijk}	0.71^{abcdef}	1.8oC^{de}	25.22^{bcdef}	1122.9^{ghi}
22	5	100	120	70	45.53^{fgh}	1.39^{g}	1.46^{bcde}	29.6^{4j}	586.72^{bc}

Means in the same column having same letter are significantly not different ($p > 0.05$)

TPC- Total phenolic Content ; TAC- Total antioxidant Capacity; TFC- Total flavonoid Content; DPPH- 2,2-di(4-*tert*octylphenyl)- 1-picrylhydrazyl (DPPH) assay; FRAP - ferric reducing antioxidant po

The highest value obtained in this work is lower than those obtained in the work of Goffman and Bergman (2004), but still within the range of rice classified as the brown group. However, the highest TPC obtained in this work is higher than that of polished rice (56.34 mgGAE/100g) reported by Ti *et al.,* (2015) but lower than that obtained for brown rice (174.04 mgGAE/100g) in the same report on the effect of cooking on TPC of rice.

Generally, significance difference ($p<0.05$) was observed by synergy of variation in the parboiling and drying temperature. This is in congruence with the work of Zielinski *et al*., (2001) on variation in phenolic contents due to temperature changes. Min *et al*., (2014) also observed a significant decline of about 16-91% in the amount of free phenolic contents in rice cultivars due to hydrothermal processing. Effect of temperature on TPC is connected to polymerization of phenolic molecules and decarboxylation which is catalysed by heat treatments (Kadiri, 2017).

There was significance difference at $p<0.05$ associated with the variation in steeping duration. Treatments with steeping duration of 1 day has the highest TPC value of 62.66 mgGAE/100g followed by treatments with 3 days steeping which has its highest TPC at 56.30 mgGAE/100g while 5 days steeping has its highest TPC value of 53.92 mgGAE/100g.. Also the overall lowest TPC value 25.42 mgGAE/100g was obtained with 5 days steeping treatments. This showed that the duration of steeping may be considered as one of the factors that can influence the TPC content of processed rice. Therefore, it could be opined from this work that fermentation has a decreasing effect on the total phenolic contents. Although, increase in TPC was reported after fermentation of some cereals and legume (Seki *et al*., 2008; lee *et al*., 2008; Wang *et al.*, 2014), decrease observed in this work might be due to the fact that greater percentage of phenolics are stored in the bran of rice and thus removed during dehulling and debranning.

Adjusted R^2, R^2, and adequate precision are presented in Appendix 6 while model coefficient showing the influence of treatment conditions on TPC is presented in Appendix 7. The values presented for TPC showed lack of fit of the model for TPC. This is an indication that processing conditions of initial soaking temperature, soaking duration, parboiling temperature and drying temperature does not have significant

effect on TPC of milled Ofada rice. However, from the model, increase in soaking duration and initial steeping temperature negatively correlated with the TPC of rice samples. Whereas increasing drying and parboiling temperatures increases the TPC of rice samples.

Different flavour response such as astringent and bitter taste is associated to the presence of phenolic compounds in cereals. Change in concentration of total phenolic content has been linked to processing (Soto-Vaca *et al*., 2012). Thermal processes has been reported to have significant reducing effect on the concentration of total phenolic content in cereals (Zielinski *et al*., 2001; Finocchiaro *et al*., 2007; Parra *et al*., 2007). This is largely due to changes in the form of phenolic compounds present or other chemical reactions such as polymerization, oxidation and appearance of Maillard reaction products. This could be linked to the observation of Heiniö *et al*., (2008) who observed that the desirable and undesirable flavours in cereals are contributed by their phenolic content. Challacombe, Abdel-Aal, Seetharaman and Duizer (2012) correlated the perceived bitterness of whole wheat bread and crackers to the total phenolic content.

Flavour of cereal products are largely influenced by acids (amino and organic), as well as phenolic components that are non-volatile (Dimberg *et al.*, 1996; Peterson, 2001). Ferulic acid, which is a phenolic acid that influence flavour is abundantly present in cereals. Dimberg *et al.,*(1996) noted thatdespite the low percentage of free phenolic acids in cereals, their presence might have significant effecton flavour perception.

Phenolic compounds are derivatives of tyrosine or phenylalanine identified as plant secondary metabolites. Structurally, they are made up of aromatic group having one or more hydroxyl functional groups attached to the aromatic ring (Shahidi and Naczk, 2003). Simple phenolic acids are derivatives of cinnamic or benzoic acid while tannins and flavonoids belong to the complex family. Different flavour perceptions emanate from phenolic and polyphenolic compounds. This is in the range of the sweet chalcones and some glycosylated flavonoids to bitter and astringent tannin compounds. Phenolics can be closely linked with the nutritional and sensory quality of fresh and processed plant foods. However, Dimberg *et al*., (1996) and Peterson

(2001) reported that cereals flavour is influenced by the presence of non-volatile phenolic compounds. Naturally occuring phenolic compounds in spices and herbs usually serve the dual purpose as food preservatives and flavourants (Soto-Vaca *et al*., 2012).

For example, Dimberg *et al*., (1996) observed that though the percentage of free phenolic acids in cereals may be small (10- 90 mg/kg), however they have a strong influence on the perceived flavour in oat. The perception of bitter taste in cereal products has been linked to the presence of phenolic compounds such as syringic and pinoresinol acid (Heiniö *et al*., 2008). Flavour activeness of phenolic is dependent on its nature of availability. For example, free phenolics are perceived in food due to ease of adherance to the taste receptors whereas this is not so when in bound form.

More generally, polyphenolic compounds are known to impact astringent or bitter flavours. And as such much of the research interest on the compounds were focused on its flavour properties. Flavour imparted by phenolic compounds is of major importance in isolated proteins, wine and beer, as well as vegetables. The presence of this flavour compound could be both desirable and undesireable. Although the desireability or otherwise is largely dependent on the the concentration of the compound of interest and the type of food.

The extent of polyemerization of phenolic compounds affects the concentration and duration of their bitter or taste responses (Peleg *et al*., 1999). Although, eugenol and vanillin which belong to the asimple molecules group have a strong and characteristic flavour.However, bitter and astringent flavours are generally associated with polyphenolic compounds. The work of Callemien and Collin (2009) reportedthe numerous impact of phenolic compoundson beer, and other papers have also discussed their importance on wine flavour (Waterhouse, 2002; Cheynier *et al*., 2006; Monagas *et al*., 2005). Phenolic compounds contribute to food flavors in multiple ways. In addition, and because of their antioxidant properties, they participate in the lipid oxidation process that takes place in many foods upon processing

4.3.2 Total flavonoid content of Ofada rice

Total flavonoid content has been associated with a taste response of astringency and bitterness in cereals (Kobue-Lekalake, Taylor and De-Kock, 2007; Kobue-Lekalake, 2008). In this work, total flavonoid content of processed rice range from 0.66 to 1.50mgQuercetin/g. Paddy steeping for five days at 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature gave the highest flavonoid content of 1.50 mgQuercetin/g. The lowest value of 0.66 mgQuercetin/g was obtained at three days steeping, 100^{o}C initial steeping temperature, 100 ^{o}C parboiling temperature and 50^{o}C drying temperature. This result agrees with the observation of Wang *et al.*, (2014) who noted that fermentation improves the amount of TFC while a significant decrease in TPC occurs with thermal treatment as reported by Georgetti *et al.,* (2009) during tartary buckwheat flour thermal processing. Cooking as a thermal treatment was reported to cause a significance decrease in the concentration of TFC in brown and polished rice (Ti *et al.*, 2015).

The highest value for 1day steeping with variation in other processing conditions was 0.98mgQuercetin/g while the lowest was 0.71 mgQuercetin/g. The highest value for 3 days steeping treatments was 0.89 mgQuercetin/g while the lowest was 0.66 mgQuercetin/g. The highest value of the TFC for 5 d steeping with variation in other processing conditions was 1.50 mg while the lowest value was 0.71mgQuercetin/g. There was no significant different at $p>0.05$ among treatments that were steeped for 1 and 3d. This could be that the effect of thermal treatments had no effect on the variation in the TFC caused by steeping duration. Whereas for treatments that involves 5 d steeping, significant difference was observed at $p<0.05$.

As presented in Appendix 6 and 7, closeness of R^2 and Adjusted R^2 to 1 in the value for TFC of Ofada rice showed the dependence of TFC of Ofada rice on the pre-treatment conditions in this study. The value of adequate precision greater than 4 is an indication of the degree of fitness of the model. Positive coefficient of soaking duration on TFC is an indication of positive influence on the TFC while initial soaking temperature, parboiling temperature and drying temperature has negative influence on the TFC of milled Ofada rice since the coefficient of the model is negative.

4.3.3 Total antioxidant capacity of Ofada rice

Total antioxidant capacity (TAC) of Ofada rice varied from 0.25 to 3.99 mgGAE/g. The highest for TAC obtained for 1day steeping treatments was 3.99 mgGAE/g, while the lowest was 0.54 mgGAE/g. For three days steeping treatments, the highest TAC obtained was 3.27 mgGAE/g while the least value was 0.25 mgGAE/g. And for 5 days steeping treatments, the highest TAC value was 2.88 mgGAE/g while the lowest was 0.26 mgGAE/g. There is significant differences (at $p<0.05$) among the highest TAC obtained for different steeping duration.

The highest value (3.99 mgGAE/g) for TAC was obtained at 1day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature. Paddy steeping for 3 d at 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature had the least value of 0.25 mgGAE/g,. Significant differences ($p<0.05$) were observed among samples with 1, 3 and 5 d steeping with other processing conditions varied.

The value of R^2 and adequate precision value for TAC were 0.72 and 5.23. Only parboiling temperature has positive effect on the TAC of Ofada rice while other treatment conditions (Initial soaking temperature, parboiling temperature and drying temperature) has negative effect on TAC of milled Ofada rice. However, Initial soaking temperature possess the highest negative influence on the model as presented in Appendix 6 and 7.

Studies on the effect of cooking and boiling on the total antioxidant capacity of red, black and white variety of rice by Zaupa *et al*.,(2015) reported a significant decline in the total antioxidant capacity after the hydrothermal processes. Correlated to the antioxidant property are flavonoids, tannins and phenolic acid contents (Kadiri, 2017). Decrease in the TAC in this work may be due to the decrease in the TPC which is caused by hydrothermal treatment of Ofada rice. Chmiel *et al.,* (2017) reported a strong positive correlation for TPC and TAC in cooked rice. This correlation was also reported for rice grains by other researchers (Surh and Koh, 2014; Walter *et al.,* 2013).

Antioxidants are added to fats and oils or foods containing fats to prevent the formation of various off-flavors and other objectionable compounds that result from the oxidation of lipids (Namiki, 1990). Most natural antioxidants are phenolic in nature. The antioxidant activities of phenolic compounds have been recognized for decades, and research and development on the use of natural substances or food ingredients containing phenolic antioxidants will continue to be of great interest to the food industry (Ho, 1992).

4.3.4 Ferric reducing antioxidant property of Ofada rice

Ferric reducing Antioxidant property (FRAP) of Ofada Rice results varied from 19.07 to 33.43 mgTrolox/g. The highest FRAP was obtained at five days paddy steeping with 100^{o}C, 80^{o}C and 50^{o}C initial steeping, parboiling and drying temperatures repectively. The lowest result for FRAP (19.07 mgTrolox/g) was obtained when paddy rice was steeped for five days, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. FRAP highest and lowest result for 1day paddy rice steeping treatments were 26.89 and 20.01 mgTrolox/g respectively. The highest and the lowest FRAP for three days paddy steeping treatments were 28.90 and 23.63 mgTrolox/grespectively. While for five days paddy steeping treatments, the highest and lowest FRAP were 33.43 and 19.07 mgTrolox/g respectively.

There were significant differences observed among treatments at $p<0.05$. However, significant difference ($p>0.05$) does not exist between the highest FRAP values for one and three days treatments. Whereas there was significant difference between the highest value for one and three days steeping treatments, and five days steeping treatments. FRAP was reported to have decreased significantly with fermentation of adlay, chestnut, lotus seed and walnut (Wang *et al*., 2014). On the contrary in this present study, the highest value of FRAP obtained for five days fermentation was higher than three days and that of three days was higher than one day steeping treatments. This difference may be due to the fact that Wang *et al*., (2014) only worked on legumes while the present work is on cereal. Although, Kadiri (2017) noted that germination leads to increase in FRAP

All the pre-treatment conditions had positive effect on FRAP value obtained milled rice samples. However, the values of R^2, Adjusted R^2, and adequate precision of the model presented in Appendix 6 and 7 showed that processing conditions of Ofada rice does not significantly influence the FRAP value of milled Ofada rice.

4.3.5 DPPH of Ofada rice

The DPPH was measured in I50 (µg/mL). The lowest I50 of 277.89 µg/mL was obtained from treatments with one day steeping 65^{o}C, initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature. The highest value of DPPH of 1372.78 µg/mL was obtained from rice processed with the following conditions: 1day steeping 100^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 70^{o}C drying temperature. The highest and least value of I50 for three days treatments were 1011.27 and 615.22 µg/mL respectively while for five days paddy steeping treatments, the highest and the least value were 107.661 and 613.11 µg/mL respectively.

Treatments had significant differencesat $p<0.05$ among a day steeping results of DPPH. There was no significant difference among treatments for 3 and 5 d paddy steeping. Wang *et al.,* (2014) reported a significant decline in DPPH due to fermentation in adlay, chestnut, lotus seed and walnut. However, in this study, significant differences were observed mostly due to processing temperature variation. This could be attributed to the influence of thermal treatments on the process of polymerization, decarboxylation and strong complexes formation of free phenolics (Massaretto *et al.*, 2011).

The result of R^2, Adjusted R^2, and adequate precision of the model (Appendix 6 and 7) showed lack of fitness of the model in depicting the effect of processing conditions on the value of DPPH of milled Ofada rice.

4.3.6 Optimisation of flavonoid, phenolic and antioxidant properties of Ofada rice

Result showing the optimisation and the level of desirability for flavonoid, phenolic and antioxidant properties of Ofada rice is presented in Appendix 8. Maximum TPC,

maximum TFC, maximum TAC, maximum FRAP and minimum DPPH were used for the optimisation. The desirability obtained consists of ten solutions which ranged from 0.52 to 0.64. The most desirable processing conditions 1 day soaking, 35 ^{o}C initial soaking temperature, 80 ^{o}C parboiling temperature and 38.14^{o}C drying temperature. The selected desirable solution is characterised with reduced processing duration and temperature.

4.4 Amino acid profile of Ofada rice

Amino acid present in a food sample has been reported to have significant effect on its organoleptic properties.Average percentage of amino acids detected were asparagine (3.141), Cysteine (0.016), Aspartic acid (2.450), Glutamic acid (1.644), Serine (1.362), Glycine (1.401), Histidine (0.010), arginine (0.013), threonine (2.851), Alanine (16.995), Proline (9.826), tryprtophan (0.007), Methionine (1.697) and phenylalanine (0.009). Amino acids found at low percentages were cysteine, histidine, arginine, tryptophan, methionine and phenylalanine, while Asparagine, glutamic acid, Alanine, proline and threonine were found at relatively higher percentages in processed Ofada rice. This relative percentage of abundance has also been reported by other researchers with respect to different rice cultivars undergoing varying processing conditions (Matsuzaki *et al.*, 1992; Kamara *et al.*, 2010).

Rice quality in terms of nutrition is valuable for its protein content and the balance of essential amino acids, however, free amino acids (FAAs) may be responsible for the perceived flavour or act as precursors for flavour production. FAA profile has been successfully used for discrimination of variety and origin of natural foods in food authentication (Kamara *et al.*, 2010; Maro *et al*., 2011; Cometto *et al.*, 2003). In higher plants, amino acids serve as precursors for secondary metabolism. The free amino acids also act as precursor or substrate for several processes. For example acrylamide, which is a carcenogenic compound formed during heating has been discussed in literature to be related to the presence of asparagine (Curtis *et al*., 2010; Postles *et al*., 2016). The influence of free amino acids on perceived flavour could be related to their percentages or activity as flavour precursors. For example, when maillard reaction takes place at high temperatures,compounds such as pyrroles,furfuralsand pyrazines are flavour active, and they are produced from amino acids (Heiniö, 2003).

Although amino acids are present in small percentage in rice, its reaction with soluble sugars is a major determinant of organoleptic characteristics of rice (Kamara *et al.*, 2010; Verma *et al*, 2017). The importance of amino acid in food palatability is reflected in its perception by the gustative nerve (Jinap and Hajeb, 2010). Though, they contribute to the production of some volatile compounds through different pathways by acting as flavour precursors. Amino acids in food have been reported to have tastes such as sweet, sour, bitter or umami. Therefore, in order to improve the taste of food, numerous foodprocessing operations that will increase the amino acid present in food has been adopted (Ishiwatari *et al*., 2013).

There are important flavour precursors which includeamino acids. Amino acids have been reported to be responsible for flavour formation (Heiniö, 2003). Small peptides, free amino acids, sugars, phenolic compounds and volatile compounds have been identified as markers that significantly influence the perceived flavour in rice.

Amino acid, which includes leucine, Isoleucine and valine were reported by Mukai *et al.,*(2007) to be bitter extremely, and their perceived odour is unpleasant. Among the bitter amino acids are tryptophan, phenylalanine and tyrosine. When compared to its crystal forms, proline, serine and cysteine in solution looses their bitterness (Asao *et al*., 1987). However, Roudot-Algaron (1996) and Asao *et al.,*(1987) observed the sweetness of some bitter L-amino acids D- enantiomers.

Microorganisms break down amino acid to produce different volatiles such as amines, pyrazines and pyridines (Licthenthaler *et al*., 1997). One of the pathways through which this occurs is the Erhlich's pathwaywhich involves both transamination to an alpha keto acid and a decarboxylation step to produce aldehydes, alcohols and acids.Maloney *et al*., (2010) noted that aldehydes are also products of amino acids through the anabolic pathway during its synthesis. Many of the aldehydes have fruity or malty flavour (for example isobutanal, 2 methyl butanal, 3-methy butanal) or flowery taste (for example benzene acetaldehyde). Although most of the aldehydes produced are further transformed to their alcohol and acids. The acids obtained are easily esterified and permit the synthesis of series of esters with the alcohols produced

by the same metabolism or from the one issued from other pathways. Percentages of amino acid of processed rice are as presented in Table 4.4.

4.4.1 Asparagine content of Ofada rice

Significant differences existed in the percentage of asparagine among most treatments at $p<0.05$. The maximum and minimum values of asparagine obtained were 4.795 and 2.198% respectively. The mean value of asparagine obtained of processed rice was 3.141%. Ofada paddy steeped fora day with $30^{o}C$ initial steeping temperature, parboiled at $120^{o}C$ and dried at $30^{o}C$ had the maximum value of asparagine while the minimum value was from a day steeping with initial steeping temperature of $30^{o}C$, parboiled at $80^{o}C$ and dried at $50^{o}C$. The treatment with highest parboiling temperature had the highest value of asparagine, thus, variation in the percentage of asparagine can be associated with processing temperature rather than steeping duration.

The highest and the least value of asparagine for 1day steeping treatments were 4.795 and 2.198% respectively. Significant differences were observed at $p<0.05$ for all the treatments that involve one day steeping. For three days steeping treatments, the highest and the least value of asparagine were 3.42 and 2.407% respectively. Significant differences also exist among three days steeping treatment at $p<0.05$. The highest and least percentage of asparagine for three days treatments were 3.760 and 2.531%, respectively.

Five days steeping treatments had significant differences(at $p<0.05$) among samples initially steeped at the same temperature. As such, processing temperature could directly be linked to the significant difference obtained for 1, 3 and 5 d steeping treatments in their percentage of Asparagine. On the contrary, significant difference does not exist (at $p>0.05$) with respect to steeping duration. Percentage of asparagine in this work did not show a significant variation with steeping duration.

Table 4.4. Amino acid profile of Ofada rice in percentage

Run	Steeping duration (Days)	Initial Steeping Temperature(°C)	Parboiling Temperature (°C)	Drying Temperature (°C)	Asparagine %	Cysteine %	Aspartic Acid %	Glutamic acid %	Serine %	Glycine %	Histidine %
2	1	30	80	50	2.198^{a}	0.017^{hijkl}	1.305^{a}	8.542^{a}	0.927^{b}	0.610^{ab}	0.001^{a}
6	1	30	100	70	2.799^{cdef}	0.009^{cdefg}	4.162^{i}	21.878^{ij}	0.821^{a}	0.500^{a}	0.005^{h}
15	1	30	120	30	2.972^{defgh}	0.000^{a}	2.750^{fg}	16.553^{fg}	2.092^{m}	1.982^{j}	0.012^{n}
24	1	30	120	30	4.795^{l}	0.018^{jkl}	2.938^{g}	15.468^{cdef}	1.758^{k}	1.902^{ij}	0.003^{c}
8	1	65	100	50	3.259^{hi}	0.015^{ghijk}	3.736^{h}	22.896^{j}	1.273^{fg}	0.917^{bc}	0.004^{d}
11	1	65	120	70	3.719^{j}	0.013^{efghij}	2.494^{def}	17.197^{fg}	1.167^{cde}	1.525^{efghi}	0.004^{de}
18	1	100	80	70	2.843^{cdefg}	0.011^{defgh}	2.763^{fg}	16.282^{ef}	1.972^{i}	1.78^{0ghij}	0.008^{k}
20	1	100	80	30	3.160^{fghi}	0.014^{fghij}	2.674^{efg}	17.355^{fg}	2.087^{m}	1.808^{hij}	0.004^{f}
25	1	100	80	30	3.865^{k}	0.014^{fghij}	2.411^{cdef}	13.489^{bcde}	1.404^{i}	1.583^{efghi}	0.015^{p}
13	1	100	120	50	3.109^{efgh}	0.017^{ijkl}	3.433^{h}	26.171^{h}	1.363^{hg}	0.908^{bc}	0.007^{j}
16	3	30	100	50	3.270^{hi}	0.005^{abc}	2.034^{bc}	16.931^{fg}	1.25^{efg}	1.703^{jklmn}	0.013^{o}
12	3	30	120	70	2.407^{ab}	0.016^{hijk}	2.42oC^{def}	19.201^{gh}	1.526^{j}	1.374^{def}	0.026^{r}
7	3	65	100	70	3.420^{ij}	0.012^{efghi}	2.116^{bcd}	15.535^{cdef}	1.256^{efg}	1.677^{jklmn}	0.005^{g}
14	3	65	100	40	2.876^{cdefg}	0.025^{lm}	1.896^{b}	15.165^{bcdef}	1.258^{efg}	1.505^{efgh}	0.022^{r}
5	3	100	100	50	3.365^{i}	0.090^{m}	2.059^{bcd}	12.502^{b}	1.104^{c}	1.202^{cde}	0.018^{q}
9	5	30	80	30	3.158^{fghi}	0.003^{ab}	2.268^{bcde}	15.649^{cdef}	1.230^{defg}	1.468^{defgh}	0.006^{i}
10	5	30	80	30	3.217^{ghi}	0.020^{kl}	2.207^{bcd}	12.980^{bc}	1.184^{cdef}	1.358^{def}	0.002^{b}
17	5	30	80	70	3.330^{hi}	0.01oC^{defg}	2.207^{bcd}	16.494^{fg}	1.300^{gh}	1.558^{efghi}	0.011^{m}
1	5	30	120	50	2.531^{abc}	0.012^{defghi}	1.868^{b}	15.106^{bcdef}	1.17oC^{de}	1.446^{defg}	0.007^{ij}
21	5	65	100	50	2.654^{bcd}	0.006^{bcd}	2.373^{cdef}	15.939^{def}	1.387^{hi}	1.474^{defgh}	0.007^{j}
23	5	100	80	50	3.273^{hi}	0.008^{bcde}	2.042^{bcd}	15.744^{cdef}	1.154^{cd}	1.414^{defg}	0.046^{t}
3	5	100	120	70	2.610^{bcd}	0.022^{lm}	2.384^{cdef}	15.295^{bcdef}	1.749^{k}	1.496^{defgh}	0.002^{b}
4	5	100	120	30	2.748^{bcde}	0.018^{jkl}	2.269^{bcde}	13.105^{bcd}	1.388^{hi}	1.461^{defgh}	0.003^{d}
19	5	100	120	30	3.182^{ghi}	0.013^{efghij}	2.044^{bcd}	15.443^{cdef}	1.118^{c}	1.272^{cde}	0.010^{l}
22	5	100	120	70	3.760^{jk}	0.009^{cdef}	2.409^{cdef}	19.996^{hi}	1.106^{c}	1.108^{cd}	0.004^{e}

Means in the same column having same letter are significantly not different ($p > 0.05$)

This agrees with the fact that asparagine does not determine the protein value in rice (Sung-Wook *et al.*, 2015). However, researchers (Saikusa *et al.*, 1994; Kamara *et al.*, 2010; Sasaki *et al.*,2013; Liyanaarachchi *et al.*, 2018) reported asparagine to be among the amino acids that significantly contribute to the taste of rice.

The result of the estimated coefffiecent of the model on the effect of pre-treatment condition on the amino acid profile is as presented in Appendix 9. Both parboiling and initial steeping temperature were positively correlated to the asparagine content of the milled Ofada rice. Therefore, increase in initial soaking temperature and parboiling temperatures increases the the asparagines content of milled rice.

4.4.2 Cysteine content of Ofada rice

Cysteine been a sulphur amino acid can be synthesize from methionine and as such not among essential amino acids. Cysteine has been indicated to be the source of flavour compounds such as marcapto-ethanal, 2–mercapto ethanol, and mercaptoacetic acid. These flavour compounds are formed through the Ehrlich-neubauer pathway.Zhou *et al.,* (2002) noted that protein oxidation and reaction between carbonyl group and sulfhydryl groups on cysteine causes a decrease in the level of volatile sulphur compounds**.** This has been reported to limit the contribution of cysteine to rice aroma.

As presented in Table 4.4, the highest and the lowest percentage of cysteine in Ofada rice were 0.09 and 0.00% respectively and having a mean value of 0.016%. Some treatments had significant differences among samples at $p<0.05$. The processing condition of three days steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C had the highest percentage of cysteine.The lowest was from one day steeping, 30^{o}C, initial steeping temperature, 120^{o}C parboiling temperature and 30^{o}C drying temperature. The very lowcysteine content of rice was associated to acid hydrolysis (Barbeau and Hilu, 1993; Shahin *et al.,*2009) . The work of Anuonye *et al*. (2016) also reported a relatively low value for cysteine.

For a day steeping treatments, the percentage of cysteine ranged from 0.00 to 0.018% whereas the range for 3 d steeping treatments was 0.005 to 0.090%. And for five days

steeping treatments, the range of percentage of cysteine was 0.003 to 0.022%. There is significant difference among treatments of three days steeping at $p<0.05$. This showed the effect of processing temperature when the percentage of cysteine available is originally high and since hydrothermal processes have been linked to a significant reduction in the amount of cysteine (Zhou *et al*., 2002).

Cysteine is among amino acid use in determining the protein nutritional value of food (Sung-Wook *et al.*, 2015).. The highest percentage of cysteine which is obtained after steeping for three days showed that protein nutritional value of Ofada rice is highest on the third day but may later depreciated due to the fact that they are used as volatile precursors. The result of the estimated coefffiecent of the model on the effect of pre-treatment condition on the amino acid profile is as presented in Appendix 9. Parboiling and initial steeping temperature were positively correlated to the asparagine content of the milled Ofada rice. Therefore, increase in initial soaking temperature and parboiling temperatures increases the cysteine content of milled rice.

4.4.3 Aspartic acid content of Ofada rice

Aspartic acid is one of the amino acids that has been reported to have effect on the sweetness and taste of cooked rice (Tran *et al*., 2004). It could have a flat , sour and slightly bitter taste (Roudot-Algaron, 1996). The maximum and minimum percentages of aspartic acid obtained were 4.162 and 1.305 % respectively while the mean value was 2.450%. A day steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 70^{o}C drying temperature produced the highest percentage of aspartic acid while a day steeping at 30^{o}C initial steeping temperature, parboiling at 80^{o}C and drying at 50^{o}C drying temperature produced the lowest percentage of aspartic acid.

Effect of processing temperature was evident among one day steeping treatments, as significant differences exist among treatments of one day steeping at $p<0.05$ whereas there was no significant difference among of 3 and 5 d steeping treatments. This might be due to the stability of the aspartic acid to temperature as fermentation duration increases.

The highest and the least value of aspartic acid for one day steeping treatments were 4.162 and 1.305% respectively. The percentage of aspartic acid content for three days steeping treatments ranges from 1.896 to 2.42% while that of five days steeping

treatments range from 1.868 to 2.409%. There is significant difference between the highest and the lowest value of aspartic acid in all steeping treatments. However, as the fermentation duration increases, the highest value obtained for each steeping treatments decreases. This may be due to the observation of Shurtleff and Aoyagi (2014) who noted that some amino acids are used for metabolic activities and growth of some microorganism during fermentation.

The result of the model (Appendix 9 and 10) fitted for aspartic content of milled Ofada rice showed that only steeping duration is negatively correlated to the aspartic content. Whereas, increase in aspartic content in milled rice could be linked to increase in initial steeping, drying and parboiling temperatures. R^2 value of 0.841 is a reflection of the strong correlation of the pre-treatment conditions to the value of aspartic acid.

4.4.4 Glutamic acid contents of Ofada rice

Through the Ehrlich pathway, 4-oxo butyric acid, 4 hydroxy butyric acid or butyrolactobe and butyric di-acid are produced from glutamic acid (Spinnler, 2011). Roudot-Algaron (1996) associated glutamic acid to meaty, salt, bitter taste depending on taste threshold. It may be sour when the taste threshold is high and umami when with a low taste threshold (Kato *et al*., 1989). Glutamic acid has generally been associated with its ability to enhance food flavour. A combination of aspartic acid and glutamic acid with sodium salt has been associated with umami taste in food (Kasai *et al*., 2001; Lee *et al*., 2008).

Percentage of glutamic acid in processed range from 8.542 to 26.171% with an average of 16.440 %. There were significant differences among some treatments at $p<0.05$. The maximum percentage was obtained from paddy treatment of a day steeping, 100^{o}C, 80^{o}C and 50^{o}C initial steeping, parboilingband drying temperatures repectively. The minimum value of glutamic acid was obtained with paddy treatment of a day steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature.

It could be inferred that fermentation does not have effect on the amount of glutamic present. However, variation in processing temperature could have effect on the

amount of glutamic acid present in a food matrix. Significant change in the amount of glutamic acid due to hydrothermal process has been reported (Sasaki *et al*., 2007) and it has been linked to the solubility of the amino acid in water (Xu *et al.*, 2016). Changes due to hydrothermal process could also be due to the fact that amino acids were used up during volatile formation as reported by Liu *et al*., (2007).

The percentage of glutamic acid for 1 day steeping treatments ranged from 8.542 to 26.171%. Also, glutamic acid content ranged from 12.502 to 19.201% for 3 d steeping treatments. While for five days treatments the range obtained was 12.980 to 19.996 %. Significant difference (at $P>0.05$) does not exist among five days steeping treatments, whereas there was significant difference among 1 and 3 d steeping treatments. For 1 and 3 d steeping treatments, the differences among treatments could be relatedto variation in processing temperatures.

The result of the model (Appendix 9 and 10) fitted for glutamic acid content of milled Ofada rice showed that only steeping duration is negatively correlated to the glutamic acid content. Therefore, increase in glutamic acid content in milled rice could be linked to increase in initial steeping, drying and parboiling temperatures.

4.4.5 Serine content of Ofada rice

Ofada rice had serine content ranging from 0.821 to 2.097%, with mean of 1.362. For serine content, treatments have significant differences at $p<0.05$ in their percentages. The maximum percentage of serine was obtained when paddy rice was steeped for a day at 30 ^{o}C initial steeping temperature, parboiling at 120^{o}C and dried at 30^{o}C. The minimum percentage of serine was gotten from the treatment: a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature. Higher drying temperature led to a lower percentage of serine in processed rice.

For 3 d steeping treatments, the maximum and minimum values were 1.526 and 1.104% while the range for five days steeping treatments was 1.106 and 1.749%. The average value for 1, 3 and 5d steeping treatments were 1.486, 1.279 and 1.279 % respectively. At 5 % level of significant there is significant difference among 1 day steeping treatments and also 3 days steeping treatments. While among 5 d steeping

treatments significant difference does not exist at $p < 0.05$. The significant differences could be linked to the effect of processing temperatures and not the steeping duration. Although serine is not among the aromatic amino acids,however, it was detected in this work at a percentage greater than one. Serine, though bitter always lose its bitterness when in solution (Asao *et al.*, 1987).

The result of the model (Appendix 9 and 10) fitted for serine content of milled Ofada rice showed that steeping duration and drying temperature are negatively correlated to the serine content. However, increase in serine content in milled rice could be linked to increase in initial steeping and parboiling temperatures.

4.4.6 Glycine content of Ofada rice

Result of glycine is as presented in Table 4.5. The highest and the least value of glycine were 1.982 and 0.600% with a mean value of 1.401%. There was no significant difference at $p<0.05$ among most of the treatments. This shows that glycine content of rice is not significantly affected with temperature changes and fermentation duration. Treatments with a day steeping at 30^{o}C initial steeping temperatures, parboiling at 120^{o}C and drying at 30^{o}C gave the maximum result for glycine. The least value was obtained from a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature.

The maximum and minimum values obtained for 3 d steeping temperature were 1.703 and 1.202% respectively. While for 5 days steeping treatments were 1.538 and 1.108% respectively. As the steeping duration increased, percentages of glycine in Ofada rice decreased although not significantly. Glycine has been attributed to a pleasant sweet taste in which the higher the percentage present the sweeter the food (Xu *et al*., 2016). Comparing the sweet taste of glycine with other amino acids that have a pleasant sweet taste, it was reported by Solms (1969) that glycine has lowest sweet taste. Aside the natural taste of glycine, it can also be used in the formation of volatile compounds (Lilly *et al*., 2006) though they will not enter Ehrlich pathway directly for the process.

The result of the model (Appendix 9 and 10) fitted for glycine content of milled Ofada rice showed that drying and parboiling temperature are negatively correlated to

the glycine content. While, increase in glycine content in milled rice could be linked to increase in steeping duration and initial steeping temperature. R^2 value of 0.812 and p value of 0.039 are indications of the strong correlation of the pre-treatment conditions to the value of glycine and fitness of the quadratic model.

4.4.7 Histidine content of Ofada rice

Histidine imparts bitter taste in food (Kato *et al*., 1998). In katsuobushi, a dried, fermented, and smoked skipjack tuna (*Katsuwonuspelamis*), histidine is known to impart sourness and umami taste. Though the percentage of histidine is very low in rice samples, significant differences exist among treatments at $p<0.05$ in the percentage of histidine present in processed rice. The percentage of histidine in Ofada rice ranged from 0.001 to 0.046%. The highest percentage was obtained by 5 d steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature, 50^{o}C drying temperature. While the least percentage of histidine was obtained at a day steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature. As the steeping duration increases, the percentage of histidine in Ofada rice increases.

For a day steeping treatments, the maximum and minimum valueswere 0.015 and 0.001% respectively. While 3 d steeping treatments have maximum and minimum value of 0.026 and 0.013% respectively. The 5 d steeping treatments ranged from 0.002 to 0.046%. Aside the significant effect steeping duration has on the percentage of histidine among treatments, there also exist significant difference due to the effect of variation in processing temperature. In agreement with the increase in percentage of histidine due to steeping duration is the significant increase in the amount of histidine reported in the work of Offiah *et al.,* (2017) on the co-fermentation of maize and soybean.

The result of the model (Appendix 9 and 10) fitted for histidine content of milled Ofada rice showed that only parboiling temperature is negatively correlated to the histidine content. Increase in histidine content in milled rice could be linked to increase in steeping duration, initial steeping temperature and drying temperature. R^2 value of 0.869 and p-value of 0.009 are indications of the strong correlation of the pre-treatment conditions to the value of histidine and fitness of the quadratic model.

4.4.8 Arginine content Of Ofada rice

The highest and lowest percentages of arginine found in processed Ofada rice were 0.029 and 0.001% respectively. The highest value was from paddy steeped for 3 d with 30^{o}C initial steeping temperature, parboiled and dried at 100^{o}C and 50^{o}C respectively. Arginine content was lowest with 3 d steeping treatment at initial temperature of 65^{o}C, parboiled at 100^{o}C and dried at 30^{o}C. The treatment with 5 d steeping, 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature. Higher percentage of arginine due to steeping for 3 d and later decreased at 5 d steeping treatments showed the possibility that some amount of arginine was used by fermenting microorganism for growth and metabolic activities (Shurtleff and Aoyagi, 2014)

Significant difference does not exist at $p<0.05$ among treatments steeped for a day, except for treatments processed with initial steeping temperature of 30^{o}C, and with either parboiling at 80^{o}C and drying at 50^{o}C or parboiling at 100^{o}C and drying at 70^{o}C. Significant differences existed among all 3 d steeping treatment while significant different also existed among most of the 5 d steeping treatments. This significant difference could be attributed to the hydrothermal process which could have led to the leaching of arginine (Xu *et al.*, 2016). Generally from the result of the model fitted for arginine contents of Ofada rice, increase in initial steeping temperature and parboiling led to increase in the arginine contents. Whereas, soaking duration and drying temperature are negatively correlated to the arginine content of milled rice samples (See Appendix 9 and 10)

Organoleptically, arginine is reported to be bitter in taste and also attributed to it is a characteristic odour. However, it has been used to reduce the bitterness of extremely bitter amino acids like valine (Tokuyama *et al.*, 2006). Arginine also has been reported to increasse salty taste in food (Zhao *et al*., 2016). Kani *et al*., (2008) reported that bitterness of arginine could be masked by the presence of NaCl and glutamic acid. It was also observed that there is a traceable sweetness in the bitter nature of arginine.

Table 4.4: Cont'd: Amino acid profile of Ofada rice in percentage

Run	Steeping duration (Days)	Initial Steeping Temperature(°C)	Parboiling Temperature (°C)	Drying Temperature (°C)	Arginine %	Threonine %	Alanine %	Proline %	Tryptophan %	Methionine %	Phenylalanine %
2	1	30	80	50	0.023^{a}	1.605^{a}	7.398^{a}	3.053^{a}	0.005^{cde}	0.656^{a}	0.005^{bcd}
6	1	30	100	70	0.024^{f}	1.262^{a}	8.112^{a}	2.455^{a}	0.009^{gh}	0.760^{b}	0.006^{bcd}
15	1	30	120	30	0.014^{ef}	3.555^{ghi}	21.365^{ijk}	15.148^{i}	0.006^{cde}	1.956^{m}	0.003^{ab}
24	1	30	120	30	0.013^{e}	3.673^{hi}	19.402^{ghi}	9.554^{cde}	0.004^{bc}	1.296^{f}	0.009^{de}
8	1	65	100	50	0.003^{abc}	2.777^{bcdef}	12.500^{b}	11.361^{efg}	0.004^{b}	0.782^{b}	0.006^{bcd}
11	1	65	120	70	0.002^{ab}	2.992^{cdefg}	17.284^{defg}	7.202^{b}	0.000^{a}	1.164^{e}	0.009^{de}
18	1	100	80	70	0.005^{bcd}	3.531^{ghi}	22.872^{jk}	16.027^{i}	0.005^{cd}	2.454^{p}	0.016^{f}
20	1	100	80	30	0.003^{abc}	3.992^{i}	23.648^{k}	18.809^{i}	0.006^{cde}	2.644^{r}	0.004^{ab}
25	1	100	80	30	0.028^{h}	2.640^{bcdef}	16.685^{cdefg}	8.618^{bcd}	0.008^{fg}	1.713^{k}	0.004^{ab}
13	1	100	120	50	0.017^{f}	2.662^{bcdef}	13.219^{b}	10.341^{def}	0.000^{a}	0.939^{c}	0.004^{ab}
16	3	30	100	50	0.029^{h}	3.120^{bcdef}	20.373^{hij}	10.115^{def}	0.010^{gh}	1.483^{h}	0.006^{bcd}
12	3	30	120	70	0.003^{abc}	2.924^{bcdef}	17.933^{fghi}	11.553^{efg}	0.000^{a}	2.795^{s}	0.008^{cde}
7	3	65	100	70	0.003^{abc}	2.993^{cdefg}	22.465^{jk}	12.059^{fg}	0.005^{cd}	1.548^{i}	0.005^{abc}
14	3	65	100	40	0.001^{a}	2.785^{bcdef}	19.517^{ghi}	10.236^{def}	0.003^{ab}	1.750^{i}	0.001^{a}
5	3	100	100	50	0.017^{f}	2.360^{b}	14.714^{bcd}	7.458^{bc}	0.005^{cde}	1.735^{kl}	0.004^{bc}
9	5	30	80	30	0.006^{cd}	2.780^{bcdef}	16.298^{cdef}	6.924^{b}	0.010^{gh}	1.439^{g}	0.005^{abc}
10	5	30	80	30	0.001^{a}	2.481^{bcd}	16.184^{cdef}	7.243^{b}	0.006^{cde}	1.615^{j}	0.003^{ab}
17	5	30	80	70	0.007^{d}	2.943^{bcdef}	21.722^{ijk}	9.996^{def}	0.010^{h}	1.451^{gh}	0.008^{cde}
1	5	30	120	50	0.008^{d}	2.529^{bcde}	$18.196f^{gh}$	8.267^{bcd}	0.007^{e}	1.014^{d}	0.010^{e}
21	5	65	100	50	0.001^{a}	3.115^{efg}	17.423^{defg}	12.840^{h}	0.002^{a}	3.199^{t}	0.003^{ab}
23	5	100	80	50	0.008^{d}	2.927^{bcdef}	15.206^{bcde}	7.297^{b}	0.013^{i}	1.523^{i}	0.005^{bcd}
3	5	100	120	70	0.003^{abc}	3.181^{fgh}	18.465^{fgh}	13.088^{h}	0.006^{de}	2.537^{q}	0.011^{e}
4	5	100	120	30	0.024^{g}	3.054^{defg}	17.012^{defg}	12.12^{fg}	0.006^{cde}	2.223^{o}	0.006^{bcd}
19	5	100	120	30	0.005^{bcd}	2.451^{bc}	14.101^{bc}	6.904^{b}	0.010^{gh}	1.629^{j}	0.024^{g}
22	5	100	120	70	0.015^{ef}	2.934^{bcdef}	12.792^{b}	6.967^{b}	0.005^{cd}	2.123^{n}	0.003^{ab}

Means in the same column having same letter are significantly not different ($p > 0.05$).

4.4.9 Threonine content of Ofada rice

Sensory evaluation of threonine has been reported to have a flat to sweet taste and it may be bitter while it can have a sour of fatty taste in solution (Roudot-Algaron, 1996). The maximum and least percentage of threonine were 3.673 and 1.262 respectively. The maximum value was obtained from rice processed with a day steeping at 30^{o}C initial steeping temperature, parboiled at 120^{o}C and dried at 30^{o}C. Whereas the lowest result of threonine was from paddy rice processed with a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature. Significant difference does not exist at $p<0.05$ among most treatments for the threonine content.

For a day steeping treatments, the highest and the lowest percentage of threonine were 3.673 and 1.605% respectively. Three days steeping treatments have the highest value of threonine as 3.120% while the lowest was 2.360%. Five days steeping treatments had 3.115 and 2.451% as the highest and lowest results of threonine for processed rice respectively. Variation in processing conditions has no significant effect on the percentage of threonine at $p<0.05$ since threonine is not among the amino acids that determine the protein nutritive value. Also the decline in the highest percentage of threonine from a day steeping treatments to 3 and 5 d steeping treatment may be due to the fact that threonine acts as precursor to some flavour compounds that are formed during fermentation and thermal processes.

From the result of the model fitted for threonine contents of Ofada rice (Appendix 9 and 10), increase in steeping duration, initial steeping temperature and parboiling tmperature led to increase in the threonine contents. Whereas, drying temperature is negatively correlated to the threonine content of milled rice samples.

4.4.10 Alanine content of Ofada rice

Alanine serves as a precursor for the formation of acetyaldehyde, alcohol and acetic acid (Spinner, 2011). Organoleptically, a pleasant sweet taste has been associated with alanine (Xu *et al*., 2015). In this research work, the percentage of alanine in milled rice samples ranged from 7.398 to 23.648% having a mean of 16.995%. Steeping for a day at 30^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 50^{o}C had the

lowest percentage of alanine while a day steeping, 100^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C gave the least percentage of alanine.

Processing conditions had significant effect on alanine content at 5% level of signficance. 23.648 and 7.398% were the highest and lowest value of alanine respectively for 1 d steeping treatments. Three days steeping treatments had its highest and lowest percentage of alanine in Ofada rice as 20.373 and 14.714% respectively. 5 d steeping treatments had the highest value of 21.722 while the least value was 12.792%. An observable but not significant decrease in the amount of alanine in processed rice could be due to increase in fermentation duration. This alighned with the report of Offiah *et al*., (2017) on fermented maize.

Mille rice samples with same steeping duration were significant different at $p<0.05$. This showed that major variation in the percentages of alanine in processed rice is due to the hydrothermal processes rather than the steeping durations. This is reflected in the fact significant difference (at $P>0.05$) does not exist among the highest value of the 1, 3 and 5 d steeping treatments.

With respect to the quadratic model fitted for the amino acid profile in Ofada rice (Appendix 9 and 10), steeping duration and initial steeping temperature were positively correlated to the alanine content of Ofada rice. Whereas, higher parboiling and drying temperature causes decrease in the percentage of alanine content of Ofada rice.

4.4.11 Proline content of Ofada rice

Bitter taste of peptides has proline as its contributory factor (Pratama, 2018). Proline content in Ofada rice varied from 2.455 to 18.809% with an average of 9.826. Proline content was highest when the processing conditions was a day steeping at 100^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 30^{o}C . Whereas steeping for a day at 30^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 50^{o}C had the lowest proline content

There was significant difference at $p<0.05$ in the proline content of different treatments. Samples with 1, 3 and 5 d steeping had their highest proline content as 18.809, 12.059, and 13.058%, respectively. While the lowest values were 2.455,

7.458 and 6.924% respectively. Percentage proline content in Ofada rice significantly decreased as steeping duration decreases in this work. This might be due to two reasons which are utlilisation of proline by fermenting microbes for metabolic activities and the fact that proline acts as precursor for formation of volatile compounds. Proline is precursor for the formation of 2-acetyl pyrolline (Adams and de-Kimpe, 2007) which is a compound that differentiate frangrant rice from non-fragrant rice(Wongpornchai *et al*. 2003; Itani *et al*., 2004; Champagne, 2008). Proline reacts with glucose by the maillard reaction to form 2-AP.

The result of the model (Appendix 9 and 10) fitted for proline content of milled Ofada rice showed that only drying temperature is negatively correlated to the proline content. Increase in proline content in milled rice could be linked to increase in steeping duration, initial steeping temperature and parboiling temperature.

4.4.12 Tryptophan content of Ofada rice

Percentage of tryptophan in processed Ofada rice varied from 0.00 to 0.013 with the mean value of 0.007%. The three treatments that gave the least value were a day steeping, 100^{o}C initial steeping temperature and 120^{o}C parboiling temperature, 50^{o}C drying temperature and three days steeping, 30^{o}C initial steeping temperature, 120^{o}C parboiling temperature, 70^{o}C drying temperature and a day steeping, 65^{o}C initial steeping temperature, 120^{o}C parboiling temperature, 70^{o}C drying temperature. The highest percentage of tryptophan was obtained when paddy rice was steeped for 5 d at 100^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 50^{o}C.

Significant difference existed at $p < 0.05$ on the amount of tryptophan present when paddy was processed with different treatments. The highest and the least value for 1 day steeping treatments were 0.008 and 0.000 % respectively while for 3 d steeping treatments were 0.005 and 0.00% respectively and it was 0.013 and 0.00% respectively for 5 d steeping treatments. As presented in Appendix 9 and 10, steeping duration, initial steeping temperature and drying temperature significantly led to increase in the percentage of tryptophan in milled Ofada rice smaples. Increase in parboiling temperature led to decrease in the percentage of tryptophan.

Very low percentage of tryptophan may be due to their elimination through acid hydrolysis (Shahin *et al*., 2009). Tryptophan though is a limiting amino acid in rice (Sekhar and Reddy, 1982; Sotelo *et al.*, 1994)., is among the aromatic amino acid (Kang *et al*., 2014). Tryptophan is a precursor in the synthesis of indyl acetyaldehyde, 2-indyl ethanol and indyl acetic acid. Strecker degradation of tryptophan produce 2-aminoacetophenone (Christoph *et al*., 1999) which has been reported to give off-odour characteristics of naphthalene or floor polish odor to brown rice (Rapp *et al.*, 1993).

4.4.13 Methionine content of Ofada rice

Rice has been reported to be rich in methionine which can be used to synthesis cysteine and also a determinant of the protein nutritive value (Anuonye *et al*., 2016). The methionine content of the processed rice showed a significant difference $p < 0.05$ among all treatments. The maximum and minimum percentages of methionine in the processed rice were 3.199 and 0.656 respectively. Five days steeping, 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature gave the highest value for methionine while least value was recorded when paddy was steeped for a day with initial steeping temperature of 30^{o}C, parboiling at 80^{o}C and dried at 50^{o}C. Steeping duration significantly ($p<0.05$) have effect on the percentage of methionine in processed rice.

For a day steeping treatments, the highest and lowest value obtained were 2.644 and 0.656% respectively while for 3 d steeping treatments ranged from 1.483 and 2.795%. 5 d steeping treatments has 3.199% as the maximum value of methionine while the lowest recorded was 1.014%. The content of methionine significanttly ($p<0.05$) increasesas as the steeping duration increases. Kamara *et al*., (2010) reported that germination increase the percentage of methionine in rice. In the present study, it could be inferred that fermentation also increases the percentage of methionine in rice. This variation was also observed from the result of the model fitted for amino acid profile presented in appendix 9 and 10. Increase in steeping duration and initia steeping temperature led to increase in the methionine content of milled Ofada rice. Whereas, parboiling and drying temperatures were negatively correlated to the methionine content of Ofada rice.

It was reported by Sekhar and Reddy (1982) that scented rice varieties has higher values of methionine compared to non-scented rice varieties. Invariably, higher value of methionine in the current work can be linked to the extent of aroma that would be perceived in the Ofada rice. As such, fermenting paddy rice for 5 d has a tendency to influence the organoleptic properties of processed rice. Methionine being a precursor in the synthesis of methional, methionol and 3-methythio-propionic acid serves as a major flavour contributor in rice (Spinnler, 2011)

4.4.14 Phenylalanine content of Ofada rice

Significant difference ($p>0.05$) does not exist in the phenylalanine content of milled rice among most treatments. The highest percentage of phenylalanine obtained was 0.024 and this was gotten when paddy was steeped for five days with the initial steeping temperature of 100^{o}C, parboiled at 120^{o}C and dried at 30^{o}C. The least value obtained for phenylalanine was 0.001% and 3 d steeping, 65^{o}C initial steeping temperature and 40^{o}C drying temperature gave the least value.

The highest and lowest percentages of phenylalanine in processed rice for a day steeping treatments were 0.016 and 0.003%. Significant difference (at $p>0.05$) does not exist among most of the one day steeping treatments. Three days steeping treatment had its range from 0.001 to 0008% while 5 d steeping treatments ranged from 0.003 to 0.024%. At 5% level of significance, there was no significant difference among three days steeping treatments, whereas significant difference exist among five days steeping treatments.

Although the quadratic model fitted for phenyl alanine was not significant, however there was a positive correlation of all pre-treatment conditions (Initial steeping temperature, steeping duration, drying temperature and parboiling temperature) with the percentage of phenyalanine obtained. Phenyalanine is a precursor to phenyl acetaldehyde, 2-phenyl ethanol and phenyl acetic acid. 2-phenylacetaldehyde is known as a major volatile linked to hyacinth and lilac (Knudsen *et al*., 1993). 2-Phenyl ethanol have an odour that can be likened to faded roses.

4.3.6 Optimisation of Amino acid content of Ofada rice

Result showing the optimisation and the level of desirability for amino acid of Ofada rice is presented in Appendix 11. All amino acids were set at maximum for the

optimisation. The desirability obtained consists of ten solutions which ranged from 0.43 to 0.56. The most desirable pre-treatment condition was 2.06 d soaking, 100^{o}C initial steeping temperature, 89.84^{o}C parboiling temperature and 69.98^{o}C drying temperature.

4.5 Organic acid profile of Ofada rice

In cereals, organic acids are formed through the Embden Meyerhof Parnas pathway and hexose monophosphate pathway (Shukla *et al.,* 2010) by a combination of microbial activity, biochemical metabolism and acid hydrolysis (Ren *et al*., 2016). Table 4.5 is the percentage of different organic acid detected in processed rice. The average percentages of detectable organic acids in Ofada rice by liquid chromatography mass spectrometry (LCMS) were 2-aminobutyric acid (1.735), nicotinic acid (1.98), panthotenic acid (0.416), Malic acid (0.524), pyruvic acid (0.007), lactic acid (0.463), citric acid (6.664), succinic acid (0.236), fumaric acid (0.116), and orotic acid (0.001). Organic acids detected in relatively high percentages were 2-aminobutyric acid, nicotinic acid while the highest percentage of organic acid present is citric acid.

Acids have been indicated to be majorly useful in the enhancement of flavour in food and they could also serve as a source of flavour (Hartwig and McDaniel,1995). Aside its effects on flavour, organic acids have been reported to impart olfactory and tactile sensations (Neta *et al.*,2007). Organic acids have been implicated to be the source of sour taste in foods (Neta *et al.*, 2007). However, the intensity of sourness has been reported to be linked to the properties of organic acids present (Siebert, 1999) and the tendency of binding or releasing hydrogen ions into water solution which is dependent on the percentage of the organic acid present (Ogiso *et al.*, 2000; Lyall *et al.*,2001; Lugaz *et al.*, 2005).

Saltiness, bitterness and astringency which are non-sour taste characteristics can also be found with organic acids (Rubico and McDaniel, 1992). The major non-sour taste perceived as observed by Neta (2007) is bitterness and it has an intensity that ranges from moderate to strong taste in perception. Hartwig and McDaniel (1995) reported that organic acid concentration, its pH and type of the acid are factors that determines the characteristics impart of the non-sour taste.

Table 4.5: Organic acid profile of Ofada rice

Run	Steeping time (days)	Initial Steeping Temperature °C	Parboiling Temperature °C	Drying Temperature °C	2-aminobutyric acid %	Nicotinic acid %	Panthotenic acid %	Malic acid %	Pyruvic acid %	Lactic acid %	Citric acid %	Succinic acid %	Fumaric acid %	Orotic acid %
2	1	30	80	50	1.727^{cde}	1.057^{a}	0.462^{fgh}	0.361^{cd}	0.006efg	0.420^{kl}	3.099^{a}	0.063^{a}	0.011^{b}	0.000^{b}
6	1	30	100	70	2.075^{defg}	2.020^{ghij}	0.749^{j}	0.524^{e}	0.008^{jk}	0.535^{l}	7.650^{def}	0.072^{ab}	0.021^{efgh}	0.000^{a}
15	1	30	120	30	2.813^{efgh}	1.542^{bcd}	0.296^{a}	0.211^{ab}	0.006^{ghij}	0.237^{cdefg}	4.578^{abc}	0.181^{cdef}	0.015^{c}	0.002^{h}
24	1	30	120	30	3.024^{h}	2.455^{lm}	0.331^{abc}	0.199^{a}	0.013^{n}	0.140^{ab}	3.770^{ab}	0.128^{abcd}	0.017^{cde}	0.000^{a}
8	1	65	100	50	2.221^{fg}	2.095^{hijk}	0.474^{gh}	1.957^{g}	0.004^{cde}	0.339^{hij}	9.097^{f}	0.644^{j}	1.051^{p}	0.008^{j}
11	1	65	120	70	2.360^{g}	3.098^{n}	0.356^{abcde}	0.299^{cde}	0.006^{fghij}	0.246^{cdefg}	6.966^{def}	0.257^{fgh}	0.000^{a}	0.000^{a}
18	1	100	80	70	1.148^{b}	1.938^{fghi}	0.41oC^{defgh}	0.351^{bcd}	0.003^{abc}	0.315^{ghi}	6.208^{cde}	0.176^{cdef}	0.022^{ghi}	0.000^{ab}
20	1	100	80	30	2.06^{defg}	2.573^{m}	0.500^{hi}	0.422^{cde}	0.004^{bcd}	0.180^{abc}	7.302^{def}	0.218^{defg}	0.018^{cdef}	0.001^{ef}
25	1	100	80	30	2.471^{g}	2.206^{ijkl}	0.370^{abcdef}	0.436^{cde}	0.000^{ab}	0.208^{abcdef}	6.454^{cde}	0.143^{abcd}	0.090^{i}	0.000^{a}
13	1	100	120	50	2.135^{efg}	2.109^{hijk}	0.567^{i}	2.010^{abc}	0.019^{o}	0.280^{defgh}	9.006^{f}	0.563^{j}	0.998^{o}	0.001^{g}
16	3	30	100	50	1.995^{cdefg}	1.702^{cdef}	0.343^{abcd}	0.468^{de}	0.004^{cdef}	0.300^{fghi}	6.021^{cde}	0.458^{i}	0.019^{defg}	0.000^{ab}
12	3	30	120	70	0.065^{a}	2.300^{jklm}	0.472^{gh}	0.391^{cde}	0.008ijk	0.241^{cdefg}	6.494^{cde}	0.159^{abcde}	0.048^{k}	0.003^{i}
7	3	65	100	70	1.585^{c}	1.973^{fghi}	0.451^{efgh}	0.38oC^{de}	0.007^{hij}	0.216^{bcdef}	6.261^{cde}	0.280^{gh}	0.018^{cdef}	0.000^{a}
14	3	65	100	40	1.62oC^{d}	1.442^{bc}	0.346^{abcd}	0.406^{cde}	0.006^{fghij}	0.416^{kl}	6.222^{cde}	0.273^{gh}	0.018^{cdef}	0.000^{a}
5	3	100	100	50	2.251^{fg}	1.951^{fghi}	0.422^{cdefgh}	0.437^{cde}	0.002^{ab}	0.116^{a}	6.960^{def}	0.110^{abc}	0.095^{m}	0.001^{ef}
9	5	30	80	30	1.858^{cdef}	2.347^{klm}	0.351^{abcd}	0.422^{cde}	0.011^{defg}	0.290^{efghi}	6.394^{cde}	0.251^{efgh}	0.007^{a}	0.000^{a}
10	5	30	80	30	0.971^{ab}	2.053^{ghij}	0.41oC^{defgh}	0.389^{g}	0.005^{lm}	0.197^{abcde}	6.407^{cde}	0.092^{abc}	0.023^{ghi}	0.000^{a}
17	5	30	80	70	2.161^{efg}	1.368^{b}	0.380^{abcdefg}	0.498^{de}	0.006^{ghij}	0.196^{abcde}	5.838^{bcde}	0.332^{h}	0.016^{cd}	0.002^{h}
1	5	30	120	50	1.676^{cde}	1.335^{b}	0.309^{ab}	0.421^{cde}	0.009^{kl}	0.967^{m}	5.337^{bcd}	0.313^{h}	0.000^{a}	0.000^{a}
21	5	65	100	50	0.076^{ab}	2.111^{hijk}	0.505^{hi}	0.399^{cde}	0.001^{a}	0.190^{abcd}	8.071^{ef}	0.162^{bcde}	0.026^{i}	0.001^{cd}
23	5	100	80	50	2.077^{defg}	2.274^{jkl}	0.401^{bcdefg}	0.378^{cde}	0.007^{hijk}	0.007^{jkl}	7.710^{def}	0.298^{gh}	0.019^{cdef}	0.000^{a}
3	5	100	120	70	0.054^{a}	1.606^{bcde}	0.374^{abcdef}	0.377^{cde}	0.003^{abc}	0.212^{bcdef}	7.410^{def}	0.148^{abcd}	0.021^{fgh}	0.000^{a}
4	5	100	120	30	2.019^{cdefg}	1.783^{defg}	0.431^{defgh}	0.373^{cde}	0.004^{cde}	0.185^{abcd}	7.887^{ef}	0.136^{abcd}	0.024^{hi}	0.001^{de}
19	5	100	120	30	1.787^{cdef}	1.864^{efgh}	0.334^{abcd}	0.308^{abc}	0.006^{fghi}	0.354^{hijk}	7.335^{def}	0.142^{abcde}	0.036^{j}	0.001^{fg}
22	5	100	120	70	1.825^{cdef}	2.300^{jklm}	0.344^{abcd}	0.691^{f}	0.011^{m}	0.433^{l}	8.119^{ef}	0.305^{gh}	0.283^{n}	0.000^{a}

Means in the same column having same letter are significantly not different ($p > 0.05$)

Flavour of cereal products are largely influenced by acids (amino and organic), as well as phenolic components that are non-volatile (Dimberg *et al.*, 1996; Peterson, 2001).

4.5.1 2-Amino butyric acid content of Ofada rice

The maximum and minimum values of 2-aminobutyric acid present were 3.024 and 0.054% respectively. The treatment that gave the highest percentage of 2-aminobutyric acid was a day steeping at 30^{o}C initial steeping temperature, parboiled at 120^{o}C and dried at 30^{o}C. While the least value was when paddy was steeped for five days at 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature. The mean value of 2-aminobutyric Ofada rice obtained was 1.735. Steeping duration, Initial steeping temperature and drying temperature constituted to the significant difference (at $p<0.05$) between the highest and lowest percentage of 2-aminobutyric acid.

Significant differences existed among most treatments at $p <0.05$. The highest value for one, three and five days steeping treatments were 3.024, 2.251 and 2.161%, respectively.While lowest contents were 0.063, 0.065 and 0.0766%, respectively. As the steeping duration increases, it led to decrease in the percentage of 2-amino butyric acid in Ofada rice processed in this work. Significant difference (at $p>0.05$) does not exist in the percentage of 2–aminobutyric acid among a day steeping treatments whereas the contrary holds for 3 and 5 d steeping treatments.

Effect of variation in processing temperature (initial steeping temperature, Parboiling temperature and drying temperature) is more evident in the 3 and 5 d steeping treatments. This may be attributed to increase in permeability of water because of the softening of the rice paddy, thus leading to leaching of 2-amino butyric acid in the paddy rice matrix However, from the result of the model presented in Appendix 12 and 13, steeping duration and drying temperature were negatively correlated to the percentage of 2–aminobutyric acid. While, increase in initial steeping temperature and parboiling temperatures increased the percentage of 2–aminobutyric acid..

4.5.2 Nicotinic content of Ofada rice

As presented in Table 4.5, a day steeping, 65^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 30^{o}C drying temperature gave the highest percentage (3.098) of nicotinic acid while the least value obtained (1.057%) was gotten after a day steeping with initial steeping temperature of 30^{o}C, parboiled at 80^{o}C and dried at 50^{o}C. Significant differences existed among most treatments at $p < 0.05$.

The highest values for 1, 3 and 5 d steeping treatments were 3.098, 2.300 and 2.347% respectively while the lowest values were 1.057, 1.442, and 1.335 respectively.Significant differences existed at $p < 0.05$ between the highest value for 1 and 3 d steeping treatments. Significant differences also existed between the least value for 1 d and other day's steeping treatment. From the result of the coefficient of model fitted for the nicotinic content of Ofada rice, it was observed that increase in steeping duration and drying temperature led to decrease in the percentage of nicotinic acid. While increasing the initial steeping temperature and parboiling temperature increased the nicotinic content of milled rice.

4.5.3 Panthotenic acid content of Ofada rice

The maximum and minimum values of panthotenic acid were 0.749 and 0.296%. A day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature gave the highest value while the minimum value was obtained with treatments that was processed with a day steeping, 30^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 30^{o}C drying temperature. Significant difference does not exist (at $p < 0.05$) among most treatments. This shows that the effect of processing temperature and steeping duration are not significant in this study.

The highest percentage for one, three and five days steeping treatments were 0.749, 0.472 and 0.505%. Significant differences existed at $p < 0.05$ between highest value for a day steeping treatments and other steeping treatments (3 and 5 d). A significant decrease in the percentage of panthotenic acid after 3 and 5 d steeping could be linked to the effect of fermentation and the soluble nature of organic acids. The lowest values of panthotenic acid for 1, 3 and 5 d steeping treatments were 0.296, 0.343 and 0.309%. Significant difference does not exist ($p<0.05$) among the lowest values of

panthotenic acid of different steeping treatments. Result of the model fitted (Appendix 12 and 13) for panthotenic acid showed that only steeping duration is negatively correlated to the percentage of panthotenic acid in milled rice. Whereas, the percentage of panthotenic acid in milled Ofada rice increased with increase in the initial steeping temperature, drying and parboiling temperature.

4.5.4 Malic acid content of Ofada rice

Organoleptically, Neta (2007) reported that malic acid is characterised with a smooth tartness when present in food. As such in rice, the percentage of malic acid imparts the level of tartness that is sensed in rice. There were no significant differences among most treatments at $p < 0.05$ in the percentage of malic acid detected in Ofada rice. Therefore, variation in the sensory impart of tartness imparted by malic acid may not be felt as the processing conditions varies. The maximum and least value of malic acid were 2.010 and 0.199% respectively. The average value of malic acid obtained was 0.524%. The maximum value was obtained from a day steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature while treatment of a day steeping, 30^{o}C initial steeping temperature, parboiling at 120^{o}C and drying at 30^{o}C had the lowestt percentage of malic acid.

The highest percentages of malic acid for 1, 3 and 5 d steeping treatments were 2.010, 0.468 and 0.691% respectively. At 5% level of significance, no significant differences exist among all 3 d steeping treatments. There were significant differences among highest value for 1, 3 and 5 d steeping treatments. The least values for malic acid for 1, 3 and 5 d steeping treatments were 0.199, 0.380 and 0.308% respectively. Significant differences exist between the least value for a day steeping treatments and other steeping treatments (3 and 5 d).

Presented in Appendix 12 and 13 is the modelling result of organic acid identified in Ofada rice samples. With respect to malic acid, initial steeping temperature, drying and parboiling temperature are postitively correlated to the percentage of malic acid. On the contrary, steeping duration was observed to have an indirect relationship with the percentage of malic acid in milled rice samples.

4.5.5 Pyruvic acid content of Ofada rice

The maximum and minimum percentages of pyruvic acid in Ofada rice obtained were 0.019 and 0.001 respectively. The mean value was 0.007%. Paddy steeping for a day at initial soaking temperature of 100 ^{o}C, parboiling at 120^{o}C and drying at 50^{o}C had the largest percentage of pyruvic acid. The smallest percentage was from 5 d paddy steeping, 65^{o}C initial steeping temperature, parboiling at 100^{o}C and drying at 50^{o}C. Significant difference does not exist at $p < 0.05$ among most treatments.

The highest pyruvic content for 1, 3 and 5 d steeping treatments were 0.019, 0.008 and 0.011%, respectively, while the lowest were 0.000, 0.002 and 0.001%, respectively. Very low percentage of pyruvic acid detected in rice is a contributory factor why there is no significant difference observed with variation in processing conditions. However, decrease was observed in the present work. This is contrary to the work of Muyanja *et al*., (2012) who reported an initial decrease and subsequent increase in pyruvic acid after twelve hours of fermentation of bushera (Malted or non-malted sorghum or millet. Generally, pyruvic acid content of Ofada rice samples was positively correlated to the initial steeping temperature, parboiling and dyring temperatures. However, increase in the fermentation duration decreased the percentage of pyruvic acid content (Appendix 12 and 13)

4.5.6 Lactic acid content of Ofada rice

The milled rice lactic acid content varied from 0.116 to 0.967% with an average of 0.463%. Padddy steeping for 5 d at an initial steeping temperature of 30^{o}C, parboiling at 120^{o}C and dried at 50^{o}C had the largest percentages, while the least value was from a day paddy steeping, 100^{o}C initial steeping temperature, parboiling at 80^{o}C and drying at 50^{o}C. Significant difference existed amongmost treatments at $p < 0.05$. Steeping treatments of 1, 3 and 5 d had their largest percentages as 0.535, 0.426 and 0.967, respectively. There were significant differences among the highest values of lactic acid at $p < 0.05$. The lowest percentages of lactic acid for 1, 3 and 5 d steeping treatments were 0.140, 0.116 and 0.185 respectively.

Significant increase in the percentages of lactic acid could be attributed to the effect of fermentation of rice paddy by lactic acid bacteria. Lactic acid being one of the bye products of lactic acid bacteria increases as steeping duration increases. Variation within same duration of steeping (Fermentation) could be attributed to the volatility

and solubility of lactic acid in during hydrothermal processes. Sensory attribute associated with lactic acid is an acrid taste (Neta, 2007). This can also be linked to sour or bitter taste in fermented foods. The acrid taste could be pleasant or unpleasant depending on intensity of lactic acid present in the food and pH. A combination of lactic and citric acid has been reported to give intense sourness which persists for long.

4.5.7 Citric acid content of Ofada rice

The maximum and minimum values of citric acid obtained from processed rice were 9.097 and 3.099 % respectively. The maximum value was obtained with a day steeping at 65 ^{o}C initial steeping temperature, parboiled at 100^{o}C and dried at 50^{o}C. The least value was from a day paddysteeping, 30 ^{o}C initial steeping temperature, parboiling at 80^{o}C and drying at 50^{o}C. Significant difference does not exist at $p < 0.05$ among treatments. The highest value for 1 , 3 and 5 d treatments were 9.097, 6.960 and 8.119% respectively while least values were 3.099, 6.021 and 5.337% respectively. Though present in relatively high percentage compare to other organic acids, variation in the percentage of citric acid is due majorly to hydrothermal processes that leads to leaching because of citric acid solubility. Decrease in citric acid during fermentation was also reported by Muyanja *et al*., (2012) during fermentation of bushera (Malted or non-malted sorghum or millet).

Sour taste imparted by citric acid to food has been described as fresh (although tart) by Gardner (1977). This intense sour taste has been reported to disappear after a short time. This might be the reason why citric acid is found to be higher in one day steeping treatments compared to 3 and 5 d steeping treatments as shown in Table 4.5. Decrease in citric acid might also be associated with the utilisation of citric acid by microbes and flavour formation during rice processing.

4.5.8 Succinic acid content of Ofada rice

Succinic acid is identified in food with its tart taste which is slightly bitter in aqueous solution (Neta, 2007). Aside the sour taste, bitter notes in food has also been linked to the presence of succinic acid (Rubico and McDaniel, 1992). Panelist from different races also gave different description to the taste of succinic acid which is taste of monosodium glutamate (Oriental), Savory and brothy (Caucasians)

In this study, succinic acid content of milled rice samples ranged from 0.063 to 0.644% with a mean of 0.236%. The highest value was obtained after a day steeping at 65 ^{o}C initial steeping temperature, parboiling at 100^{o}C and dried at 50^{o}C while the smallest percentage was from a day steeping at 30^{o}C initial steeping temperature, parboiling at 80^{o}C and drying at 50^{o}C drying temperature. Most treatments had significant difference among samples at 5% level of significance.

The largest percentages for 1, 3 and 5 d steeping treatments were 0.644, 0.458 and 0.332%, respectively while the least values were 0.063, 0.110 and 0.092 respectively. The largest percentages were significantly different at $p < 0.05$, whereas there was no significant difference among their lowest values. Variation of succinic acid content as reported in this work could be attributed to the steeping duration and losses through hydrothermal processes adopted. Observable decrease in succinic acid in this work is in agreement with decrease in succinic acid reported by Muyanja *et al*. (2012) during natural flora fermentation of bushera (Malted or non-malted sorghum or millet).

4.5.9 Fumaric acid content of Ofada rice

Fumaric acid content of processed rice for different treatment showed significant difference ($p < 0.05$). The highest and lowest percentages were 1.051 and 0.011, respectively. A day steeping at 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature gave the highest value while paddy processed with a day steeping at 30 ^{o}C initial steeping temperature, parboiled at 80^{o}C and dried at 50^{o}C had the least value of fumaric acid.

The largest percentages of fumaric acid obtained for 1, 3 and 5 d steeping treatments were 1.051, 0.095 and 0.283, respectively. While the lowest percentages of fumaruc acid in 1, 3 and 5 d steeping treatments were 0.011, 0.018 and 0.016% respectively. Significant variation in the fumaric content of Ofada rice is due to variation in the heat treatments of paddy. However, slight variation could be linked to the effect of fermentation. Fumaric acid is reported to have sour taste that is tart in nature (Gardner, 1977). Jonhson and Peterson (1974) gave a Metallic taste description to fumaric acid.

4.6.9 Optimisation of organic acid in Ofada rice samples

The suitability of the model presented in Appendix 12 could be inferred from the value of the adequate precision of value for nicotinic, panthotenic, malic, lactic and fumaric acid greater than 4. The model coefficient showed that, increasing the soaking time led to increased citric and orotic acid. However, increasing the initial steeping temperature has a positive effect on the 2- aminobutyric, nicotinic, panthotenic, malic, pyruvic, citric, succinic, fumaric and orotic acids. The paddy drying temperature also correlated positively with panthotenic, malic, pyruvic, lactic, citric, succinic and fumaric acids. Whereas increasing the parboiling temperature reduces the lactic acid content of Ofada rice sample.

As shown in Appendix 14, optimisation goal for all the organic acids were set at maximum. The solutions predicted to be suitable for the optimisation goals is as presented in Appendix 14. Seven optimisation solutions were obtained, however, steeping duration of a day, initial steeping temperature of 100^{o}C, Parboiling temperature of 109.68^{o}C and drying temperature of 46.69^{o}C had the highest desirability of 0.58.

4.6 Chemical properties of Ofada rice

Chemical properties evaluated in this study were proximate, free fatty acid and amylose. Results of chemical properties of processed Ofada rice are as presented in Table 4.7.

4.6.1 Moisture content of Ofada rice

Ofada rice samples had 4.22 and 10.24 as the lowest and highest percentage of moisture content. The average moisture content of all samples was 8.89 %. The highest moisture content was obtained in two samples (Steeping for a day at 100^{o}C intial steeping temperature, parboiling at 80^{o}C with drying at 30^{o}C drying, and treatment of 5 d steeping with initial steeping temperature of 100^{o}C, parboiling at 120^{o}C and drying at 30^{o}C.

Milled rice produced by steeping paddy for a day with initial steeping temperature of 30^{o}C, parboiling at 100^{o}C and dried at 70^{o}C had the minimum moisture content .

Table 4.6.Result of Chemical Properties of Ofada rice

Run	Steeping duration (Days)	Initial Steeping Temperature °C	Parboiling Temperature °C	Drying Temperature °C	Moisture %	Ash %	Fat %	Protein %	Crude fibre %	Carbohydrate %	Energy kcal	Acid Value mgKOH/g	Free fatty acid %	Amylose %
2	1	30	80	50	8.36^{de}	0.55^{efg}	0.64^{a}	8.45^{ef}	0.75^{de}	81.30^{m}	364.73^{fgh}	1.55^{a}	0.78^{abc}	17.81^{a}
6	1	30	100	70	4.22^{a}	0.63^{ghi}	0.62^{a}	8.91^{j}	1.00^{a}	84.65^{n}	379.82^{k}	5.16^{h}	2.59^{k}	19.25^{abcd}
15	1	30	120	30	9.73^{ij}	0.57^{efg}	0.77^{abc}	7.41^{a}	0.70^{ij}	80.85^{ki}	359.97^{bcd}	2.76^{c}	1.39^{e}	18.09^{a}
24	1	30	120	30	9.49^{ghi}	0.58^{efg}	1.03^{def}	8.88^{j}	1.35^{hi}	78.70^{bc}	359.58^{b}	2.73^{c}	1.37^{e}	17.48^{a}
8	1	65	100	50	8.08^{cd}	0.71^{h}	1.04^{def}	8.61^{fghi}	1.15^{cd}	80.44^{jk}	365.56^{i}	4.08^{g}	2.05^{ij}	17.41^{a}
11	1	65	120	70	6.65^{b}	0.59^{efg}	0.98^{cde}	8.59^{fghi}	1.26^{b}	81.96^{n}	371.03^{j}	3.60^{e}	1.81^{gh}	19.11^{abcd}
18	1	100	80	70	7.72^{c}	0.47^{bcd}	1.03^{def}	8.09^{c}	0.78^{c}	81.95^{m}	369.42^{j}	2.94^{cd}	1.48^{ab}	18.43^{abc}
20	1	100	80	30	10.24^{j}	0.38^{b}	1.05^{def}	9.37^{k}	0.65^{j}	78.5^{bc}	361.08^{bcde}	4.23^{g}	2.13^{j}	17.65^{a}
25	1	100	80	30	9.45^{ghi}	0.40^{b}	1.05^{def}	7.58^{b}	0.59^{ghi}	80.96^{lm}	363.62^{efg}	5.43^{h}	2.73^{k}	17.76^{a}
13	1	100	120	50	8.72^{e}	0.40^{b}	1.73^{j}	8.66^{ghi}	0.57^{e}	79.95^{hi}	370.01^{j}	1.93^{b}	0.97^{cd}	17.61^{a}
16	3	30	100	50	9.46^{ghi}	0.41^{b}	1.19^{efgh}	8.49^{fg}	1.71^{ghi}	78.77^{c}	359.77^{bc}	2.61^{c}	1.32^{e}	22.34^{e}
12	3	30	120	70	8.64^{de}	0.24^{a}	1.11^{defg}	8.24^{cd}	0.85d^{e}	80.96^{hi}	366.79^{i}	1.99^{b}	1.0oC^{d}	21.31^{de}
7	3	65	100	70	8.24^{cde}	0.60^{fg}	1.35^{hi}	8.76^{ij}	1.2oC^{de}	79.89^{ghi}	366.74^{gh}	2.66^{c}	1.34^{e}	21.19^{de}
14	3	65	100	40	9.48^{hi}	0.58^{efg}	1.35^{i}	8.56^{fgh}	0.79^{hi}	79.27^{def}	363.48^{efg}	3.98^{fg}	2.00^{hij}	22.55^{de}
5	3	100	100	50	9.85^{ij}	0.57^{efg}	1.31^{ghi}	8.73^{hij}	1.26^{ij}	78.31^{ab}	359.97^{bcd}	6.99^{i}	3.52^{l}	21.44^{de}
9	5	30	80	30	8.77^{ef}	0.57^{efg}	1.17^{defghi}	8.86^{j}	1.22^{ef}	79.44^{efg}	363.73^{efg}	7.56^{j}	3.80^{m}	20.83^{cde}
10	5	30	80	30	9.42^{ghi}	0.57^{efg}	1.26^{fghi}	8.19^{cd}	0.99^{ghi}	80.21g^{ij}	359.53^{b}	5.44^{h}	2.73^{k}	20.83^{cde}
17	5	30	80	70	8.87^{efgh}	0.51^{cde}	1.05^{def}	8.42^{ef}	0.73^{efgh}	80.46^{ejk}	364.97^{fgh}	1.47^{a}	0.74^{b}	20.95^{cde}
1	5	30	120	50	8.81^{efg}	0.59^{efg}	1.26^{fghi}	7.52^{ab}	1.04^{efg}	80.81^{kl}	364.66^{fgh}	1.39^{a}	0.70^{a}	17.83^{a}
21	5	65	100	50	9.81^{ij}	0.43^{bc}	1.09^{defg}	8.46^{ef}	0.57^{ij}	79.66^{efgh}	362.31^{cdef}	1.96^{b}	0.98^{cd}	21.04^{cde}
23	5	100	80	50	9.45^{ghi}	0.54^{de}	0.94^{bc}	9.57^{l}	0.60^{ij}	78.93^{cd}	362.48^{def}	3.71^{ef}	1.86^{ghi}	18.39^{abc}
3	5	100	120	70	9.37^{fghi}	0.64^{gh}	1.15^{defghi}	8.50^{fg}	0.67^{fghi}	79.70^{fgh}	363.13^{efg}	1.89^{b}	0.95^{bcd}	21.71^{de}
4	5	100	120	30	10.24^{j}	0.40^{b}	1.27^{fghi}	9.42^{kl}	0.63^{j}	78.07^{a}	361.39^{bcd}	3.67^{ef}	1.84^{ghi}	20.65^{bcde}
19	5	100	120	30	10.15^{j}	0.58^{efg}	1.06^{defg}	8.3^{d}	1.01^{j}	79.23^{de}	356.98^{a}	3.25^{d}	1.64^{fg}	20.53^{bcde}
22	5	100	120	70	9.81^{ij}	0.51^{cde}	0.68^{a}	8.51^{fg}	0.84^{ij}	79.68^{efgh}	358.88^{ab}	2.09^{b}	1.05^{cd}	21.19^{de}

Means in the same column having same superscripts are significantly not different ($p > 0.05$)

Significant difference exist among most treatments. This could be linked to the different drying temperature used in this study. The maximum percentage of moisture content for 1, 3 and 5 d steeping treatments were 10.24, 9.851 and 10.24, respectively, while the lowest were 4.22, 8.24, 8.77, respectively.

Although, Anuonye *et al*., (2016) worked on the prolonged steeping and improved method of Ofada rice processing, they reported the highest moisture content from prolonged steeping of Ofada rice paddy. This was also corroborated by Adekoyeni (2014) who reported significant effect of steeping on the percentage moisture content in milled rice.

The percentage moisture content obtained in this work is in agreement with the result obtained byAnuonye *et al.,*(2016) who obtaines a range of 8.24 to 10.21 g/100g. However, result of moisture content reported by Adeniran *et al*., (2012) is higher than that obtained in the present study.Moisture conten of 7.84 to 11.72% was obtained byFagbohun and Oluwaniyi (2015) who used sun drying method in a study of milled rice storage . Inconsistency in research result of moisture content may be as a result of factors such as drying variables, prevailing weather conditions (Ebuchi and Oyewale, 2007) and some times seed coat and pericarp nature could be waxy (Roy *et al.*, 2011).

Moisture content significantly influenced rice acceptability and palalatability (Oko *et al.,* 2012). Moisture content of rice have been reported to be a major index of storage stability and product quality (Adekoyeni, 2014). Rice moisture content of 12 to 14% was suggested as the optimum storage condition for rice. Maintanance of rice at the optmum moisture content become important due to it hygroscopic nature (Fagbohun and Oluwaniyi, 2015) alongside the need for the extending the shelf life of rice by not preventing microbial growth caused by high moisture content.

4.6.2 Ash content of Ofada rice

The percentage ash content of Ofada rice ranged from 0.24 to 0.71, with a mean of 0.515%. Three days steeping, 30^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature gave the lowest value while the highest value was obtained from a day steeping, 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and drying at 50^{o}C. Significant difference ($p<0.05$) does not

exist among treatments. Adeniran *et al*., (2012) obtained a higher percentage of ash compared to the result of the present study. However, result obtained by Adekoyeni (2014) in a study of influence of storage and processing condition on Ofada rice quality is in congruence with the result of ash in the present work .

The highest percentages of ash for 1, 3 and 5 d steeping treatments were 0.71, 0.60 and 0.64 % respectively while the lowest percentages obtained were 0.38,0.24, 0.40%. Adekoyeni (2014) reported his highest percentage of ash when Ofada rice paddy was stored for 5 months, steeped for a day, parboiled at 80^{o}C and dried at 30^{o}C. However, in the present work, the highest percentage was obtained at a different processing condition, although storage duration not considered in this work may be the source of variation. Storage influence on moisture can lead to an increase in the ash content obtained (Fagbohun and Oluwaniyi, 2015). The work of Fagbohun and Oluwaniyi (2015) also reported a decline in the percentage of ash when milled rice was sun dried and stored.

The percentage of ash reported in this work is in agreement with the study done by Anuonye *et al.* (2016), who noted that overnight paddy steeping had the maximum percentage (1.78) of ash and steeping for eight days had the minimum (1.17%) ash content. This significant ($P< 0.05$) reduction in the ash content of processed rice had been connected to possible nutrient loss caused by leaching that occurs during elongatedsteeping of paddy.

4.6.3 Fat content of Ofada rice

The range of milled rice fat content was 0.62 to 1.73%. Treatment with a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature gave the lowest fat content while the highest content of fat was obtained from 1 d steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 50^{o}C drying temperature. The fat content of milled rice from most of the paddy pre-treatment conditions were not significant different ($p <0.05$), while some treatments had significant differences. This variation in the percentage of fat in milled rice could be attributed to the complexity of processing temperature (Fennema, 1996). Paddy treatments that gave the maximum and minimum fat percentages in milled rice samples in this work agreed with the report of Fennema (1996). Minimum

value of fat was reported by Adekoyeni (2014) when the paddy drying was done at lower temperatures.

The highest value for 1 and 3 d, and 5 d steeping treatments were 1.73, 1.33 and 0.65 respectively while lowest value were 0.62, 1.11 and 0.68 respectively. Significant difference ($p<0.05$) existed in the values of fat obtained for different steeping treatments. The significant decrease as the steeping duration increase may be attributed to the action of lipase on fat to produce numerous flavour compounds (Fagbohun and Oluwaniyi, 2015). Range of fat content obtained in this work is within the range of that obtained by Zhou *et al*. (2011) in a study on the influence of storage duration on rice quality. Local rice in Nigeria has been reported to have the minimum and maximum fat content as 0.5 to 3.5% respectively (Oko and Ugwu, 2011). Fat content reported by Adeniran *et al.,* (2012) for Ofada rice processed with traditional method is within the range of that obtained in this work. Akinoso and Adeyanju (2010) reported that low fat content in milled rice was due to the fact that rice bran host greater percentage of the fat which is lost during milling operation of rice.

However, despite the low percentage of rice lipids, its impact on rice flavour is significant. Numerous flavour characteristics has been linked to the presence of lipids due to the hydroxyl fatty acids emanating from lipid oxidation and free fatty acids which has its source from hydrolytic oxidation catalysed by lipase (Heinio, 2003). Nawar (1996) highlighted furanones, alcohols, aldehydes, lactones,ketones, acids, and hydrocarbons as decomposition products of rice lipids. Champagne (2008) reported lipid oxidation products (hexanal, acetic acid and pentanoic acid) to have been among compounds that determine rice acceptability because they could have negative effect on rice flavour. The work of Reineccius (2006) on flavour perception reported that flavour release and perception is also controlled by lipid.

Although, in the study of rice aroma, major focus has been on 2-AP, however, lipid oxidation products have also been identified to be significant in rice aroma as they have negative impact on acceptability (Champagne, 2008).Lipid oxidation products are compounds that may become more prevalent in rice flavour with the length of storage time or due to poor post-harvest handling.Lipids have been found to indirectly influence rice flavour due to its oxidation which leads to the production of hydroxy

fatty acids or hydrolytic oxidation of lipids which are lipase-catalysed to produce free fatty acids.Also, the indirect effect of lipids on flavour could be linked to both oxidation and lipid catalysed hydrolytic oxidation of lipids to produce hydroxy fatty acids and free fatty acids respectively.

Lipid content of rice ranges from 1 to 4% (Kennedy and Burlingame, 2003; Mano *et al*., 1999). The free fatty acids in rice are mainly palmitic, stearic, oleic and linoleic acid (Zhou *et al*., 2002a). Volatile formation from lipids results from lipolysis, lipid oxidation and decomposition. Lipase produces free fatty acids that may undergo oxidation. Hydroperoxides formed with lipid oxidation readily decompose yielding a variety of products with varying molecular weights and odor thresholds. Decomposition products include aldehydes, lactones,ketones,furanones, alcohols, acidsand hydrocarbons many of which impact flavor (Nawar, 1996).

During storage, the activity of lipase and lipoxygenase increases, resulting in an enhanced production of the volatile compounds, particularly hexanal which contributes to off-flavor (Zhou *et al.,* 2002). The 2-alkanone is produced from saturated fatty acids in substantially larger quantities during thermal oxidation. Lipids are also important to the flavor of foods, because they increase the binding of lipophilic flavor compounds. Lipids, therefore, can moderate both flavor release and perception (Reineccius, 2006).

Another major causes of off-flavour and bitterness in food is rancidity. This is due to hydrolysis of lipids, and subsequent fatty acid oxidation. The occurrence of hydrolysis of lipid could be chemical reactions or enzymatic base. This depended upon the conditions of processing. Stability of whole grain flavours could be controlled by impeding the formation of free fatty acid through this processes. This established the fact that flavour statbility of food materials is significantly influenced by the hydrolysis of lipids. Tava and Bocchi (1999) only detected 2-AP and lipid oxidation products as markers for differences in fragrant rice samples.Aside 2-AP andlipid oxidation products, there is no other particular volatile or non-volatile compound that has been reported to be responsible for the rice flavour characteristics.

4.6.4 Protein content of Ofada rice

The milled rice sampls had 9.57 and 7.41% as the maximum and minimum percentage of protein respectively. The mean value of protein content was 8.52%. Samples from treatments had Significant differences at $p < 0.05$. The maximum protein content was from sample of paddy steeping for 5 d at initial steeping temperature of 100^{o}C, parboiled at 80^{o}C and dried at 50^{o}C e while the minimum protein was from paddy steeped for a day at 30 ^{o}C initial steeping temperature, parboiling at 120^{o}C and 30^{o}C drying temperature..

The maximum value obtained for 1, 3 and 5 d steeping treatments were 9.37, 8.76 and 9.57% respectively, while the least values were 7.41, 8.24 and 7.52% respectively. The present study agreed with a research work on African rice varieties which had a range of 7.22 to 11.29 in the reported percentage of protein (Joseph, 2015). The potential of fermentation in increasing cereals protein content can be linked to the reason that made 5 d steeping to have the highest protein content. Eigth days steeping (fermentation) of rice paddy by Anuonye *et al.,* (2016) also had the highest protein content in the milled rice sample. Adekoyeni (2014) in his work on how pretreatments conditions and storage influence the quality of Ofada rice quality also reported the highest protein content of 10.49% for nine months paddy, five days steeping, parboiling at 80^{o}C, and drying at 30^{o}C. Wireko-Manu and Amamoo (2017) also obtained protein content range of 5.64 - 8.40% in a study of local rice varieties in Ghana.

Amino acids being the building block of protein have been implicated to be among the determinants for perceived flavour in rice or they could act as flavour precursors (Kamara *et al*., 2010). Some of the amino acids directly impact taste on rice (Mukai *et al*., 2007; Roudot-Algaron, 1996), while others produced flavour in reaction with other compounds in the rice matrix. Although, protein in rice occurs in comparative low percentage, the reaction of free amino acids with soluble sugar has been reported to be of significance in the organoleptic characteristics of food (Kasumyan, 2016). Rice protein is important for biosynthesis of nitrogenous odour active compounds such as 2 AP and indole which has been reported to give rice its popcorn and mothball aroma respectively (Yoshihashi *et al.*, 2002)

4.6.5 Crude fibre content of Ofada rice

Crude fibre content of milled Ofada rice has its highest and lowest percentages as 1.71 and 0.60%, respectively. The average value was 0.92%. The highest value was obtained with the paddy rice treatment: three days steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature, five days steeping, 100^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature. Significant differences exist among most treatments in the content of their crude fibre at 5% level of significance. Rice samples from 1, 3 and 5 d steeping treatments have their maximum crude fibre content as 1.35, 1.71 and 1.22% respectively, with the lowest values as 0.57, 079 and 0.57% respectively.

Higher range of crude fibre was obtained in this work when compared to the result of Adekoyeni (2014) (0.12 – 0.37%). However, his observation of the effect of processing condition on the percentage of crude was also corroborated in this work. The reported range of Ofada rice crude fibre by Ibukun (2008) and Adepoju *et al*., (2016) were 1.02-1.76% and 0.94-1.19% respectively. Their outcome is in congruence with this study. The work of Wireko-Manu and Amamoo (2017) on Ghanaian local rice varieties proximate composition obtained between 0.64-1.95 % as the crude fibre. Wireko-Manu and Amamoo (2017) observed that foreign rice had lower percentage of crude fibre when compared to their local counterpart majorly due to the degree of rice milling. This assumption could be considered since rice bran is rich in crude fibre and there is underdevelopment in the technology of local rice milling operation in Africa.

4.6.5 Carbohydrate content of Ofada rice

According to Reineccius (2006) there is vapour pressure reduction due to the presence of polysaccharide and this may influence flavour release in food, hence the importance of carbohydrate in food flavour. The percentage of carbohydrate in milled Ofada rice is withing the range of 78.31 and 84.65%. The mean of the carbohydrate content obtained is 80.12%. The highest value was from paddy rice processed by a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and dried at 70^{o}C. The lowest value was from paddy treatment of 3 d steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature. Adekoyeni (2014) reported the significant effect drying temperature had

on carbohydrate. He reported the highest carbohydrate content when paddy was dried at 70^{o}C which was also obtained in the present study.

The samples carbohydrate content were significantly different ($p < 0.05$). The maximum result of carbohydrate obtained for 1, 3 and 5 d steeping were 84.65, 80.96 and 80.46% respectively. It was previosly observed by Anuonye *et al*., (2016) thatsteeping duration signifcantly influenced the carbohydrate content of Ofada rice. The findings of the present study corroborated the fact that carbohydrate content decreases as duration of steeping increases.Carbohydrate losses in steep water because of its solubility is attributable to this finding. Range of carbohydrate content in thisstudy agrees with the work of Adekoyeni (2014) who obtained a range of 78.58 to 84.35%. Whereas the present work had a higher carbonhydrate range when comparte to the study of Fagbohun and Oluwaniyi (2015) that obtained 63.72 to 61.28% for Ofada rice sample. This could be connected to the reported increment in protein content of rice during storage (Fagbohun and Oluwaniyi, 2015).

Scientific findings revealed that, even though found as minor constituents, free amino acids together with soluble sugars play a significant role in deciding the organoleptic properties of food (Kamara *et al.*, 2010; Kasumyan, 2016).

4.6.6 Energy value of Ofada rice

The maximum energy value (379.82 kcal/100g) was obtained for paddy rice steeped for a day at 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature. Theminimum energy value of 356.98 kcal/100g was estimated for paddy treatment of 5 d steeping at initial steeping temperature of 100^{o}C, parboling at 120^{o}C and drying at 30^{o}C. Significant difference existed in the result of energy of milled rice smaple of different treatments at $p < 0.05$. The average energy value was 363.99 kCal/100g. The steeping treatments of 3 and 5 d had their maximum energy value as 379.82, 366.79 and 364.97 kcal/100g respectively, while their lowest value were 359.58, 359.77 and 356.98 kcal/100g, respectively.

The energy value estimated in this study is comparatively lower than that obtained by by Adekoyeni (2014) (367.18 - 400.40 kcal/100g).However, the range of result obtained by Anuonye *et al*., (2016) (357.72 - 368.68 kcal/100g) and Adepoju *et*

al,.(2016) (366.75 - 371.50 kCal/100g) were in agreement with this work. Decreasing milled rice energy value with increase in steeping duration inferred in this work was also observed by Anuonye *et al*. (2016). In the work of Anuonye et al., (2016), 357.72 kcal/100g was reported for paddy rice steeped for eight days, while 368.68 kcal/100g was reported for paddy that was steeped overnight. This significant decrease might be due to loss of water soluble components into the steep water (Abulude, 2004; El-Qudah*et al.*, 2008).

4.6.7 Free fatty acid content of Ofada rice

Rice has four major free fatty acids which are linoleic, palmitic, stearic and oleic acid (Zhou *et al*., 2002). The maximum free fatty acid (3.80%) in milled rice sample was obtained steeping was done for five days at an initial steeping temperature of 30^{o}C, parboiled at 80^{o}C and dried at 30^{o}C. The lowest free fatty acid of 0.70% was from steeping paddy for 5 d at initial steeping temperature of 30^{o}C, parboiling at 120^{o}C and drying at 50^{o}C. The significant difference ($p < 0.05$) existed in the free fatty acid content of milled rice fromm different pretreatments. The maximum free fatty acid content for 1, 3 and 5 d steeping treatments were 2.59, 3.52 and 3.80% respectively, and with the lowest free fatty acid content as 0.78, 1.00 and 0.70%, respectively.

Higher steeping duration led to Increase in the percentage of free fatty acid present in milled rice sample. This was also observed in the work of Adekoyeni (2014) who had the free fatty acid maximum percentage of 4.78% with a month paddy storage, 5 d steeping, 80^{o}C parboiling temperature and drying at 30^{o}C whilethe minimum percentage of 1.23 was obtained with paddy storage for a month, steeping for 5 d , parboling at 120^{o}C and drying at 70^{o}C. Reduced free fatty acid content at maximum processing temperature in this work might be because lipase is inactivated at higher temperatures. At higher temperatures lipolytic enzyme actions are inhibited, and there is little or no opportunity for the production of free fatty acid which require lipase as a catalyst (Fennema, 1996)

One of the product of hydrolysis of lipids is free fatty acid. The influence of free fatty acid on rice organoleptic properties has been documented (Zhou *et al.*, 2002, Doblado-Maldonado *et al.*, 2012). When rice is used in the production of beer, Malin (2000) reported that free fatty acid greater that 0.1% could have negative effect on beer flavour. The presence of large percentage of free fatty acid in food has been

reported to cause off-flavours (Galliard, 1989). As reported by Galliard (1989), the off-flavour emanate from conjugated diene hydroperoxides which is produced during the oxidation of unsaturated free fatty acid.

4.6.8 Amylose content of Ofada rice

The texture and taste of cooked rice are the two main sensory properties influenced by amylose content (Zhang *et al*., 2010; Futakuchi and Sié, 2009). The amylose content of milled rice obtained in the present work varied from 17.41 to 22.34% with 19.73% as the mean. Steeping for 3 d at 30^{o}C initial steeping temperature, parboiling at 100^{o}C and drying at 50^{o}C drying temperature had the maximum amylose content.The lowest amylose content was obtained from milled rice processed by a day steeping at steeping temperature of 65^{o}C, parboiling at 100^{o}C and drying at 50^{o}C.

Significant difference does not exist in the amylose content of different samples at 5% level of significance. Steeping treatments of 1, 3 and 5 d had their highest as 19.11, 22.34 and 21.71% respectively while the least percentages were 17.41, 21.19 and 18.39, respectively.Danbaba *et al*., (2011) result of the amylose content range from 19.77 to 24.13% FARO 42 variety of ofade rice. This results agrees with present study on OS 6 variety of Ofada rice. Using the rice classification scheme base on amylose content by Cruz and Khush (2000), the obtained amylose content of Ofada rice in this work makes it to be in the group of intermediate amylose rice. The same classification was also agreed on for Ofada rice by other researchers (Danbaba *et al.,* 2011; Anuonye *et al*., 2016).

Adeniran *et al*., (2012) obtained a higher percentage of amylose content compared to the highest value in this study.However, the amylose content is within the range of those reported in the present study. Generally, paddy parboiling causes reduction in the amylose content of rice samples (Otegbayo *et al*., 2001; Adeniran *et al*., 2012). This reduction has been connected to solubilisation of starch which is eventually lost during steeping and parboiling of paddy (Otegbayo *et al.*, 2001). The level of loss is dependent on the duration and level of hydration. In a study by Adekoyeni *et al*., (2015) on the effect of storage, steeping, parboiling and drying temperature on milled rice, significant decline in amylose content due to severity of processingwas reported. On the contrary, the comparative study by Anuonye *et al*., (2016) on paddy processing using both traditional and improved methods, it was concluded that paddy

steeping for eight had no significant effect on the amylose content of rice samples.The present work agreed with the findings of Anuonye et al. (2016), as no significant difference was established in the amylose content of milled rice in relation to steeping. This study therefore observed that leaching of amylose was not significantly affected by the duration and level of paddy hydration.

Variation in the percentage of starch in rice species majorly distinguishes their characteristics. Milled rice was reported to have about 90% starch. And this has made rice varieties to be classified base on their amylose content as high (25 - 30%), intermediate (20 - 25%), low (10 - 20%), very low (2 - 9%) and waxy (1 - 2%)(Bao and Bergman, 2004). It has been reported that variations in composition and cooking quality of rice is mainly dependent on the genetic as well as surrounding environmental factors where they are grown (Giri and Vijaya, 2000; Singh *et al*., 2005).

Adeniran *et al.,*(2012) noted that Ofada rice is specially relished because of its characteristics flavour that develops during steeping as a result of the fermentative activities of some microorganisms. It has been reported that aside the changes in composition and distribution of nutrients in the rice kernel, Steeping also causes the leaching of rice constituents into the steep water (Otegbayo *et al*., 2001; Ibukun, 2008; Sareepuang *et al*., 2008). This leads to decrease in amylose content of rice due to leaching of starch granules (Sareepuang *et al*, 2008). Hot water steeping has also been reported to make rice undergoes gelatinization which alters its physical, chemical, nutritional, rheological and viscosity properties (Sareepuang *et al.*, 2008; Mir and Bosco, 2013). It has been reported that soft and sticky cooked texture are associated with low amylose rice while the high amylose rice have hard and flaky texture (Juliano, 1985).

4.6.9 Optimisation of chemical properties of Ofada rice

Estimated coefficient of the model showing the effect of Ofada rice paddy pre-treatments on chemical properties is presented in Appendix 14 while analysis of variance for the model is in Appendix 15. From the model coefficient, increase in soaking time led to increase in moisture content, ash, protein and amylose. Whereas increase in initial soaking temperature has a positive effect on the moisture, fat,

protein, free fatty acid and amylose contents. Increase in drying temperature led to increase in ash, carbohydrate, energy and amylose content. Raising parboiling temperature causes reduction in carbohydrate and free fatty acid contents.

Adequate precision value of moisture, fat, protein, carbohydrate, energy, FFA and amylose greater than four is an indication of the suitability of the model of the design. The negative value of predicted R-squared is an indication that mean value could be used to estimate the effect of paddy pre-treatments on chemical properties. The R^2 and p-value obtained for moisture, carbohydrate, energy, FFA and amylose affirms the fitness of their respective models.

As shown in Appendix 16, optimisation goal to obtain the most suitable paddy treatment were set to have moisture content, fat and free fatty acid at minimum, while protein, carbohydrate, crude fibre, energy and amylose content were set at maximum. The solutions predicted to be suitable for the optimisation goals is as presented in Appendix 16. Among ten (10) predicted optimisation solutions, soaking duration of 1 d: 4.8 minutes, initial soaking temperature of 35^{o}C, Parboiling temperature of 98.03 ^{o}C and drying temperature of 70^{o}C had the highest desirability of 0.68.

4.7 Compounds Identifiedin Ofada rice extract by GCMS

Rice flavour is a synergy of aroma compounds which have been reported to be a complex reaction. Researchers have reported over 300 volatiles (aroma compounds) identified in both unprocessed rand cooked rice (Wakte *et al*., 2017; Champagne, 2008). In this work, a total of 266 compounds were identified in Ofada rice due to variation in processing conditions (Steeping duration, Initial steeping temperature, parboiling temperature and drying temperature). These compounds were made up of 26 acids, 30 alcohols, 28 aldehydes, 50 hydrocarbons, 13 Nitrogenous compounds, 36 ketones, 33 esters, 15 heterocyclics, 12 phenols and 23 unclassified compounds.

Sunthonvit *et al*., (2005) in a study explored the effect of drying on thai fragrant rice reported that volatiles (aldehydes, ketones, alcohol and heterocyclics compounds) are generated, evaporated and degraded during thermal processes. This is however dependent on the flavour compound and as such could enhance or degrade flavour quality of rice. It was also reported that hexanal obtained in rice samples could be due

to the thermal decomposition of linoleate and hydroperoxides (Houston, 1972). Formation of alcohol in thai rice samples is also attributed to the decomposition of fatty acids by lipoxygenase activity. However, the reaction is catalysed by heating. The presence of 1-Octen-3-ol was linked to the action of heat on hydroperoxide of linoleic acid (Hsieh *et al*., 1994).

Change in rice flavour has been reported to be dependent on pre- and post-harvest treatments. This change has been associated to changes that are evident in the composition of the volatile constituents under diverse conditions (Borompichaichartkul *et al*., 2007). As such proper pre and post harvest activites is key to maintaining the required flavour quality for rice, and this will also influence the rice yield significantly. Cultural pre and post harvest activities during rice processing is largely dependent on the geographical location. Variations in methods with respect to temperature of paddy steeping, drying, steaming, storage moisture content, storage conditions and duration are important factors that determine the acceptability of rice flavour.

A solution adopted in post harvest handling of wet paddy was the introduction of high temperature drying by the rice industry in Thailand (Soponronnarit, 1996). However, the quality of processed rice is dependent on drying temperature. High temperatures may lead to losses and changes in some components of rice. The work of Sunthonvit *et al*.,(2005) on the influence of high-temperature fluidized bed drying operation on volatile compounds in rice (Thai jasmine) reported that increasing temperature of drying from 100 to 150^{o}C might lead to increase in the quantity of 2-AP in rice. Although this is in contrast at low temperatures according to a study by Wongpornchai *et al*., (2004) who found out that the amount of 2AP in Thai jasmine rice decreases as the temperature of drying increased from 30 to 70^{o}C. However, these variations in experimental output could be linked to the different drying techniques adopted by the researchers. In the work of Borompichaichartkul *et al.,*(2007) who combined 30 min tempering between the pass or ambient air drying and drying at various high temperatures (115–150^{o}C) reported its significant effect on the composition of volatile compounds and thus making the quality of thai jasmine rice to be acceptable commercially.

Variation in perceived flavour due to the composition of a grain could be observed in two ways. The presence of flavour-active component which include alcohols, ketones and aldehyde in the grain matrix is a first major effect of grain composition (Hansen, 1995).On the other hand, the presence of flavourprecursors (fatty acids, amino acids and phenolic compounds) which are required for flavour production during processing is the second way through which perceived flavour in grains varies (Hansen, 1995).

Most of the volatiles that are produced through metabolic pathways are dependent on the variety, agronomic practices, storage conditions and post-harvest handling among other factors. Therefore, volatile profiles may have the potential to mark the identity of the variety and to interpret the quality of rice. Volatile profiles of rice varieties is analysed so that they can be used in breeding programmes for grain quality improvement and also in quality assurance studies. Difference in the rice volatile profiles has been a major factor for differentiating composition of aromatic rice from non-aromatic rice (Liyanaarachchi *et al*., 2014).

The volatile compounds in fragrant rice, which provide the characteristic aroma and flavour have been studied by a number of researchers and more than 100 volatile components have been identified in cooked rice (Tsugita *et al*., 1980; Tsugita *et al*., 1983). Yasumatsu *et al.,*(1996) found volatile carbonyl compounds such as acetaldehyde, propanol, 2-butanone, pentanal and hexanol in fragrant rice varieties and Buttery *et al*., (1983) had previously identified 2-acetyl-1-pyrroline (2AP) as the principal fragrant compound associated with aromatic rice. The compound 2AP is present in many folds in *Pandanus* species and sometimes the rice is cooked with a piece of *Pandanus latifolius* (Rampe) leaf.

The aroma of both aromatic and non-aromatic rice cultivars consists of a complex mixture of odour-active compounds. Aromatic rice has a natural nutty, popcorn flavour and accounts for 86% of imports into the United States (USDA Economic Research Service, 2010). Jasmine, Basmati and ‘‘Della’’ type of aromatic cultivars are distinguished by their grain shape, cooked rice texture, and flavour (Bergman, Bhattacharya and Ohtsubo, 2004), Genetics, growing conditions, and post-harvest handling are factors which have been shown to affect the aroma and flavour of rice (Champagne, 2008). Rice volatiles have been intensively studied as they are

important aspects of consumer acceptance (Widjaja, Craske, and Wootton, 1996a; Lam and Proctor, 2003; Wongpornchai, Dumri, Jongkaewwattana and Siri, 2004; Yoshihashi *et al.*, 2005).

4.7.1 Acids in Ofada rice

As presented in Table 4.7, a total of 27 acids were detected in Ofada rice depending on the different processing conditions (see Appendix 2A). The variation in treatments gave different number of acids in processed rice. Treatment with a day Steeping, 30^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 50^{o}C drying temperature has the lowest number (6) of volatile acids detected while treatment with 5 d steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature had the highest number (20) of acids present. For a day steeping treatments the highest and lowest number of volatile acid.detected were 17 and 6 respectively. 3 d steeping treatments had 15 and 6 respectively. The 5 d steeping treatments has the highest and lowest number of acids detected as 20 and 13.

Undecanoic acid was detected in 21 out of 25 treatments analysed for acids, while butanoic acid and linoelaidic acid were detected in 20 of the 25 treatments in this study. Acids detected in more than half of the total treatments of Ofada rice were acetic acid, butanoic acid, 2-methyl, cyclopentaneundecanoic acid, n-decanoic acid, n-hexadecanoic acid, nonanoic acid, octanoic acid and oleic acid.

Benzoic acid was only detected among 5 d steeping treatments, while Mercaptoacetic acid, 2TMS derivative was not detected among 5 d steeping treatments but detected on other steeping treatments (1 and 3 d). Oxanilic acid, O,O'-bis(trimethylsilyl) and Myristoleic acid were only detected among 1 and 3 d steeping treatments. 9-octadecenoic acid and 9-oxononanoic acid were detected among 5 d steeping treatments of paddy rice. Acetic acid and pentanoic acid which have been detected in rice by researchers (Adekoyeni *et al*., 2018) and also detected in this work are product of lipid oxidation. These compounds have been reported to negatively impact acceptability of rice. Highest percentage (0.086) of pentanoic acid was detected in this work when Ofada paddy rice was steeped for five days with an initial steeping temperature of 100^{o}C, parboiled at 80^{o}C and dried at 50^{o}C.

Table 4.7 Acids in Ofada rice with their aroma description

S/N	Name of compound	Aroma description
1	1,2-Benzenedicarboxylic acid	
2	2-Ethyl-hexoic acid	
3	9-Octadecenoic acid	
4	9-Oxononanoic acid	
5	Acetic acid	Sour, vinegar
6	Benzoic acid	
7	Butanoic acid	Sweaty, rancid
8	Butanoic acid, 2-methyl-	Cheese-like, sweaty
9	Butanoic acid, 3-methyl-	Cheese-like, sweaty
10	Cyclopentaneundecanoic acid	
11	Dodecanoic acid	
12	Heptanoic acid	
13	Hexadecenoic acid, Z-11-	
14	Hexanoic acid	Sweaty
15	Linoelaidic acid	
16	Mercaptoacetic acid, 2TMS derivative	
17	Myristoleic acid	
18	n-Decanoic acid	
19	n-Hexadecanoic acid	
20	Nonanoic acid	
21	Octanoic acid	
22	Oleic Acid	
23	Oxanilic acid, O,O'-bis(trimethylsilyl)	
24	Pentanoic acid	Fatty and green
25	Propanoic acid	
26	Tetradecanoic acid	
27	Undecanoic acid	

The lowest percentage (0.001) of pentanoic acid was detected with treatment: 3 d steeping, 30^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature. Increase in steeping duration has been linked to the formation of compounds that have negative effect on rice flavour.In the present work, the highest percentage of pentanoic acid was obtained at the maximum steeping days. The initial steeping temperature could also be linked to the reason for high percentage of pentanoic acid because the highest initial steeping temperature of 100^{o}C was able to eliminate other bacteria. Thus, allowing the rapid activity of fermenting microorganisms.

Acetic acid was detected in all the 5 d steeping treatments except when was steeped for 5 d with an initial steeping temperature of 100^{o}C, parboiled at 80^{o}C and dried at 50^{o}C. Although, the percentage detected were very low in the range of 0.024- 0.157. Whereas, detection of acetic acid among one day steeping treatments is in a comparatively high percentage (0.007 – 8.650) but was only in 6 out of the 10 steeping treatments. This could be because of high processing temperature observable in the 4 steeping treatments. Acetic acid which was detected among most of the treatemtents however in varying percentages depending on the processing conditions has been reported to also be responsible for the aromatic nature to foods. Acetic acid was detected in its highest percentage among a day steeping treatments.

Decanoic and hexanoic acids are also voaltiles that has been reported to likely be a contributor to the aromatic characteristics of rice. Being formed from the oxidation of decanol, decanoic acid has a sweaty flavour and it is used for the formation of esters which are useful for production of artificial fruit flavours and perfumes. The presence of decanoic acid in 1, 3 and 5 d steeping treatments could be a reflection of its presence in rice matrix. However, variation in processing conditions influenced the percentage detected due to their volatility and solubility. Qian and Reineccius (2002) attributed a fatty and cheese like aroma which is comparable to goat milk to the presence of hexanoic acid. The presence of hexadecanoic and oleic acid was also reported by Choi *et al*., (2007) to be the major flavour compound in soy sauces and barley bran souces. Suomalainen and Lehtonen (1978) reported that the presence of oleic and decanoic acid could be linked to fermentation processes. Oleic acid though being an unsaturated fatty acid is both odourless and colorless (Adekoyeni *et al*.,

2018). Therefore it may not necessarily have any major direct impart on perceived flavour of rice However, increase in unsaturated fatty acids due to poor post harvest handling was reported by Bryant and McClung (2011).

In the work of Sie-farth and Buettner (2014), acceptable cocoa aroma was associated with the presence of 2-acetylbenzoic. Both benzoic acid and 1,2-benzenedicarboxylic acid were mostly detected among 5 d steeping treatments. As such presence of cocoa like aroma could be attributed to the fermentation of paddy rice during steeping operation. Of important note is the presence of hexadecanoic acid found to be present in plant and is responsible for mouth feel and texture in food material. It was detected among most treatments although at different percentages.

4.7.2 Alcohols in Ofada rice

A total number of 30 alcohols as presented in Table 4.8 were detected in processed Ofada rice with different treatments having variation in the number and type of alcohols. Treatment with 5 d steeping, 30^{o}C initial steeping treatments, 80^{o}C parboiling temperature and 30^{o}C drying temperature had the highest number of fourteen alcohol detected. In this work, no alcohol was detected when paddy was steeped for 1 d, initial steeping temperature of 100^{o}C, parboiling temperature of 120 ^{o}C, drying temperature of 50 ^{o}C and when paddy was steeped for 3 d, initial steeping temperature of 30^{o}C, parboiled at 100^{o}C and dried at 50^{o}C. Generally, 1, 3 and 5 d steeping treatments had 1, 8 and 23 numbers of alcohols detected (appendix 2B).

The compound 2,3, butanediol, [R-(R*,R*)] was detected in 15 treatments. This makes it to be the most commonly detected alcohol in processed Ofada rice. 1,5-Heptadiene-3,4-diol was only detected in two treatments out of the 5 d steeping treatments. 3-methyl-1,8,9-Anthracenetriol, was detected only among 5 (five) of the 1 d steeping treatments. 1-octanol; 1-octen-3-ol; 2-Butanol; 2-Octen-1-ol, (E); 2-Penten-1-ol, (Z)-, 3-phenylpropanol, 4,7-dimethyl-5-decyne-4,7-diol, 4-allyl,-1,6-heptadiene-4-ol; 6,11-dimethyl-2,6,10-dodecatrien-1-ol; n-tridecan-1-ol and phenylethyl alcohol were only detected among five days steeping treatments. 4-ethyl alcohol, catechol, 0-methoxy-a-a-dimethyl benzyl alcohol and p-cymene-2, 5-diol were detected only among one day steeping treatments in varying percentages (Appendix 2B)

Table 4.8. Alcohols in Ofada rice with their aroma descriptions

S/N	Name of compound	Aroma decription
1	1,2-Benzenediol, o-(2-methylbenzoyl)-	
2	1,5-Heptadiene-3,4-diol	
3	1,8,9-Anthracenetriol, 3-methyl-	
4	1-Ethylcyclopropanol	
5	1-Octanol	Waxy, fatty, metallic, citrus sharp
6	1-Octen-3-ol	Mushroom, mouldy
7	1-Penten-3-ol	
8	1-Undecanol	
9	2,3-Butanediol, [R-(R*,R*)]-	Floral, oily
10	2-Butanol	
11	2-Hexen-1-ol, 2-ethyl-	
12	2-Methyl-1-undecanol	
13	2-Octen-1-ol, (E)-	
14	2-Penten-1-ol, (Z)-	
15	3-Phenylpropanol	
16	4,7-Dimethyl-5-decyne-4,7-diol	
17	4-Allyl-1,6-heptadiene-4-ol	
18	4-Amino-1-butanol	
19	4-Ethylcatechol	
20	5-[3-(4-Methoxyphenyl)oxaziridin-2-yl]pentan-1-ol	
21	6,11-Dimethyl-2,6,10-dodecatrien-1-ol	
22	Benzeneethanol, 4-hydroxy-	
23	Benzyl alcohol	
24	Catechol	
25	à-methyl-à-propyl-Cyclopropanemethanol	
26	2-(2-ethoxyethoxy),-Ethanol	
27	n-Tridecan-1-ol	
28	o-Methoxy-à,à-dimethylbenzyl alcohol	
29	p-Cymene-2,5-diol	
30	Phenylethyl Alcohol	floral

Phenylethyl alcohol was reported by to have a flowery odor and that it is found in flowers such as rose, hyacinth, neroli, ylang-ylang, geranium, and champaca (Sirilun *et al.*, 2017). Aside the importance of phenylethyl alcohol in the flavour and fragrance industry, it also acts as a preservative in food through its bacteriostatic activity (Lilley and Brewer, 1953). As such the desirable flavour of phenylethy alcohol could only be perceived in milled rice that went through five days of paddy steeping.

1-pentanol (plastic odour), 1-heptanol (green odour) and 1-octen-3-ol (mushroom odour) have been identified as odour active components in rice derived through lipid oxidation (Yang *et al*., 2008). It was also reported that the result of GC-O gave characteristics plastic odour to 1-Pentanol, a green odour to 1-heptanol and a mush room odour to 1-octen-3-ol. 1-octen-3-ol and n-pentanol were reported to be among the major odour active compounds in red and black rice in the work of Sukhonthara *et al.* (2009). 1-octen-3-ol which has been characterised with mushroom odour was only detected among 5 d steeping treatments. However, among the five days steeping treatments, 1-octen-3-ol was absent when paddy rice was processed at high processing temperatures. This might be due to the volatility of these volatiles at high temperature.Pentanol is a product of lipid oxidation of linoleic acid (Monsoor and Proctor, 2004).

4.7.3 Aldehydes in Ofada rice

The aromas generally associated with aldehydes are sweat fruit, nutty, and caramel like in nature (Fors, 1983: Xiao *et al.,* 2016). With these characteristics they enhance food flavour. Table 4.9 showed aldehydes detected. 28 aldehydes were detected cumulatively in processed rice with differences in treatments (processing conditions)(see Appendix 2C). Treatment of five days steeping 100^{o}C initial steeping temperature, 120 ^{o}C parboiling temperature and 30^{o}C drying temperature gave the highest number (17) of aldehydes in processed rice while the lowest number of aldehydes (2) was detected when paddy rice was processed with a day steeping, 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 70^{o}C drying temperature. 1, 3 and 5 d steeping treatments had 20, 14 and 26 aldehydes detected respectively with variations with other processing conditions.

Table 4.9:Aldehydes in Ofada ricewith their aroma descriptions

S/N	Compound	Aroma description
1	1,4-Benzenedicarboxaldehyde, 2-methyl-	
2	1H-Pyrrole-2-carboxaldehyde	
3	2,2-Dimethyl-4-octenal	
4	2,4-Decadienal	
5	2,4-Decadienal, (E,E)-	Fatty, green, wood
6	2,4-Heptadienal, (E,E)-	
7	2-Butenal, 2-methyl-	
8	2-Decenal, (Z)-	Fatty, green
9	2-Heptenal, (E)-	Green, heavy pungent
10	2-Octenal, (E)-	Green, nutty, herbaceous spicy
11	2-Undecenal, E-	
12	9,12-Octadecadienal	
13	Benzaldehyde	
14	Benzaldehyde, 2-methyl-	
15	Benzaldehyde, 3-methyl-	
16	Benzaldehyde, 4-hydroxy-	
17	Benzaldehyde, 4-hydroxy-3,5-dimethoxy-	
18	Benzaldehyde, 4-methoxy-	
19	Benzaldehyde, 4-methyl-	
20	Benzeneacetaldehyde	
21	cis-4,5-Epoxy-(E)-2-decenal	Metallic
22	cis-Undec-4-enal	
23	Coniferyl aldehyde	
24	Decanal	Soapy, fatty, citrusy
25	Hexanal	Green, grassy, fatty, powerful, penetrating
26	Nonanal	Green, citrusy, soapy
27	Octanal	citrusy
28	trans-4,5-Epoxy-(E)-2-decenal	

Commonly detected among most treatments were 2, 4- Decadienal; (E,E) 2,4-decadienal; 2-Heptenal, (E)-; 2-Decenal (z)-; hexanal; nonanal; trans-4,5-epoxy-(E)-2-decenal. The highest and lowest number of aldehyde detected for 1 d steeping treatments were 13 and 02 respectively while for 3 d steeping treatments were 08 and 02 respectively. And for five days steeping treatments, the highest and lowest number of aldehyde were 17 and 04 respectively. 1,4-benzenedicarboxaldehyde, 2-methyl Benzaldehyde were mostly detected among treatments with one day steeping duration.

While 2,2-Dimethyl-4-oclenal, 2,4-heptadienal, (E,E)-2-Octenal (E)-, Benzaldehyde, 4-hydroxyl-,5-dimethoxy cis-undec-4-enal, coniferyl aldehyde and decenal were detected in only among 5 d steeping treatments. In this work, the following aldehydes were not detected among treatments with 3 d steeping duration; Benzaldehyde, 2 methyl; Benzaldehyde 3-methyl-, Benzaldehyde, 4-methoxy and octanal. Benzaldehyde have significant effect on rice flavour due to the relatively low detectable odour thresholds and consequently high impacts on the overall flavor of rice (buttery *et al*., 1988).

Yang *et al*., (2008) work on identification of volatiles in black rice using GC-O reported that hexanal, nonanal, octanal, heptanal, (*E*)-2-octenal, decanal, (*E*)-2-nonenal, (*E*,*E*)-2,4-decadienal, (*E*)-2-hexenal, and (*E*)-2-decenal were aroma active compounds. These were also detected in the present study but mostly found among 3 and 5 d steeping treatments in varying percentage depending on the other processing conditions (Initial steeping temperature, parboiling temperature and drying temperature. These aldehydes have been identified to be a product of lipid oxidation through the action of lipases and lipoxygenase (Suzuki, 1999).

Nonanal, (*E*)-2-nonenal, and hexanal were classified by Yang *et al.,* (2008) to be a strong odour-active component of black rice base on their odour threshold. Octanal, heptanal, nonanal, (*E*)-2-nonenal, decanal are product of oxidation of oleic acid while pentanal, (*E*)-2-octenal, (*E*,*E*)-2,4-decadienal and hexanal are products of linoleic acid (Monsoor and Proctor, 2004). (*E*,*E*)-2,4-decadienal was reported to have the highest aroma potency after 2AP and it has been indicated to be a major contributor to rice aroma (Sunthonvit *et al*., 2005). Hexanal has been associated with a rancid odour and

hence consumers find the rice products unacceptable (Bergman *et al.,* 2000). Lam and Proctor (2003) reported a significant increase in (*E*)-2-nonenal, octanal and hexanal rice storage which has significant input in off-flavour perception. (*E*)-2-nonenal, octanal and hexanal are linked to rancid, fatty and green odour in rice respectively. Hexanal and octanal have significant effect on rice flavour due to the relatively low detectable odor thresholds and consequently high impacts on the overall flavor of rice (buttery *et al*., 1988)

4.7.4 Hydrocarbons in Ofada rice

Presented in Table 4.10 (and appendix 2D) is the hydrocarbon detected for different treatments used in the production of Ofada rice. A total of 50 hydrocarbons were found in processed Ofada rice. 31 out of 50 hydrocarbons were detected with paddy treatments of 5 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. 40, 35 and 46 hydrocarbon was detected in 1, 3 and 5 d steeping treatments respectively.

Commonly detected among most treatments were 1, 3-dimethyl-benzene, (S)-methylethenyl, decane, eicosane 2,6,10,14-tetramethyl heptadecane, hexadecane, octacosane, pentadecane;1,1,1,5,5,5-hexamethyl 1-3,3-tridecane trisiloxane and undecane 30, 25 and 31 hydrocarbons were the highest number of detected for 1, 3 and 5 d steeping treatments. This was also obtained for 1:E-11, z-13-octadecatriene; decane, 8-methyl decane heptasiloxane hexadecamethyl and propyne. The following compounds were only detected when paddy rice was steeped for 5 d though with variation in other processing conditions (initial steeping temperature, parboiling temperature and drying temperature): 1,8,11-heptadecatriene, (7,8); 1H-indene, 1-ethyldiene; benzene 1,2,4,5-tetramethyl-;ethylbenzene; heptylcyclohexane and pentane, 1-ethenyloxyl cmp hydrocarbons found to be peculiar to 1 and 3 d steeping treatments only were 1,3-dioxlane, 2-heptyl-; cyclopenta siloxane, decamethyl-, hexalosane and spiro [2,4]hept-5-ene, 5-trimethylsilylmethyl-1-trimethylsily-.

Table 4.10 Hydrocarbonsin Ofada rice

S/N	Compound
1	1,3,5,7-Tetroxane
2	1,3-Dioxolane, 2-heptyl-
3	1,8,11-Heptadecatriene, (Z,Z)-
4	1,E-11,Z-13-Octadecatriene
5	1-Decene, 8-methyl-
6	1H-Indene, 1-ethylidene-
7	1-Iodo-2-methylundecane
8	1-Undecene, 4-methyl-
9	2,6-Dimethyldecane
10	Benzene, (1-methylethyl)-
11	Benzene, 1,2,3-trimethyl-
12	Benzene, 1,2,4,5-tetramethyl-
13	Benzene, 1,3-dimethyl-
14	Butane, 2,2-dimethyl-
15	Cyclohexene, 1-methyl-4-(1-methylethenyl)-, (S)-
16	Cyclopentasiloxane, decamethyl-
17	Cyclopentene, 1,2,3-trimethyl-
18	Cyclopropene
19	Decane
20	Decane, 2,3,5,8-tetramethyl-
21	Decane, 4-methyl-
22	Dodecane, 2,6,10-trimethyl-
23	Dotriacontane
24	Eicosane
25	Ethylbenzene
26	Heneicosane
27	Heptacosane
28	Heptadecane, 2,6,10,14-tetramethyl-
29	Heptane, 2,2-dimethyl-
30	Heptasiloxane, hexadecamethyl-
31	Heptylcyclohexane
32	Hexacosane
33	Hexadecane

34	Nonadecane
35	Nonane
36	Octacosane
37	Octadecane
38	Octane
39	Oxetane, 3-(1-methylethyl)-
40	Pentadecane
41	Pentane, 1-(ethenyloxy)-
42	Propane, 1-(ethynylsulfinyl)-
43	Propyne
44	Spiro[2.4]hept-5-ene, 5-trimethylsilylmethyl-1-trimethylsilyl-
45	Tetracosane
46	Tetradecane
47	Toluene
48	Tridecane
49	Trisiloxane, 1,1,1,5,5,5-hexamethyl-3,3-bis[(trimethylsilyl)oxy]-
50	Undecane

Thirteen (13) nitrogenous compounds were detected in processed Ofada rice as shown in Table 4.10. paddy rice treatment with 5 d steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature had the highest (9) number of nitrogenous compounds while they were not detected when paddy rice was steeped for 1 d with initial steeping temperature of 30^{o}C, 100^{o}C parboiling temperature and 70^{o}C drying temperature. Ten out of thirteen compounds detected were present in 1 d steeping treatments while only three were detected in 3 d steeping treatments. And twelve amides were found in 5 d steeping treatments. The highest number of compounds (7) present in 1 d steeping treatments was obtained when paddy rice was steeped for 1 d with 100^{o}C initial steeping temperature. The treatment that had the highest number of compounds among 3 d steeping treatments was 3 d steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature (Appendix 2E).

1, 2, 5-oxadiazole-3,4-dicarboxamide, 4 TMS derivative was only detected in one day steeping treatments and absent in other days (three and five) steeping treatments. It was only five days steeping treatments that had 1-tetradecanamine, N,N-dimethyl-; formamide, (2-acetylphenyl)- and Nonanamide. 1 and 5 d steeping treatments were the only treatments that had 1-Butanamine; 1-decanamine; 1-Naphthalenamine; N-phenyl-; 9-octadecenamide (7)-; Hexadecanamide; and methyl-methoxyl-hydroxymethyl-amine. Among all the amide detected, formamide, N,N-dibutyl- was commonly found among most treatments.

Table 4:11.Nitrogenous compounds in Ofada rice

S/N	Compound
1	1,2,5-Oxadiazole-3,4-dicarboxamide, 4TMS derivative
2	1-Butanamine
3	1-Decanamine
4	1-Naphthalenamine, N-phenyl-
5	1-Tetradecanamine, N,N-dimethyl-
6	2-Naphthalenamine, N-phenyl-
7	9-Octadecenamide, (Z)-
8	Formamide, (2-acetylphenyl)-
9	Formamide, N,N-dibutyl-
10	Hexadecanamide
11	Methyl-methoxy-hydroxymethyl-amine
12	Nonanamide
13	Norfenfluramine

4.7.6 Ketones in Ofada rice

Results showing the ketones detected is as presented in Table 4.12. a total of 36 ketones were found in processed rice cumulatively from different processing conditions. Treatment of paddy rice with 5 d steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 30^{o}C drying temperature had the highest number (22) of ketones. The most common ketones detected among most treatments was 2-Butanone. One, three and five days steeping treatments had 17, 10 and 32 ketones detected among treatments respectively which shows that 5 d steeping treatments had the highest ketones determined compared to other days steeping treatments (appendix 2F).

Five days steeping treatments had the following ketones peculiar to it only: 1-Butane, 1-Phenyl; 1-hepten-3-one; 1-pentene-3-one, 4-methyl, 2,5-cyclohexadien-1-one; 1-one, 2, 6-bis (1,1-dimethylethyl)-4-hydroxy-4methyl; 2-dodecanone, 2-octanone, 2-piperidione, 3,3-dimethyl-4-(N-(2-methylbenzyl amino)-butane-2-one, 3-buten-2-one, 3-methyl,3-methyl pyridazin-5-one, 5,9-undecadien-2-one, 6,10-dimethyl 1-,(E)-a –(N,N-dimethylamino)-4-hydroxylacetophenone, cyclohexanone, 2-octyl-and imidazolidin-2-one, 1-[2-(2-methoxyphenoxy) acetyl)]-. Detected among 1 and 5 d steeping treatments were 2(3H)-furanone, 5-butyldihydro-, 2-sec-butycyclohexanone, 2-tetradecanone, 3,4-hexanedione, 2,2,5-trimethyl, 3-hexanone, 2,5-dimethyl 4-nitro-7,9-di-tent-butyl-1-oxaspiro (4,5)deca-6,9-diene-2,8-dione. However, 2H, 8H-benzol[1,2-b:5,4-b'] dipyran-2,8-dione, 4,6-dimethyl-, 3,4-dimethylmethcathinone, 4H-1-benzopyran-4-one, 7-hydroxy-2,5-dimethyl- were only detected among one day steeping treatments

.

The compound 3-Hydroxy-4,5-dimethyl-2(5*H*)- furanone was reported to be a thermally derived products that impart a seasoning-like flavour in rice. Lipid derived odor active compounds (3-octen-2-one and 2-nonanone) detected in black rice as reported by Yang *et al.,*(2008) was also identified in the present study. 2-heptanone is also a product of lipid oxidation of oleic acid present in rice (Monsoor and Proctor, 2004). 3-octen-2-one has been reported to have a rose like odour while 2-nonanone had a fruity odor).

Table 4.12:Ketones in Ofada ricewith their aroma descriptions

S/N	Compound	Aroma description
1	1-Butanone, 1-phenyl-	
2	1-Hepten-3-one	
3	1-Penten-3-one, 4-methyl-	
4	2(3H)-Furanone, 5-butyldihydro-	
5	2,5-cyclohexadien-1-one, 2,6-bis(1,1-dimethylethyl)-4-hydroxy-4-methyl-	
6	2,5-Dichloro-3,6-bis[ç-dimethylaminopropylamino]benzoquinone	
7	2,5-di-tert-Butyl-1,4-benzoquinone	
8	2-Butanone	
9	2-Dodecanone	
10	2H,8H-Benzo[1,2-b:5,4-b']dipyran-2,8-dione, 4,6-dimethyl-	
11	2-Heptanone, 4-methyl-	Fruity, spicy, frangrant
12	2-Hexanone, 4-methyl-	
13	2H-Pyran-2-one, tetrahydro-6-pentyl-	
14	2-Octanone	
15	2-Piperidinone	
16	2-Pyrrolidinone, 5-(ethoxymethyl)-	
17	2-Sec-Butylcyclohexanone	
18	2-Tetradecanone	
19	2-undecanone, 6,10-dimethyl	
20	3,3-Dimethyl-4-(N-(2-methylbenzyl)amino)-butan-2-one	
21	3,4-Dimethylmethcathinone	
22	3,4-Hexanedione, 2,2,5-trimethyl-	
23	3-Buten-2-one, 3-methyl-	
24	3-Hexanone, 2,5-dimethyl-4-nitro-	
25	3-Methylpyridazin-5-one	
26	3-Pentanone, 2,4-dimethyl-	
27	4H-1-Benzopyran-4-one, 7-hydroxy-2,5-dimethyl-	
28	4-Hepten-2-one, (E)-	
29	5,9-Dodecadien-2-one, 6,10-dimethyl-, (E,E))-	
30	5,9-Undecadien-2-one, 6,10-dimethyl-, (E)-	
31	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	
32	à-(N,N-Dimethylamino)-4'-hydroxyacetophenone	
33	Acetophenone	
34	Cyclohexanone, 2-octyl-	
35	Ethanone, 1-cyclopropyl-	
36	Imidazolidin-2-one, 1-[2-(2-methoxyphenoxy)acetyl]-	

4.7.7 Heterocyclics in Ofada rice

Fifteen heterocyclics were found in processed Ofada rice as present in Table 4.13. the most commonly detected among treatments were 1,4:3,6-Dianhydro-a-d-glycopyranose, Furan, 2-pentyl- and furan, tetrahydro-2-methyl-. Paddy rice treatment with 5 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature had eight as the highest number of heterocyclics among all other treatments. 1,3, and 5 d steeping treatments had 10, 06 and 13 heterocyclics detected respectively. The highest number of heterocyclics was detected among the 3 days steeping treatments (see appendix 2G).

A day steeping treatments were the only treatments that had 1,6:3,4-dianhydro-2-0-acetyl-a-d-allopyranose present as a heterocyclics. One and three days steeping treatments had 1,4:3,6-Dianhydro-a-d-glucopyranose and 2 methyl-5-hydroxy benzofuran common among their treatments while these two heterocyclics were absent in 5 d steeping treatments. 2-(4-hydroxy-4-methyl-tetrahydro-pyran-3-ylamino)-3-(1H-inol-2-yl0-propionic acid was only detected among 3 and 5 d steeping treatments.

Heterocylics common to only 5 d steeping treatment were 2(3H)-Furanone, dihydro-5-pentyl-, 3,4-Diamino-1,2,4(4H)-triazole and methyl 2-0- methyl-a-D-xylopyranoside-. One and five days steeping treatments had the following common to their treatments:1,3-Dithiolo[4,5-b] Furan, tetrahydro-3a-methyl-; a-D-Glucopyranose, 1,6-anhydro-, 2-pentyl furan and methyl2,3,4,6,7-penta-0-methyl-a-Lglycero-D-manoheptopyranoside.

Pyrazine, methoxypyrazine, pyrrole, pyridine, pyrroline, pyrrolidine, pyrrolizine and piperine are nitrogenous heterocyclics that are formed through maillard reaction.This is among the major route for aroma formation, while oxygen containing heterocyclic compounds formed are maltol, furaneol, cyclotene, oxazole and oxazoline, and sulfur-containing heterocyclic compounds (thiazole and thiophenes) (Reineccius, 2006). The compound 2-methylpyridine was reported to have ash odor and it is a nitrogenous compounds reported to be derived from the rice bran (buttery *et al., 1983*).

Formation of heterocyclics in food is mainly through non-enzymatic browning reaction or maillard reaction. High processing temperature of processing was

Table 4.13:Heterocycliccompounds in Ofada ricewith aroma description

S/N	Compound	Aroma description
1	1,3-Dithiolo[4,5-b]furan, tetrahydro-3a-methyl-	
2	1,4:3,6-Dianhydro-à-d-glucopyranose	
3	1,6:3,4-Dianhydro-2-O-acetyl-á-d-allopyranose	
4	2(3H)-Furanone, dihydro-5-pentyl-	
	2-(4-Hydroxy-4-methyl-tetrahydro-pyran-3-	
5	ylamino)-3-(1H-indol-2-yl)-propionic acid	
6	2-Methyl-5-hydroxybenzofuran	
7	2-n-Butyl furan	
8	3,4-Diamino-1,2,4(4H)-triazole	
9	á-D-Glucopyranose, 1,6-anhydro-	
10	Benzofuran, 2,3-dihydro-	
11	Furan, 2,3-dihydro-4-methyl-	
12	Furan, 2-pentyl-	
13	Furan, tetrahydro-2-methyl-	Banana-like, vegetable green, bean
	Methyl 2,3,4,6,7-penta-O-methyl-à-L-glycero-D-	
14	mannoheptopyranoside	
15	Methyl 2-O-methyl-á-D-xylopyranoside	

identified as a factor that could be responsible for increase in the heterocyclics that influence rice aroma (Sunthonvit *et al*., 2005).Maillard reaction which is known to be temperature dependent has been indicated in the formation of 2AP in cooked rice (Maga, 1984). The effect of temperature was reported to give oat its desired flavour and also inactivation of lipolytic enzymes in oats (Molteberg *et al.* 1996). This was also corroborated by Sides *et al*., (2001) who reported that the cereal like flavour in oat could be associated to the heat treatments. Parker *et al*., (2000) reported the enhancement of Maillard reaction by high temperature during extrusion and described the product as a toasted and cereal like flavour.

4.7.8 Esters in Ofada rice

Esters have a fruity floral impart on food flavour and they could also be responsible for masking the intensity of free fatty acids off flavours in food (Ojinnaka and Ojimelukwe, 2013). Formation of esters is through the esterirification process of alcohols and the short chain free fatty acids in foods (Qin and Ding, 2007). The result of esters detected in different treatments during paddy rice processing is as presented in Table 4.14. A total of 33 esters were detected with the highest of 23 esters from a particular treatment (5 d steeping, 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 30^{o}C drying temperature). It was also observed that all the 33 esters were detected among 5 d steeping treatments. However, 24 and 15 were detected among 1 and 3 d steeping treatments respectively (Appendix 2H).

Dibutyl phthalate, 3-(4-methoxyphenyl)-, 2-ethylhexylester; 4-Nitrosophenyl-a-phenylpropionate; 2-propanoic acid and Dodecanone acid, 1-methylethyl ester were most commonly detected in all treatments 5 d steeping treatments were the only treatments that had 2,3-dimethylphenyl isocyanate, 4-Nitrosophenyl-a-phenylpropionate; Dodecanoic acid, 2-penten-1-yl ester, and n-butylether. esters common to both 1 and 5 d steeping treatments only were 1,2-Benzene dicarboxylic acid, bis(8-methylnonyl) ester, BenzylBenzoate,, Bis (tridecyl) phthalate; Butanoic acid, butyl ester; Dicyclohexylphthalate, phthalic acid,2,2-diurethyl pent-3-yl dodecylester; Didecan-2-yl phthalate; n-propylacetate, octadecanoic acid, ethyl ester; undecanoic acid, 11-bromo-methyl ester; undecanoic acid, ethylester and undecanoic acid methyl ester. The following esters were only found among 3 and 5 d steeping treatments: Adipic acid, 2-ethylhexyl isohexyl ester, ethyl 6,9,12-hexadecatrienoate.

Table 4.14: Esters in Ofada rice

S/N	Compound
1	1,2-Benzenedicarboxylic acid, bis(8-methylnonyl) ester
2	2,3-Dimethylphenyl isocyanate
3	2-Propenoic acid, 3-(4-methoxyphenyl)-, 2-ethylhexyl ester
4	4-Nitrosophenyl-á-phenylpropionate
5	9,12-Octadecadienoic acid, methyl ester, (E,E)-
6	9-Octadecenoic acid (Z)-, methyl ester
7	Acetate, 4-hydroxy-3-methyl-2-butenyl-
8	Benzyl Benzoate
9	Bis(2-ethylhexyl) phthalate
10	Bis(tridecyl) phthalate
11	Butanoic acid, butyl ester
12	Butyric acid, 4-pentadecyl ester
13	Dibutyl phthalate
14	Dicyclohexyl phthalate
15	Oxalic acid, butyl 2-ethylhexyl ester
16	Phthalic acid, 2,2-dimethylpent-3-yl dodecyl ester
17	Adipic acid, 2-ethylhexyl isohexyl ester
18	Didecan-2-yl phthalate
19	Diisooctyl phthalate
20	Dodecanoic acid, 1-methylethyl ester
21	Dodecanoic acid, 2-penten-1-yl ester
22	Dodecanoic acid, methyl ester
23	Dodecyl acrylate
24	Ethyl 2-cyano-3-methyl-2-(O-methylbenzyl)butanoate
25	Ethyl 6,9,12-hexadecatrienoate
26	Heptadecyl acetate
27	n-Butyl ether
28	n-Propyl acetate
29	Octadecanoic acid, ethyl ester
30	Phthalic acid, 2,2-dimethylpent-3-yl dodecyl ester
31	Undecanoic acid, 11-bromo-, methyl ester
32	Undecanoic acid, ethyl ester
33	Undecanoic acid, methyl ester

4.7.9 Phenolic compounds in Ofada rice

Twelve phenolic compounds were detected in all treatments for Ofada rice processing as presented in Table 4.15. Treatments of paddy rice with 1 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature gave the highest (09) number of phenolic compound among other treatments.Phenol, 4-ethylphenol and vanillin were the most detected among all treatments with 23, 24 and 15 treatments having them present respectively.

Phenolics detected were 10, 06 and 11 with 1, 3 and 5 d steeping treatments (See Appendix 2I) while phenol 2,6-bis(1,1-dimethylethyl)-4 methyl-methylcarbonate was found to be present in only 3 and 5 d steeping treatments. Commonly detected among 1 and 5 d steeping treatments were 2-methoxyl-4-vinyl phenol, phenol, 2-(2H-1,2,3-benzotriazol-2-yl)-4,6-bis(1,1-dimethyl ethyl)-, 2-ethylphenol and 2-methoxyphenol.2-methoxy-4-vinylphenol, 4-vinylguaiacol and 4-vinylphenol were produced when a combination of thermal and enzymatic action takes place with cooked rice. These compounds produce an undesirable pharmaceutical odour (Coghe *et al*., 2004). These odourants are produced through the decarboxylation of ferulic acid.

Vanillin was detected among all 5 d steeping treatment. Vanillin is associated with vanilla flavour used for food flavouring because of its desirable characteristics. Steeping duration might be a factor that determines the detection of vanillin in rice since it was only found among 5 d steeping treatment. Flavour perception of vanillin in food is significantly affected by the interactions of vanillin and proteins (Weerawatanakorn *et al*., 2015). Decrease in vanillin flavour could also affect flavour release during comsumption of the food material.

Table 4.15a. Serial numbers of phenolic compounds with their aroma description

S/N	Compound	Aroma description
1	2,4-Di-tert-butylphenol	
2	2-Methoxy-4-vinylphenol	Spicy, clove-like
3	Butane, 2-phenyl-3-hydroxy-4-cyano-	
4	Phenol	
5	phenol, 2-(2H-1,2,3-benzotriazol-2-yl)-4,6-bis(1,1-dimethylethyl)-	
6	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl-	
7	Phenol, 2,6-bis(1,1-dimethylethyl)-4-methyl-, methylcarbamate	
8	Phenol, 2-ethyl-	
9	Phenol, 2-methoxy-	smoky
10	Phenol, 2-methyl-5-(1-methylethyl)-	
11	Phenol, 4-ethyl-	
12	Vanillin	Vanilla-like, sweet

4.7.10 Miscellanous compounds detected in Ofada rice

Presented in Table 4.16 is the percentage of 26 unclassified compounds detected in Ofada rice.Commonly detected among all treatments were butylated hydroxytoluene, 3-methyl-indole naphtathalene, 1,4,5-trimethyl- and pyrene. Detected among 5 d steeping treatmentsalone were Apocynin; indole; L-(1)-threose, aldononitrite, triacetate; pregnane, 20-[5-methyl-2-oxanyl]-and p-xylene while 3-(4-hydroxyl-2 (1H)-oxo-3-quinolyl) propionitrite and 4-ethoxystyrene was found only in one day steeping treatments. Common to both 3 and 5 d steeping treatments was 0-cymene (see Appendix 2 J). The results of the unclassified compounds found in Ofada rice had some compounds that are found in both 1 and 5 d steeping treatments which are 1, 2-benzylsothiazole;4,8,12,16-tetramethylheptadecan-4-olide;4,8,12-trimethyltridecan-4-olide; fernesolisomera, Norfenfluramine and thymol.

Indole which is a nitrogen containing compounds mostly found in rice bran has a sour odour as reported by Buttery *et al., (*1983*)*. Yoshihashi *et al.*, (2002) ascribed a mothball aroma to indole detected in rice. However, Adekoyeni *et al*., (2018) attrributed a sweet, floral and burnt aroma to the presence of indole in food material. Indole was only detected among 5 d steeping treatment. This showed that higher steeping duration is a major determinant of the presence of indole which could be one of the major aroma components that give Ofada rice its relish flavour. Slightly sweet aroma was also reported to be associated with the presence of benzothiazole (Bryant and McClung, 2011). p-cresol identified mostly among 3 and 5 d steeping treatments was reported as a potent off-flavour compound in citral degradation (Ueno *et al*., 2004; Schieberle *et al*., 1988). The presence of butylated hydroxyl toluene (BHT) has no effect on formation of undesirable flavour compounds through citral degradation (Kimura *et al*., 1983).

There are several studies that have evaluated a number of rice cultivars from different genetic backgrounds and the results showed a significant qualitative and quantitative variation in flavor compounds (Bergman *et al*., 2000; Laguerre *et al.*, 2007; Yang*et al*., 2008a; Zeng *et al.*, 2008). Rice storage duration and variation in storage condition have been reported to lead to depletion of desirable aroma compounds and production of undesirable aroma compounds (Widjaja, Craske, and Wootton, 1996a; Suzuki *et al*., 1999; Zhou, Robards, Helliwell, and Blanchard, 2002; Wongpornchai, Dumri,

Table 4.16miscellanous compound with aroma description

S/N	Compound	Aroma description
1	1,2-Benzisothiazole	
2	2,6-Diisopropylnaphthalene	
3	3-(4-Hydroxy-2(1H)-oxo-3-quinolyl)propionitrile	
4	3,4-Diamino-1,2,4(4H)-triazole	
5	4,8,12,16-Tetramethylheptadecan-4-olide	
6	4,8,12-Trimethyltridecan-4-olide	
7	4-Ethoxystyrene	
8	Apocynin	
9	Azulene	
10	Butylated Hydroxytoluene	
11	Farnesol isomer a	
12	Indole	Sweet, burnt
13	Indole, 3-methyl-	
14	L-(+)-Threose, aldononitrile, triacetate	
15	Mesitylene	
16	Naphthalene, 1,4,5-trimethyl-	
17	n-Butyl ether	
18	Norfenfluramine	
19	o-Cymene	
20	o-Xylene	
21	p-Cresol	
22	Pregnane, 20-[5-methyl-2-oxanyl]-	
23	p-Xylene	
24	Pyrene	
25	Squalene	
26	Thymol	

Jongkaewwattana, and Siri, 2004; Tulyathan, Srisupattarawanich, and Suwanagul, 2008). Volatiles that contributed to rice variety aroma are aldehydes, ketones, alcohols and heterocyclic compounds (Liyanaarachchi *et al*., 2014). In the work of Yang *et al*., (2008) on the characterisation of aromatic volatiles in black rice, 2-AP, guaiacol, indole, and *p*-xylene largely influenced the difference between the aroma in cooked black and white rice.

4.8 Sensory acceptability of Ofada rice

Result of sensory evaluation with respect to appearance, taste, aroma, mouthfeel, off flavour and overall acceptability is as presented in Table 4.17. The most acceptable with respect to appearance is sample that was steeped for 5 d with initial steeping temperature of 30^{o}C, parboiling temperature of 80^{o}C, and dried at 30^{o}C while the least acceptable with respect to appearance was when paddy was steeped for 3 d, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature. The most liked with respect to appearance among a day steeping treatments was 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature while for 3 d steeping treatment was 30 ^{o}C initial steeping temperature, 120^{o}C parboiling temperature, and 70^{o}C drying temperature.

With respect to appearance, lower initial steeping temperature gave milled rice liked mostly by panellist while at higher initial steeping temperature; panellist assessment of the cooked Ofada rice is low. This could be due to severity of high temperature on the paddy rice which later affected the efficiency of the milling operation and thus Ofada rice appearance. Despite the variation in likeness, significant differences ($p<0.05$) does not exist in appearance among processed rice produced with different treatments. This shows that variation in the processing conditions does not affect significantly the acceptability of Ofada rice.

Panelist adjudged the highest likeness in taste to rice processes by paddy steeping of a day, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature while the least liked taste is from paddy rice steeped for 5 d, initial steeping temperature of 30^{o}C, parboiled at 80^{o}C and dried at 30^{o}C. There exists significant difference with respect to taste between the most and least liked by

Table 4.17. Result of sensory evaluation of Ofada rice

Run	Steeping duration (Days)	Initial Steeping Temperature (°C)	Parboiling Temperature (°C)	Drying Temperature (°C)	Appearance	Taste	Aroma	mouth feel	Off flavour	overall acceptability
2	1	30	80	50	7.33^{abcd}	6.83^{f}	6.56^{de}	6.0oC^{d}	5.00^{bcdef}	7.22^{ghij}
6	1	30	100	70	6.88^{abcd}	6.47^{def}	6.82^{e}	6.59^{d}	4.67^{bcde}	6.88^{efghi}
15	1	30	120	30	6.11^{abcd}	6.33^{def}	6.39^{cde}	6.11^{d}	5.46^{defg}	6.61^{defgh}
24	1	30	120	30	5.78^{abcd}	6.0oC^{def}	6.00^{bcde}	5.83^{cd}	4.44^{abcde}	6.33^{cdefgh}
8	1	65	100	50	5.44^{ab}	5.39^{abcde}	5.44^{bcd}	5.33^{bcd}	5.27^{cdef}	6.06^{bcdefg}
11	1	65	120	70	8.00^{abcd}	6.71^{ef}	6.76^{e}	6.59^{d}	6.20^{efgh}	7.44^{hij}
18	1	100	80	70	6.44^{abcd}	6.17^{def}	6.11^{bcde}	6.56^{d}	6.8^{fgh}	6.18^{cdefgh}
20	1	100	80	30	6.78^{abcd}	6.44^{def}	5.89^{bcde}	6.33^{d}	5.89^{defg}	6.35^{cdefgh}
25	1	100	80	30	6.89^{abcd}	6.58^{def}	5.95^{bcde}	6.11^{d}	6.11^{efgh}	6.56^{defgh}
13	1	100	120	50	5.50^{abc}	6.33^{def}	5.83^{bcde}	5.78^{cd}	5.58^{defg}	5.65^{bcde}
16	3	30	100	50	6.44^{abcd}	5.94^{bcdef}	5.89^{bcde}	6.22^{d}	5.11^{bcdef}	7.06^{fghij}
12	3	30	120	70	6.50^{abcd}	5.56^{abcdef}	5.56^{bcde}	5.44^{bcd}	4.11^{abcde}	5.88^{bcdef}
7	3	65	100	70	5.72^{abcd}	4.78^{abc}	5.00^{b}	4.67^{abc}	3.86^{abcd}	5.18^{bc}
14	3	65	100	40	6.50^{abcd}	5.61^{abcdef}	5.89^{bcde}	5.33^{bcd}	5.80^{defg}	5.88^{bcdef}
5	3	100	100	50	5.33^{a}	4.67^{ab}	3.78^{a}	3.89^{a}	2.62^{a}	4.82^{b}
9	5	30	80	30	8.44^{cd}	4.44^{a}	3.72^{a}	3.89^{a}	3.20^{abc}	3.71^{a}
10	5	30	80	30	7.22^{abcd}	6.67^{ef}	6.11^{bcde}	5.65^{bcd}	4.25^{abcde}	6.50^{defgh}
17	5	30	80	70	6.39^{abcd}	6.06^{cdef}	5.94^{bcde}	6.0oC^{d}	4.10^{abcde}	5.94b^{cdefg}
1	5	30	120	50	5.61^{abcd}	5.22^{abcd}	5.22^{bc}	4.39^{ab}	3.17^{ab}	5.41^{bcd}
21	5	65	100	50	6.67^{abcd}	6.11^{def}	6.39^{cde}	6.0oC^{d}	6.14^{efgh}	6.67^{defgh}
23	5	100	80	50	6.83^{abcd}	6.44^{def}	6.44^{cde}	5.61^{bcd}	5.22^{bcdef}	6.78^{efghi}
3	5	100	120	70	6.78^{abcd}	5.82^{bcdef}	6.11^{bcde}	6.06^{cd}	5.13^{bcdef}	5.83^{bcdef}
4	5	100	120	30	6.78^{abcd}	6.17^{def}	6.61^{de}	6.28^{d}	5.00^{bcdef}	6.94^{efghi}
19	5	100	120	30	6.44^{abcd}	5.71^{abcdef}	5.67^{bcde}	5.71^{bcd}	5.44^{defg}	6.33^{cdefgh}
22	5	100	120	70	6.44^{abcd}	6.67^{ef}	6.17^{bcde}	6.28^{d}	6.09^{efgh}	6.94efghi

Means in a column having same letter are significantly not different ($p > 0.05$).

panellist at 5% level of significance. Variation in taste could be linked to the steeping duration which leads to the production of some undesirable flavour compounds.

The most liked taste of processed rice among 3 d steeping treatments was obtained with 30^{o}C initial steeping temperature, 100^{o}C initial steeping temperature and 50^{o}C drying temperature. Significant difference does not exist among all 3 d steeping treatments in panellist taste assessment at 5% level of significance. While for 5 d steeping treatments the most liked taste were from 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature. Significant difference ($P<0.05$) does not exist in taste among 5 d steeping treatment. This is an indication of the predominant effect of steeping duration on the taste of processed rice in this study. As such variation in initial steeping temperature, parboiling and drying temperatures does not affect Ofada rice taste.

Rice aroma most liked by panellists was from rice processed with 1 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature. While the least acceptable with dislike from panellists, was when paddy rice was processed with 5 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. The most liked aroma of Ofada rice for 3 d steeping treatment was from 65 ^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 40^{o}C drying temperature. While for 5 d steeping treatments most liked aroma is from 100^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature.

The work of Noble (1996) described the relationship that existed between taste and aroma. It was observed that the apparent intensity of taste may lead to increase in the aroma while the apparent intensity of aroma might also lead to increase in taste. Complexity in the factors that determines flavour perception also makes flavour description of a food to be a herculean task even with the use of standardised method during sensory evaluation. Variation in senses of flavour due to individual experiences and knowledge is a factor of consideration in understanding flavour perception and standardisation.

Off flavour disliked most by panellist were from 3 and 5 d steeping treatments. For three days steeping treatments, the most dislike off- flavour is from 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50 ^{o}C drying temperature while for 5 d steeping treatments is form 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. For 1 d steeping treatments, most acceptable level of off-flavour is from 65^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature. While the most acceptable for 3 d steeping treatments is from 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 40^{o}C drying temperature. And for 5 d steeping treatment, the most acceptable level of aroma is from 65^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature.

Aside the steeping duration, the level of acceptability of off-flavour of Ofada rice is dependent on the initial steeping temperature since the most acceptable off flavour for each days steeping treatment was when the initial steeping temperature was 65^{o}C. The desireable characteristic taste of rice could be linked to flavour and fragrance components such as 2-acetyl-1-pyrroline (2AP) and the aromatic alcohols, while the negative effect could be off-flavours of hexanal and 2-pentylfuran amongst others.In Nigeria, affinity for Ofada rice is primarily due to its unique flavour, but consumers are sometimes put off by the inconsistencies in flavour and the off flavour present. This could be attributed mainly to the processing method adopted (Adeniran *et al.*, 2012). Hot water soaking of rice paddy was reported to have eliminated undesireable fermented odour in milled rice (Anuonye *et al.* 2016).

With respect to mouth feel, a day steeping, 65^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature had the highest value of likeness while the lowest was from 3 d steeping, 100^{o}C initial steeping temperature, 100^{o}C parboiling temperature, 80^{o}C drying temperature and 5 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. The most liked among the 3 d steeping treatments with respect to mouth feel is when rice paddy was steeped for 3 d with initial steeping temperature of 30^{o}C, 100^{o}C parboiling temperature and 50^{o}C drying temperature. While 5 d steeping treatments had the most liked processed rice with 100^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature

On the overall acceptability of Ofada rice, treatment with one day steeping, 65^{o}C initial steeping temperature, 120^{o}C parboiling temperature and 70^{o}C drying temperature had the highest acceptability while the lowest acceptable on the overall was from 5 d steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 30^{o}C drying temperature. Significant difference existed between the overall most and least acceptable processed rice at 5% level of significance. This variation may be linked to the synergistic effect of steeping duration and other processing conditions adopted in this work. High processing temperature of 120^{o}C parboiling temperature and 70^{o}C drying temperature gave the highest overall acceptability of processed rice. However, the overall least acceptable was obtained from paddy processed at low temperature. The most acceptable milled rice was obtained with paddy processing of 3 d steeping treatments with 30^{o}C initial steeping temperature, 100^{o}C parboiling temperature and 50^{o}C drying temperature.

Over the years, consumers have been able to define four basic tastes in food product which include sweet, salty, sour and bitter. However other tastes such as umami and metallic have evolved in both definition and acceptance. It is worthy of note that researchers varies in their opinion on the definition of this basic taste in food due to the technicality in the synesrgistic effect in their interactions (Breslin, 1996; Brannan *et al*., 2001; Keast and Breslin, 2002). Flavour compounds have their unique taste when found alone, however the concentration of the taste solutions is a major factor in the taste interactions (Keast and Breslin 2002). For example, Breslin (1996) noted that moderate concentrations of salts and acids enhance each others flavour, while they suppress themselves at high concentrations.

4.9 Correlations between sensory attributes and aroma active compounds

Presented in Table 4.18 is the correlation loadings of the principal components of the correlations between sensory attributes and aroma active compounds in Ofada rice sample. The frst five components accouted for over 99% of the variance (Appendix 17. The loadings plot presented in Appendix 18 on the principal component one (PC 1) showed that the sensory attributes except appearance positively correlated with all the paddy pre-treatments conditions. The off-flavour attribute correlated positively and closely to initial steeping temperature, butanoic acid, 2-methy butanoic

Table 4.18: Correlation loadings of the first five components

Variables	PC 1	PC 2	PC 3	PC 4	PC 5
Steeping duration	0.051307	0.040652	-0.03159	-0.88539	0.18564
Initial Steeping Temperature	0.99975	-0.01985	0.010659	6.82E-05	-2.71E-05
Parboiling temperature	0.075392	0.85354	-0.51551	0.002562	7.06E-06
Drying temperature	0.003827	0.61589	0.78781	-0.00016	-0.00204
Decanal	0.039439	0.058684	0.2738	0.14449	0.051134
Appearance	-0.15937	-0.3152	0.13841	-0.02921	0.20377
Taste	0.10093	-0.0636	0.05861	0.504	0.64391
Aroma	0.023705	0.16542	0.047978	0.4172	0.76816
Mouthfeel	0.14977	0.12132	0.11304	0.49587	0.64663
Off-flavour	0.44704	-0.00609	0.050145	0.48956	0.55382
Overal acceptability	0.048765	0.11156	0.000394	0.4394	0.72029
Acetic acid	-0.11148	-0.2313	0.29797	0.57647	0.15831
butanoic acid	0.39218	0.28682	0.086768	-0.52964	0.26405
Butanoic acid, 2 methyl	0.15453	-0.03164	0.28476	-0.4548	0.21872
Butanoic acid, 3 methyl	0.27003	0.31959	0.26808	-0.43038	0.33957
Hexanoic acid	-0.18791	0.17351	-0.03469	-0.07248	0.65603
Pentanoic acid	0.1816	-0.36111	0.28203	-0.26716	0.13068
1-octanol	-0.3608	0.11454	-0.04921	-8.53E-05	-0.09894
1-Octen-3-ol	-0.04438	0.1447	0.32056	-0.00223	0.51234
2,3 butanediol	0.073564	-0.13699	0.23833	-0.42684	0.17439
Phenylethyl alcohol	0.21684	0.3347	0.15187	0.04647	0.13096
2,4-Decadienal	0.28675	0.18051	-0.18228	0.39115	-0.29878
2-Decenal	0.15727	0.074344	0.24569	-0.34592	0.46897
2-Heptenal	0.067295	0.067392	0.20909	-0.41056	0.48541
2-Octenal	-0.05295	-0.31818	0.22129	-0.07871	0.13543
Cis-4,5-Epoxy-(E)-decenal	-0.1063	-0.12847	0.052728	-0.15225	0.098494

acid, 3-methyl butanoic acid and 2,4- decadienal. Whereas, off flavour attributes negatively correlated with 1-Octanol. Ofada rice taste was positively correlated with 2,3-butanediol.

The second principal components (PC2) showed that there exist a significant positive correlation aroma sensory attribute and aroma active compounds of 3 methyl butanoic acid, butanoic acid, and phenylethyl alcohol. This showed that aroma sensory acceptability of Ofada rice could be dependent on the three aroma active compounds responsible for rancid, cheese-like and floral flavour notes. The presence of this compounds is also positively correlated to the parboiling and drying temperatures. All the aforementioned were also negatively correlated to acetic acid, pentanoic acid and 2-Octenal which impart sour, fatty, green, nutty and herbaceous flavour notes.

The result of the third principal component showed that positive correlation exist with respect of drying temperature, acetic acid, mouthfeel, 2-methyl butanoic acid, butanoic acid, pentanoic acid, 3-Methyl butanoic acid, 2,3- butanediol, 2-decenal, 2-heptenal and 2-octenal. Steeping duration is negatively correlated to taste , aroma, mouthfeel, off-flavour and overall acceptability in the third principal component (PC4). However, steeping duration has a direct relationship with butanoic acid, 2-methyl butanoic, 3- methyl butanoic, pentanoic acid, 2,3- butanediol, 2- decenal and 2-heptenal. It was also observed that an inverse relationship between steeping duration and aroma active compounds of 2, 4- decadienal and acetic acid

CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Flavour compounds were identified in Ofada rice with consideration on the effect of variation in processing conditions of steeping duration, initial steeping temperature, parboiling temperature and drying temperature. Response surface methodology was used as a design tool to provide an adequate mixture of the processing conditions. Liquid Chromatography-Mass Spectrometry was used to determine the primary metabolites responsible for flavour variation in Ofada rice while GCToFMS was used for the detection of aroma compounds in Ofada rice processed with varying processing conditions.

Variation in the steeping duration and initial steeping temperature had a significant effect on the microbial community. Percentage occurrence of proteobacteria decreases as the initial paddy steeping temperature increases, while the percentage occurrence of firmicutes increases as the initial steeping temperature increases from 30^{o}C to 100^{o}C. Results of total phenolic content showed that a significant difference ($p<0.05$) was observed by the synergy of variation in the steeping duration, parboiling and drying temperature which implied a variation in the astringent and bitterness flavour from rice. Treatments with a steeping duration of one day had the highest TPC value of 62.66 mgGAE/100g followed by treatments with 3 d steeping which had its highest TPC at 56.30 mgGAE/100g while 5 d steeping treatments has its highest TPC value of 53.92 mgGAE/100g. An increase in steeping duration leads to a decrease in TPC, whereas the total flavonoid increases with an increase in fermentation duration.

Amino acids found at low percentages were cysteine, histidine, arginine, tryptophan, methionine and phenylalanine, while asparagine, glutamic acid, alanine, proline and threonine were found at relatively higher percentages in processed Ofada rice. Among all the amino acids detected by LCMS, there was nosignificant difference ($p>0.05$) in the percentage of glycine, threonine and phenylalanine. The average percentages of

detectable organic acids in Ofada rice were 2-aminobutyric acid (1.735), nicotinic acid (1.98), pantothenic acid (0.416), Malic acid (0.524), pyruvic acid (0.007), lactic acid (0.463), citric acid (6.664), succinic acid (0.236), fumaric acid (0.116), and orotic acid (0.001).

A total of 266 compounds were identified in Ofada rice due to variation in processing conditions (Steeping duration, Initial steeping temperature, parboiling temperature and drying temperature). These compounds were made up of 26 acids, 30 alcohols, 28 aldehydes, 50 hydrocarbons, 13 Nitrogenous compounds, 36 ketones, 33 esters, 15 heterocyclics, 12 phenols and 23 unclassified compounds. Organoletpically, Negatively impacting aroma compounds detected as the steeping duration, initial steeping temperature increases and with decreasing parboiling and drying temperature were hexanal, acetic acid, pentanoic acid, 1-pentanol, 1-heptanol, 1-octen-3-ol, (E)-2-nonenal, octenal, pentanal and hexanal. Whereas, aroma compounds that have a desirable impact on rice such as 2-methyl-Butanoic acid, phenylethyl alcohol, 3-Hydroxy-4,5-dimethyl-2(5*H*)- furanone, 2-heptanone, 3-octen-2-one were only detected among five days steeping treatment at reduced parboiling and drying temperature. As steeping duration increases, there was an increase in aroma compounds responsible for desirable and undesirable flavour attributes of Ofada rice.

Sensory assessment of Ofada rice by panelists showed that there exists a significant difference with respect to taste between the most and least liked at a 5 % level of significance. Panelists adjudged the highest likeness in taste to rice processed by paddy steeping of a day, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature while the least liked taste is from paddy rice steeped for 5 d, initial steeping temperature of 30^{o}C, parboiled at 80^{o}C and dried at 30^{o}C. Rice aroma most liked by panellists was from rice processed with one day steeping, 30^{o}C initial steeping temperature, 80^{o}C parboiling temperature and 50^{o}C drying temperature.

5.2 Recommendations

Having identified compounds found in Ofada rice with different processing conditions, the following were recommended

1. Undesirable flavour compounds are only present at five days steeping duration, as such Ofada paddy steeping should not exceed three days steeping if off flavours are to be eliminated

2. Steeping Ofada paddy at ambient temperature led to a delay in the commencement of fermentation due to the competitiveness of other bacteria with the fermenting bacteria. Therefore, an initial steeping temperature of 65^{o}C and above is recommended for paddy steeping so as to reduce steeping duration.

3. Intense hydrothermal processes led to a loss of water soluble flavour components and as such its severity should be minimal in order to retain flavour components that significantly influence Ofada rice acceptability.

4. Further research work into eliminating off-flavour from Ofada rice paddy steeped for longer days is a green area of research.This is important because some desireable and off-flavour compounds increase as the steeping duration increases.

5.3 Contributions to knowledge

This work was able to:

1) obtain a suitable extraction protocol to determine the primary metabolites in Ofada rice using LC-MS.

2. establish the variation that exists in the bacteria community in fermenting steeping water.

3. report the effect of different processing conditions (Initial steeping temperature, Steeping duration, parboiling temperature and drying temperature) on the flavour components of Ofada rice.

4. establish that the relish Ofada rice flavour is not a genetic characteristic (because 2AP was not detected) but rather can be attributed to processing conditions.

5. observed that off-flavour masked Ofada rice flavour as the steeping duration increases.

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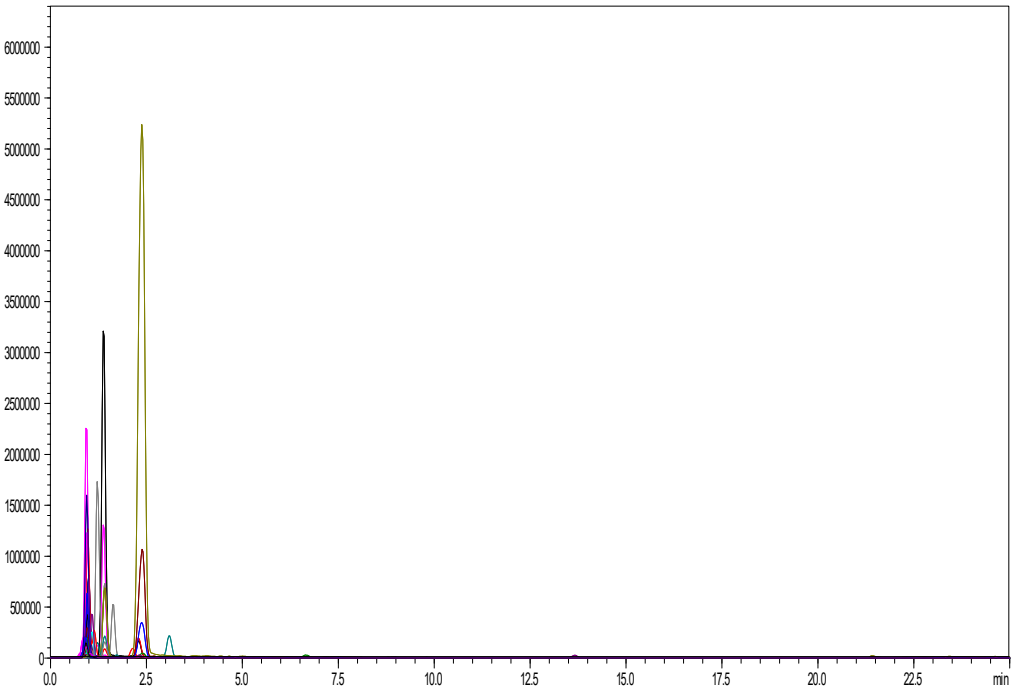
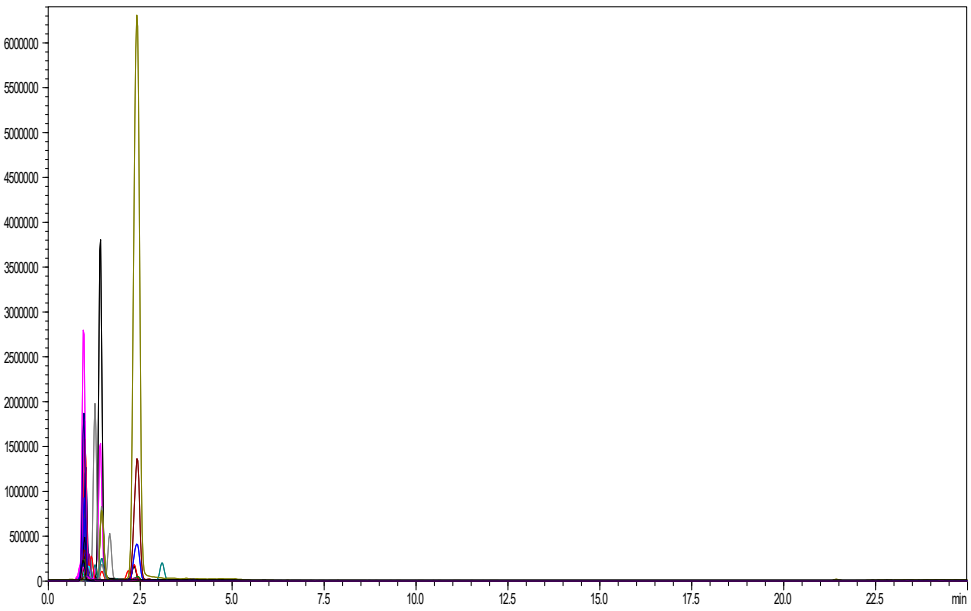
APPENDIX 1

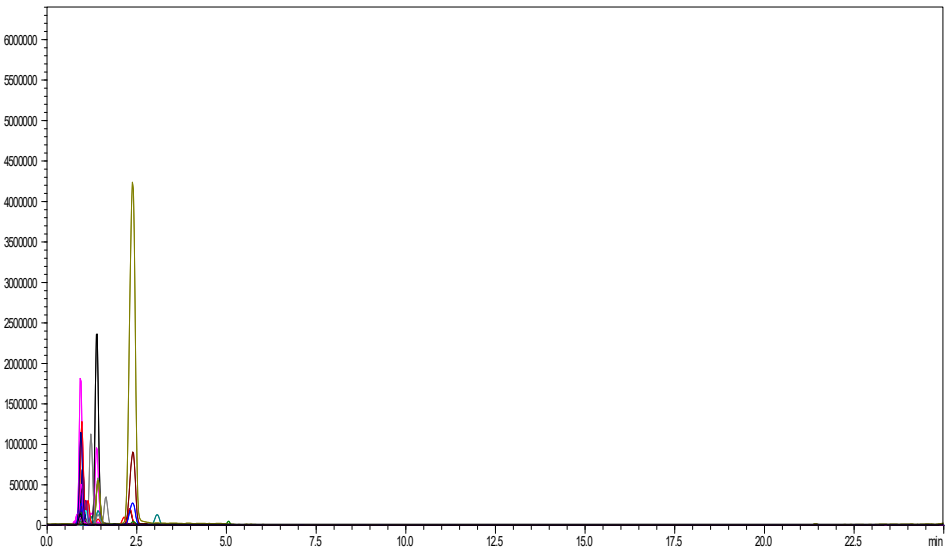
Chromatographs for primary metabolites from LCMS



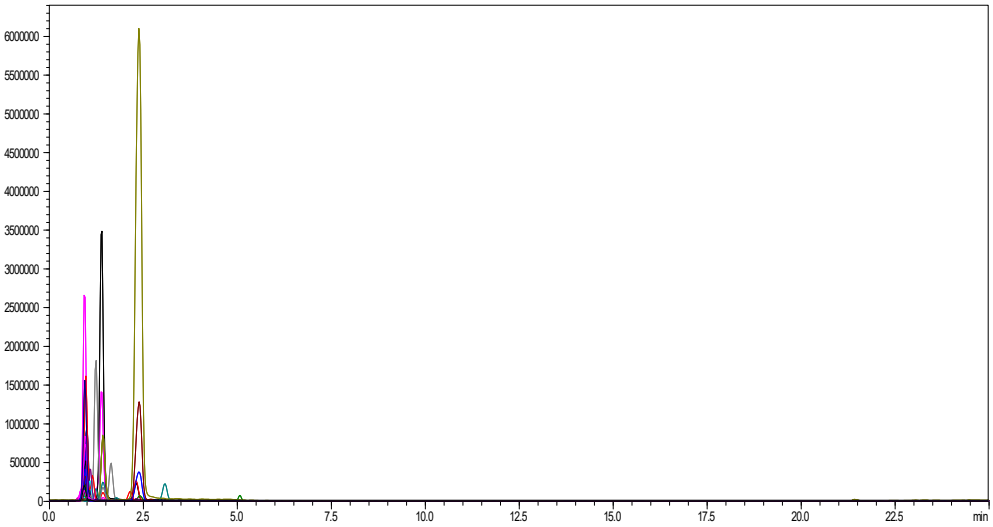
LCMS chromatograph of Sample treatment 1



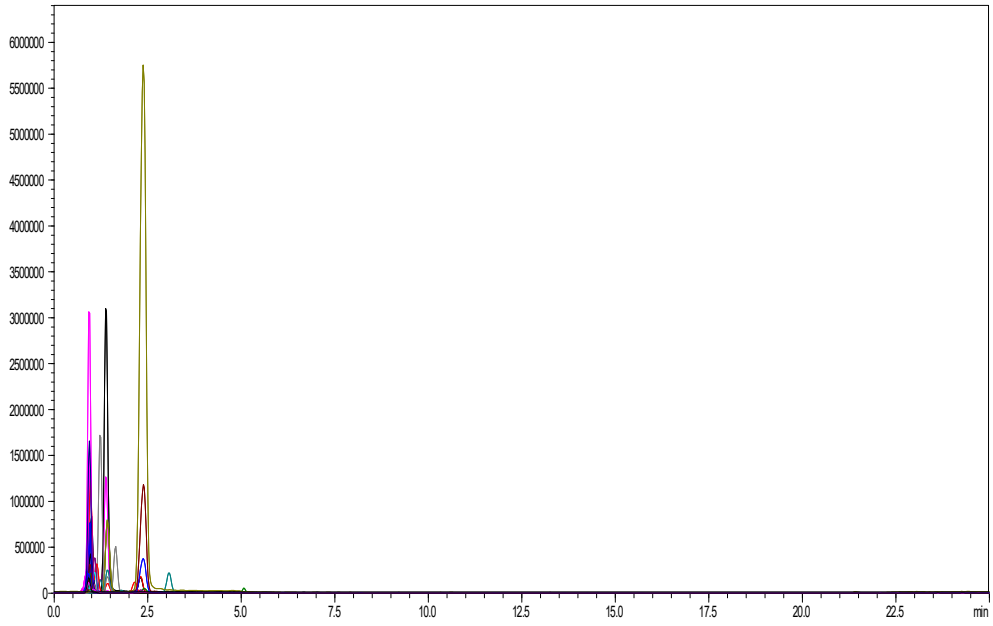
LCMS chromatograph of Sample treatment 2



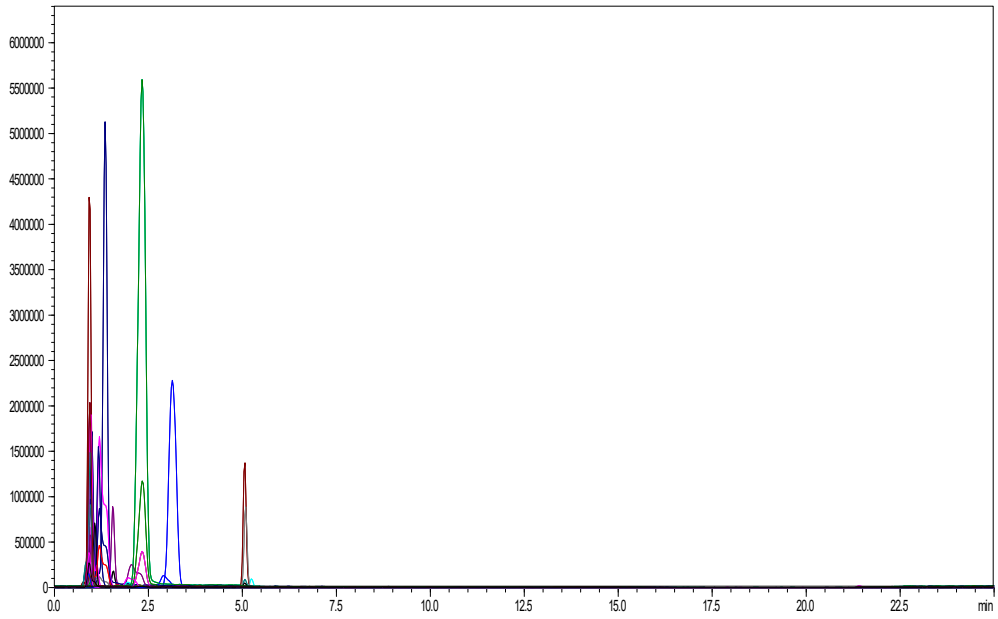
LCMS chromatograph of Sample treatment 3



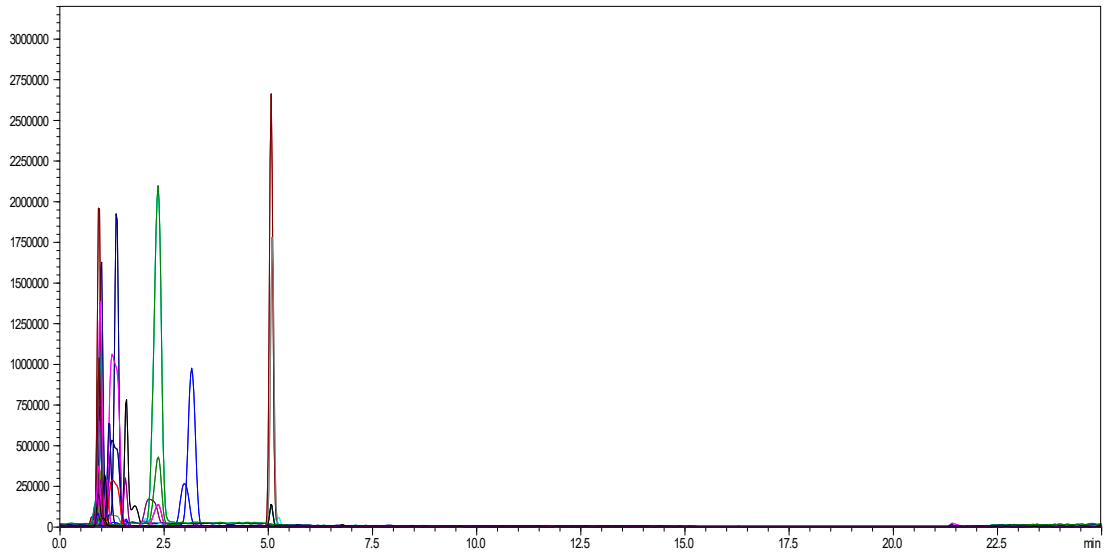
LCMS chromatograph of Sample treatment 4



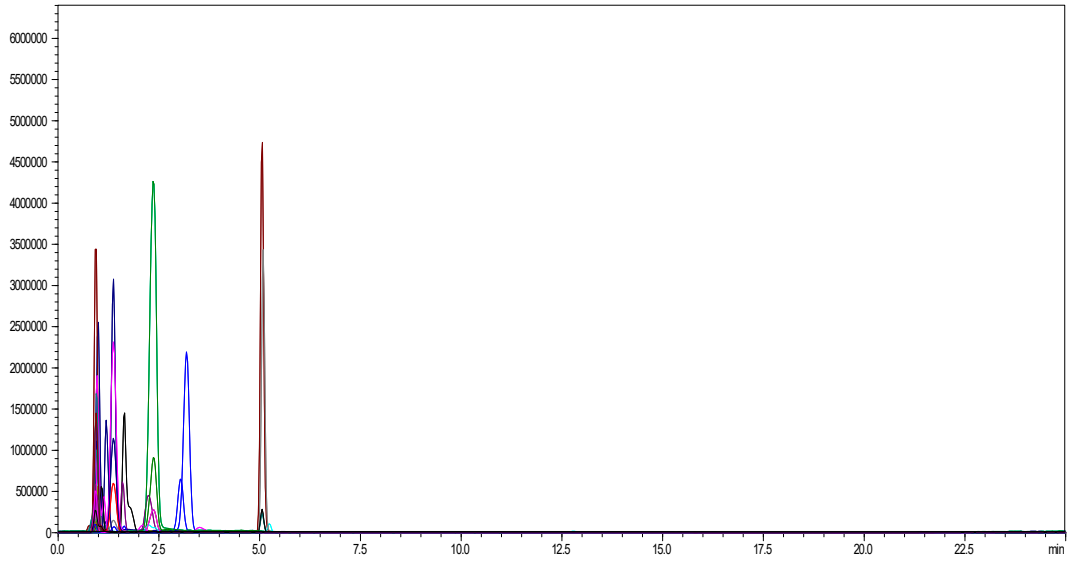
LCMS chromatograph of Sample treatment 5



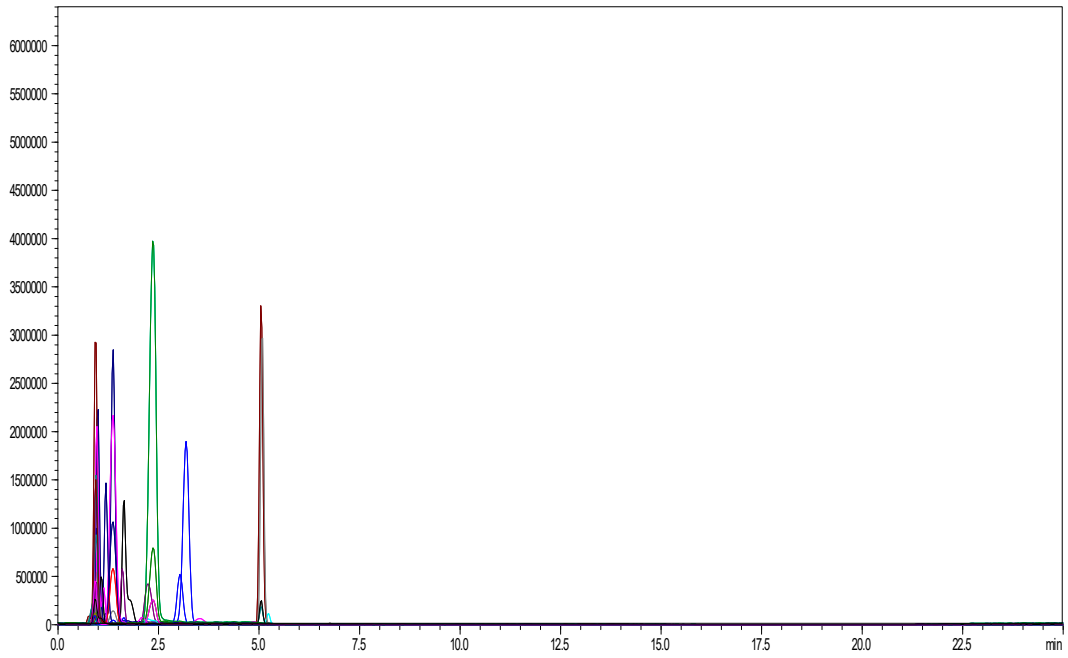
LCMS chromatograph of Sample treatment 6



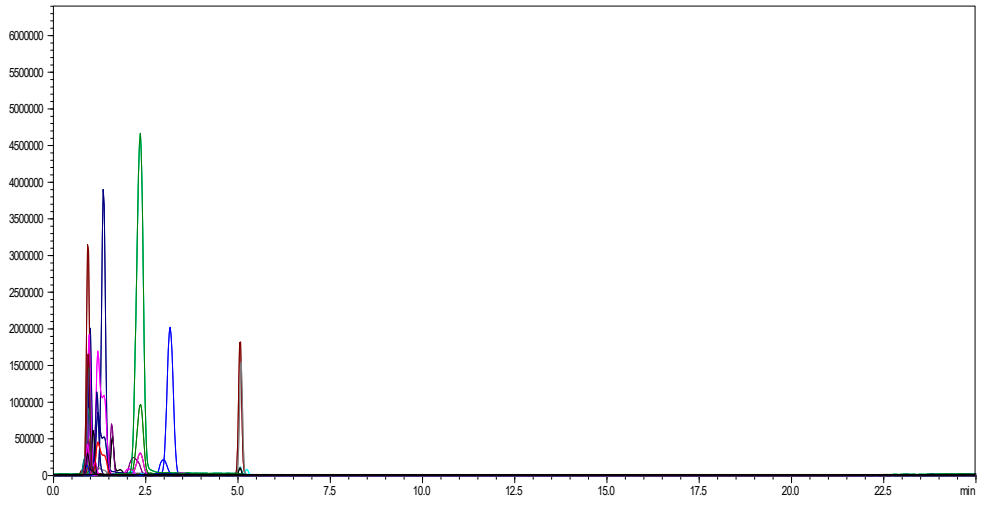
LCMS chromatograph of Sample treatment 7



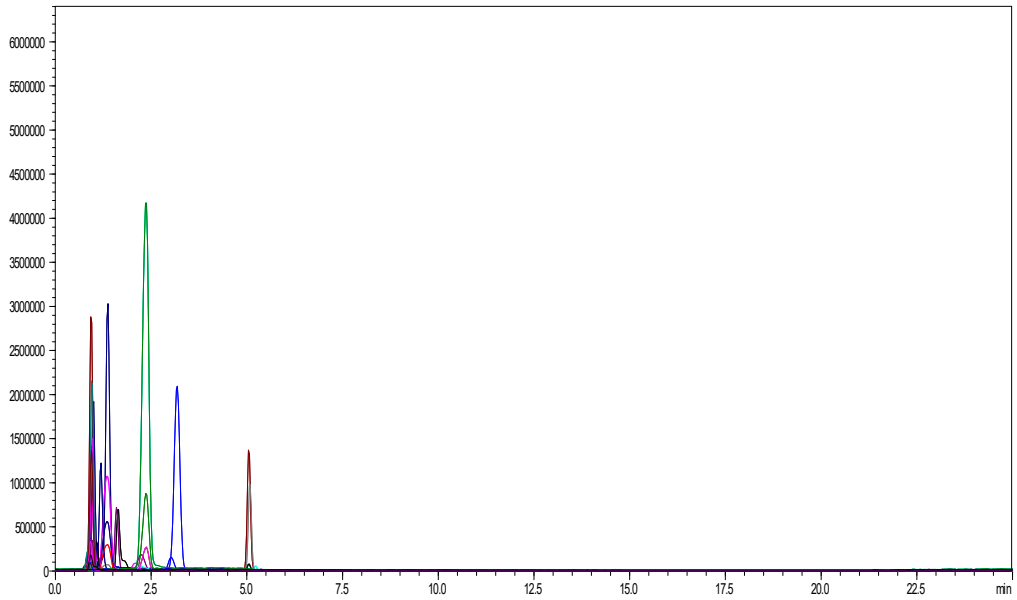
LCMS chromatograph of Sample treatment 8



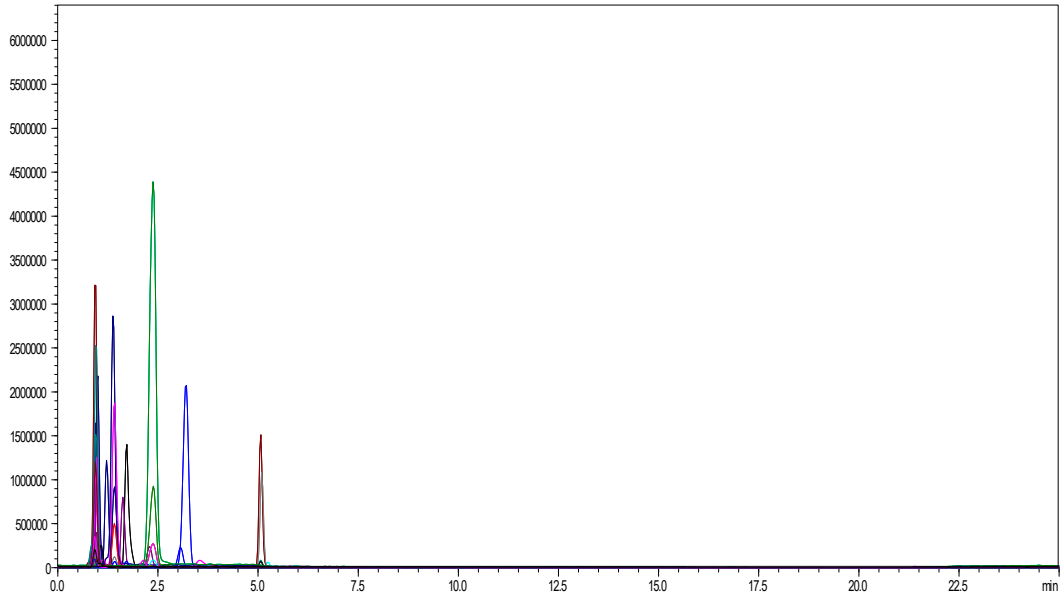
LCMS chromatograph of Sample treatment 9



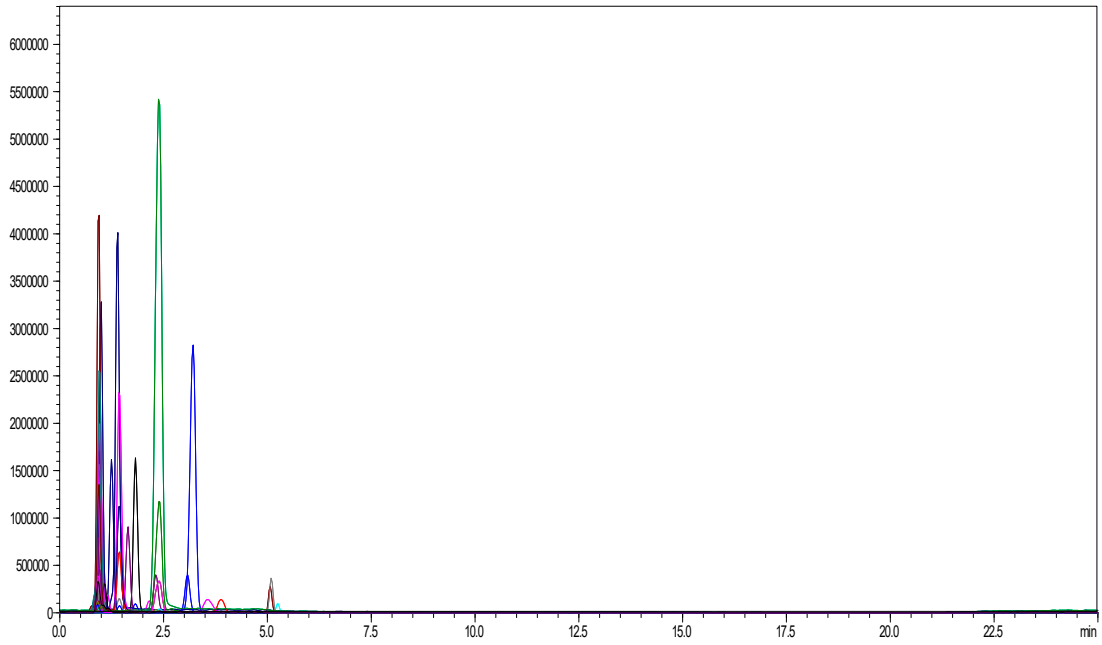
LCMS chromatograph of Sample treatment 10



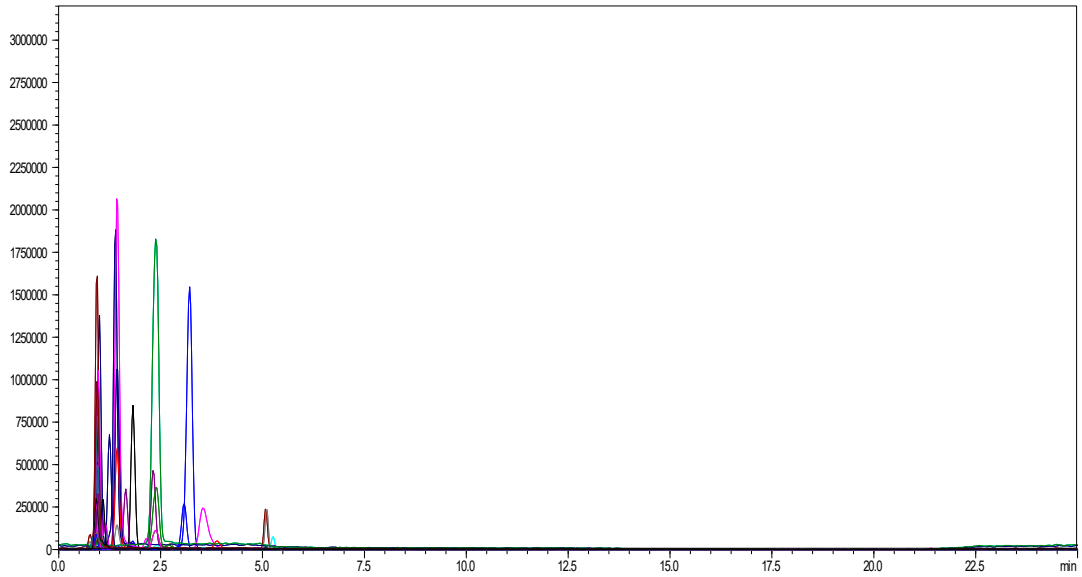
LCMS chromatograph of Sample treatment 11



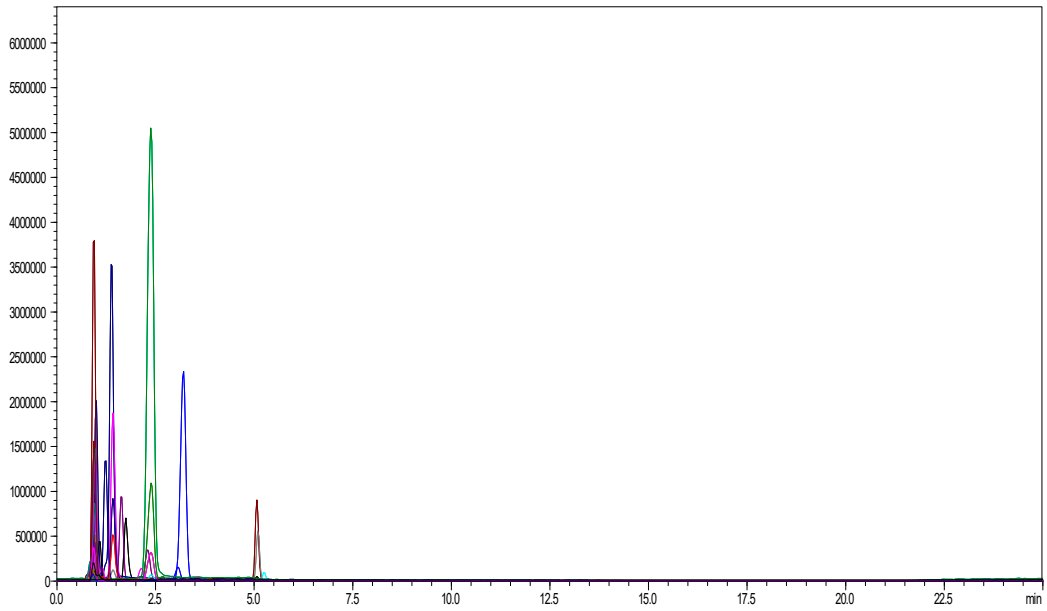
LCMS chromatograph of Sample treatment 12



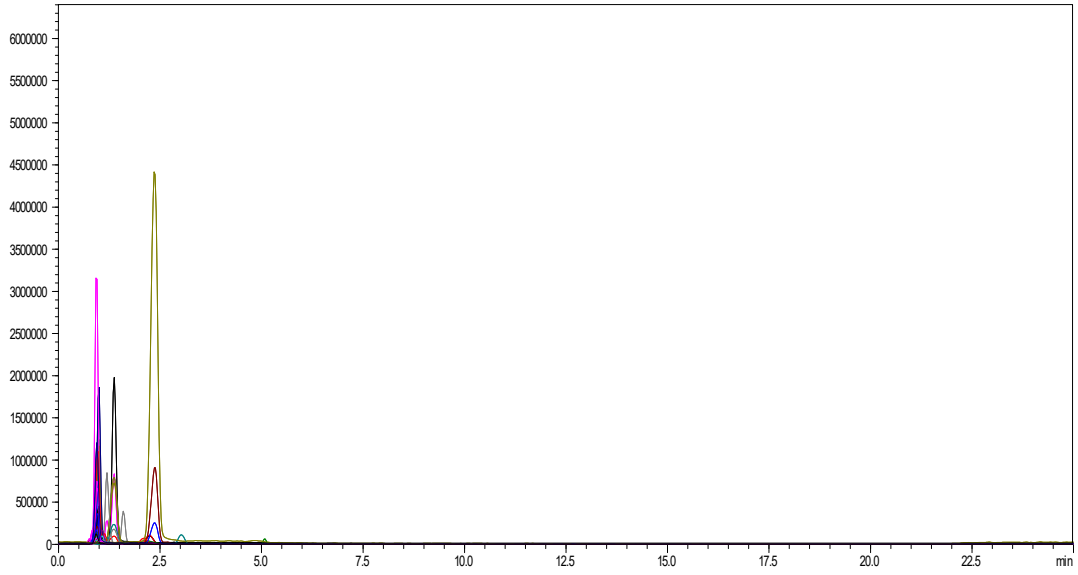
LCMS chromatograph of Sample treatment 13



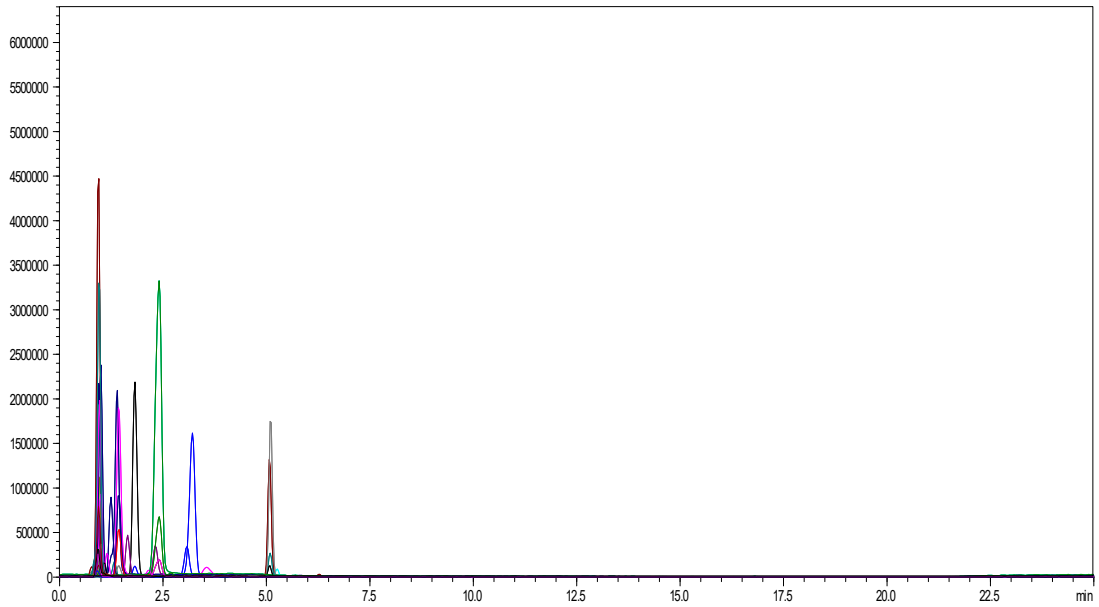
LCMS chromatograph of Sample treatment 14



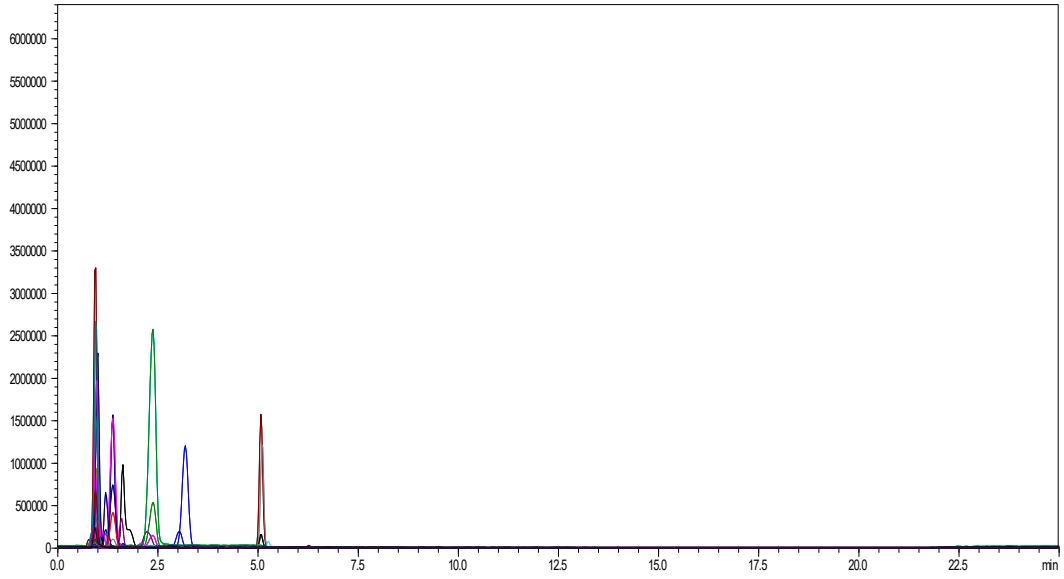
LCMS chromatograph of Sample treatment 15



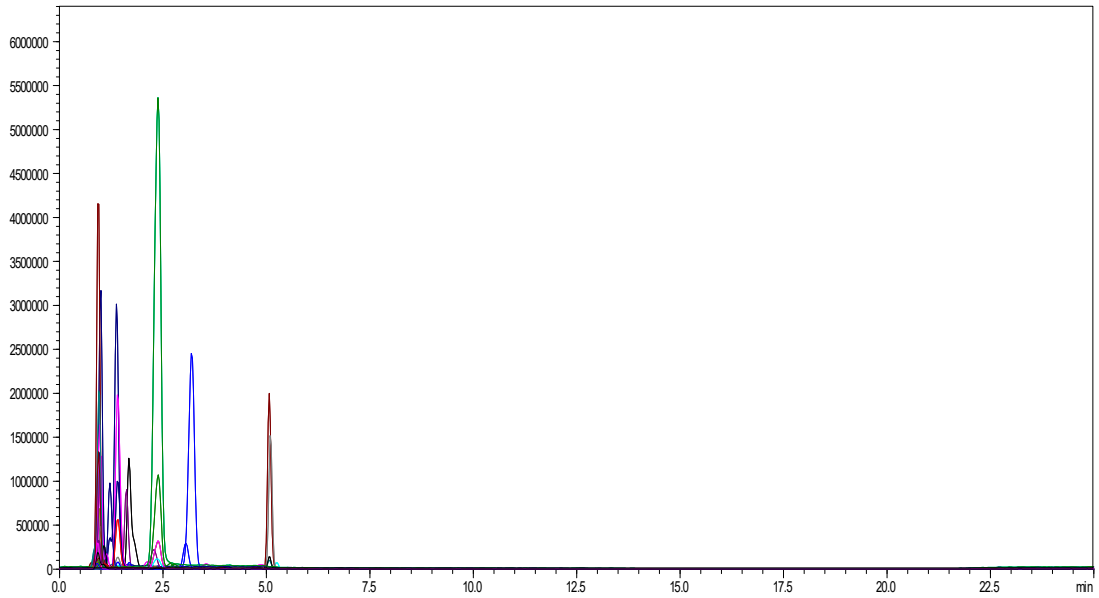
LCMS chromatograph of Sample treatment 16



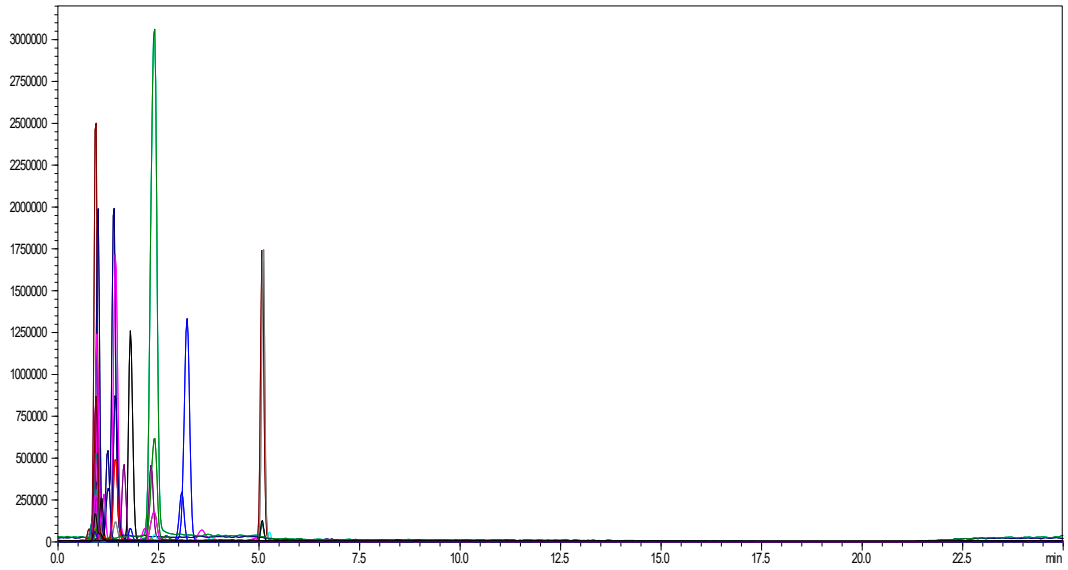
LCMS chromatograph of Sample treatment 17



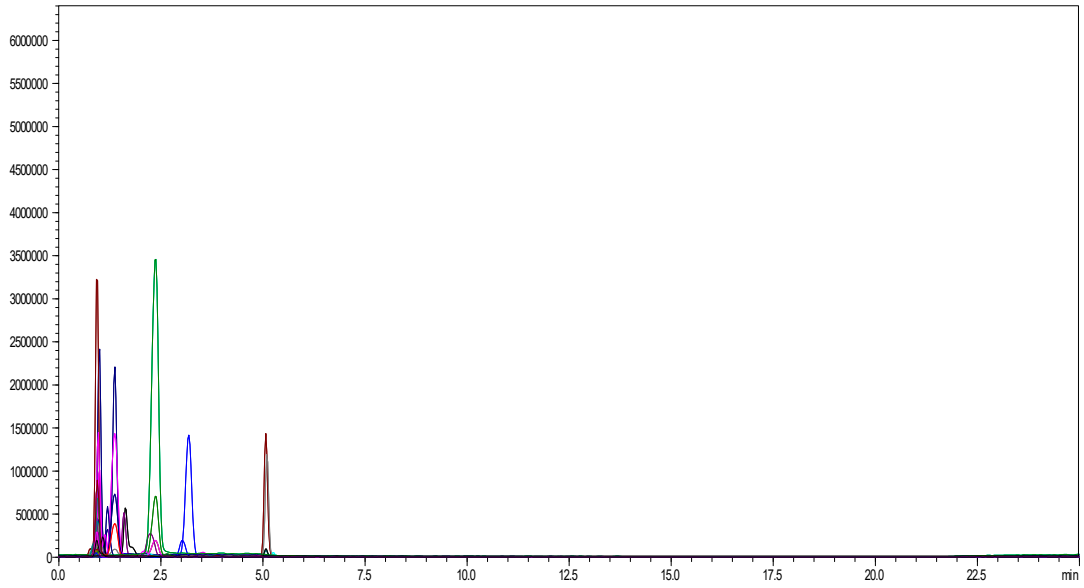
LCMS chromatograph of Sample treatment 18



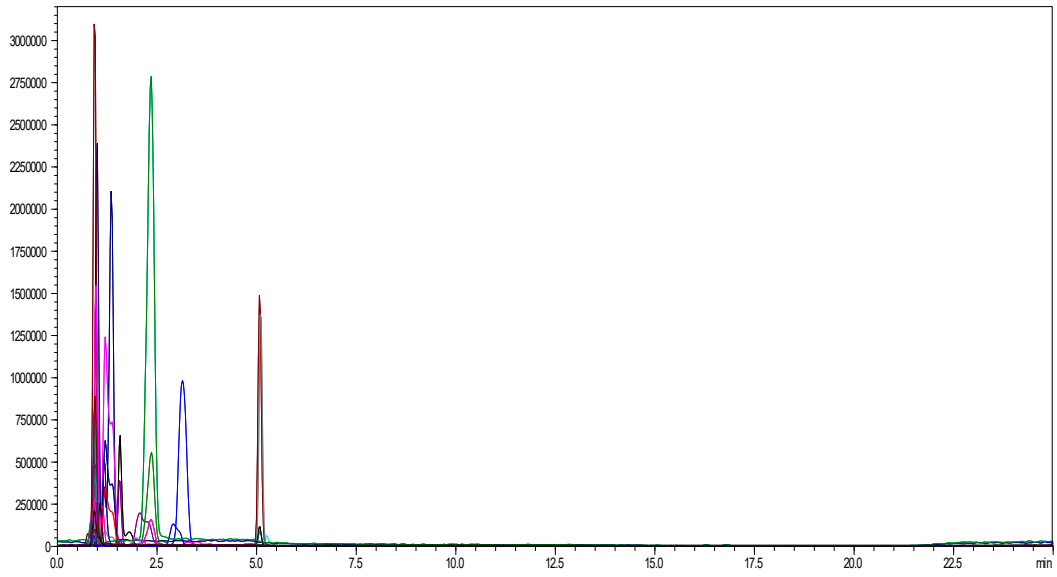
LCMS chromatograph of Sample treatment 19



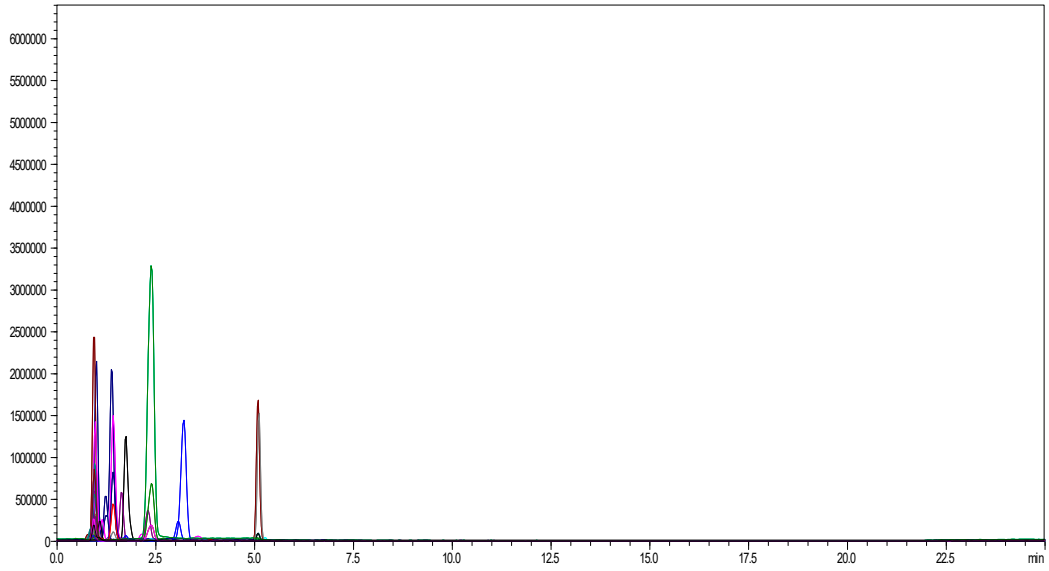
LCMS chromatograph of Sample treatment 20



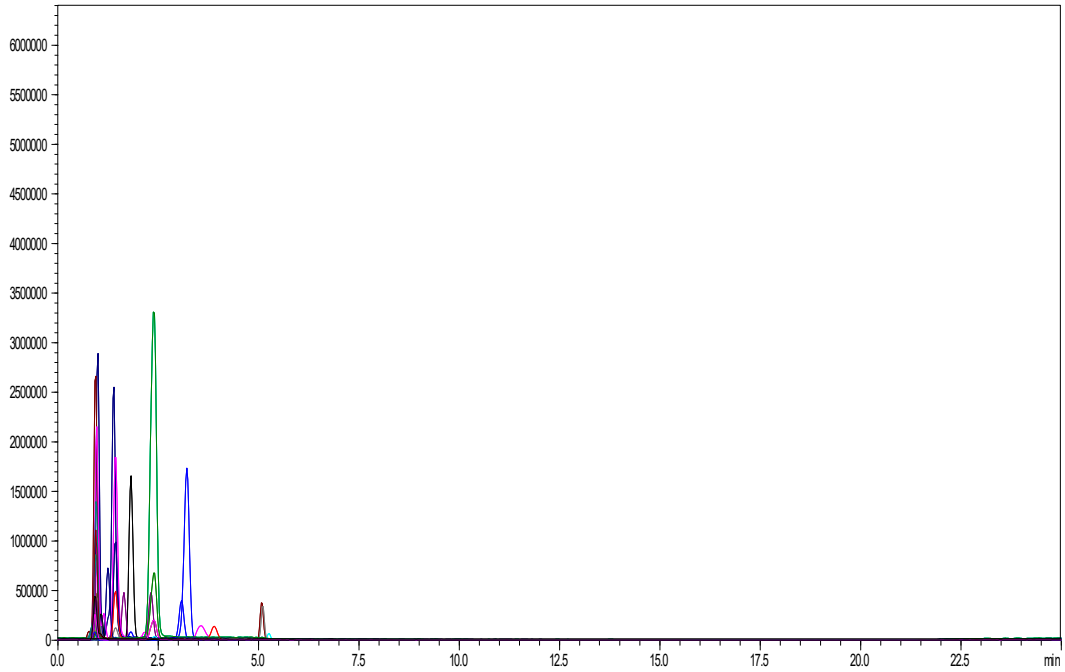
LCMS chromatograph of Sample treatment 21



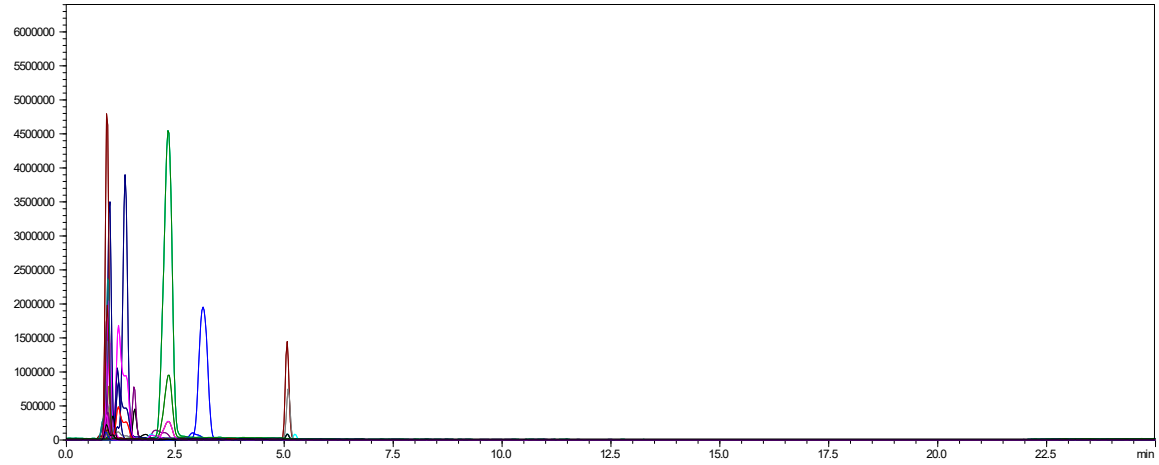
LCMS chromatograph of Sample treatment 22



LCMS chromatograph of Sample treatment 23



LCMS chromatograph of Sample treatment 24



LCMS chromatograph of Sample treatment 25

Appendix 2: Percentages of aroma compounds in Ofada rice

A: Percentages of acids in Ofada rice

	Serial Number of Compounds																
Trts	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
2	NDIS	0.012	NDIS	NDIS	8.650	NDIS	0.030	0.001	0.006	0.008	0.018	NDIS	0.029	1.001	0.148	NDIS	0.085
6	NDIS	NDIS	0.995	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.112	NDIS	0.901	NDIS	NDIS	NDIS	0.712	0.655
15	NDIS	0.016	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	0.011	NDIS	NDIS	3.038	0.021	NDIS	0.132	1.488	NDIS
24	0.106	NDIS	NDIS	NDIS	0.015	NDIS	0.053	0.001	0.004	NDIS	NDIS	NDIS	NDIS	4.617	0.083	NDIS	NDIS
8	0.013	NDIS	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.019	0.022	0.004
11	NDIS	NDIS	NDIS	NDIS	1.011	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	0.170	NDIS
20	NDIS	NDIS	NDIS	NDIS	3.283	NDIS	0.003	NDIS	NDIS	NDIS	20.933	0.022	NDIS	NDIS	0.009	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	1.335	NDIS	0.036	NDIS	0.001	NDIS	NDIS	9.053	NDIS	NDIS	0.025	6.743	NDIS
18	0.004	NDIS	NDIS	NDIS	NDIS	NDIS	0.039	NDIS	NDIS	NDIS	NDIS	4.940	NDIS	0.799	0.046	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	0.306	0.013	0.033	NDIS	0.257	0.838	NDIS	0.257	0.036	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	0.063	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.057	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.018	NDIS	NDIS
5	0.005	NDIS	NDIS	NDIS	0.055	NDIS	NDIS	NDIS	0.018	NDIS	NDIS	0.006	NDIS	0.003	0.034	1.190	0.005
9	NDIS	NDIS	8.378	0.004	0.041	NDIS	0.053	0.014	0.032	0.000	NDIS	NDIS	NDIS	0.001	0.036	NDIS	NDIS
10	0.005	NDIS	1.203	0.017	0.024	0.001	0.174	0.021	0.047	NDIS	21.706	6.723	NDIS	NDIS	1.863	NDIS	NDIS
17	NDIS	NDIS	0.190	NDIS	0.142	NDIS	2.500	0.375	0.469	NDIS	NDIS	NDIS	0.010	3.235	0.372	NDIS	NDIS
1	0.065	NDIS	0.227	NDIS	0.091	NDIS	1.480	0.184	0.371	0.006	NDIS	0.022	0.019	5.042	0.275	NDIS	NDIS
21	0.382	NDIS	NDIS	0.013	0.157	NDIS	1.346	0.154	0.296	NDIS	NDIS	NDIS	0.014	7.118	0.667	NDIS	NDIS
23	NDIS	NDIS	NDIS	0.015	NDIS	NDIS	4.009	0.226	0.404	0.001	0.071	NDIS	NDIS	NDIS	0.440	NDIS	NDIS
4	9.328	NDIS	NDIS	NDIS	NDIS	NDIS	5.383	NDIS	1.048	0.105	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	NDIS	0.018	NDIS	0.017	0.055	0.004	0.600	0.033	0.067	NDIS	0.005	NDIS	NDIS	NDIS	0.080	NDIS	0.017
3	0.022	0.075	0.341	NDIS	0.060	NDIS	4.413	0.206	0.296	NDIS	NDIS	0.052	NDIS	0.046	0.399	NDIS	NDIS
22	NDIS	0.008	NDIS	0.008	0.047	0.003	1.664	0.122	0.233	NDIS	NDIS	NDIS	NDIS	NDIS	0.591	NDIS	NDIS

Name of compounds with their aroma description on Table 4.7a; NDIS – Not detected in sample

A. Cont'd Percentage of acids in Ofada rice

	Serial number of compounds									
Trts	18	19	20	21	22	23	24	25	26	27
2	NDIS	1.393	0.132	0.023	NDIS	0.004	NDIS	0.080	NDIS	0.967
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.056	NDIS	NDIS	3.345
15	0.077	0.190	0.049	0.012	2.622	NDIS	0.011	0.095	0.001	0.101
24	NDIS	3.042	0.012	0.010	0.036	NDIS	NDIS	NDIS	NDIS	0.128
8	0.018	0.056	0.018	NDIS	0.109	NDIS	NDIS	0.011	0.076	0.114
11	0.001	10.569	0.000	0.000	15.444	NDIS	0.000	NDIS	NDIS	NDIS
20	NDIS	0.912	0.019	0.005	6.282	0.019	NDIS	NDIS	NDIS	NDIS
25	0.007	0.602	0.011	0.003	9.283	NDIS	NDIS	NDIS	NDIS	0.065
18	0.016	0.227	0.013	0.004	NDIS	NDIS	NDIS	0.005	0.017	0.154
13	13.201	5.231	NDIS	NDIS	17.562	NDIS	NDIS	0.007	0.009	4.370
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.000
12	NDIS	NDIS	0.039	0.007	0.001	NDIS	0.001	0.000	0.025	0.007
14	NDIS	0.199	NDIS	NDIS	0.294	0.013	NDIS	NDIS	NDIS	15.755
7	NDIS	0.163	NDIS	NDIS	0.371	NDIS	NDIS	NDIS	NDIS	NDIS
5	0.011	0.213	0.193	0.008	NDIS	NDIS	0.034	NDIS	0.001	0.060
9	2.325	14.587	0.006	0.006	2.659	0.000	NDIS	NDIS	NDIS	NDIS
10	0.006	7.003	0.014	0.017	5.604	NDIS	NDIS	0.122	NDIS	1.164
17	NDIS	NDIS	0.034	0.038	7.661	NDIS	NDIS	0.002	NDIS	2.710
1	0.007	1.947	NDIS	0.005	NDIS	NDIS	NDIS	NDIS	0.012	0.098
21	0.029	NDIS	0.038	0.015	7.214	NDIS	NDIS	NDIS	0.015	12.289
23	0.008	1.978	0.052	NDIS	NDIS	NDIS	0.086	0.048	NDIS	0.160
4	NDIS	NDIS	NDIS	NDIS	8.576	NDIS	NDIS	0.058	NDIS	0.859
19	0.004	2.921	0.011	0.018	0.140	NDIS	0.016	0.006	0.013	0.077
3	NDIS	NDIS	0.035	0.010	3.152	NDIS	NDIS	NDIS	NDIS	0.070
22	0.002	0.480	0.015	0.014	16.008	NDIS	0.028	NDIS	NDIS	0.003

Names of voilatile corresponding to the serial number with their aroma description is presented on Table 4.7; NDIS – Not detected in sample

B. Percentages of alcohols in Ofada rice

	Serial Number of Compounds														
Trts	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
2	0.001	NDIS	0.002	NDIS	0.014	NDIS	NDIS	NDIS	0.008	NDIS	0.109	NDIS	NDIS	NDIS	NDIS
6	NDIS	NDIS	0.383	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	0.057	NDIS	NDIS	NDIS	NDIS
24	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	0.005	0.053	NDIS	0.011	NDIS	NDIS	NDIS	NDIS
8	NDIS	NDIS	0.011	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.004
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.993	NDIS	0.005	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
20	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.078	0.024	NDIS	NDIS	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	0.000	NDIS	0.010	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
9	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	0.084	0.000	NDIS	NDIS	NDIS	NDIS	0.001
10	0.012	0.002	NDIS	0.004	0.006	NDIS	NDIS	NDIS	0.167	0.000	NDIS	0.033	0.002	NDIS	NDIS
17	NDIS	NDIS	NDIS	0.013	NDIS	0.007	NDIS	0.055	0.901	0.018	NDIS	NDIS	NDIS	NDIS	NDIS
1	NDIS	NDIS	NDIS	NDIS	0.031	NDIS	NDIS	NDIS	0.163	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
21	NDIS	NDIS	NDIS	0.010	0.008	0.008	NDIS	NDIS	0.167	NDIS	0.012	NDIS	0.009	NDIS	0.030
23	NDIS	0.006	NDIS	NDIS	0.006	NDIS	NDIS	NDIS	0.380	NDIS	0.020	23.056	NDIS	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	NDIS	NDIS	NDIS	NDIS	0.003	0.003	NDIS	NDIS	0.095	0.000	0.004	NDIS	0.003	NDIS	NDIS
3	0.022	NDIS	NDIS	NDIS	NDIS	NDIS	0.009	NDIS	0.650	NDIS	NDIS	NDIS	0.014	0.000	3.237
22	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	0.005	0.006	0.091	0.000	0.005	NDIS	0.002	NDIS	0.010

Name of compounds corresponding to their serial number and the aroma descriptions is presented on Table 4.8; NDIS – Not detected in sample

B. Cont'd. Percentage of alcohols in Ofada rice

	Serial Number of Compounds														
Trts	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
2	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	0.002	0.035	NDIS	NDIS	NDIS	0.071	NDIS	0.162	NDIS
6	NDIS	NDIS	NDIS	0.009	NDIS	NDIS	NDIS	NDIS	0.181	NDIS	NDIS	NDIS	0.126	0.192	NDIS
15	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	0.008	NDIS	NDIS	0.031	NDIS	0.034	NDIS
24	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	0.006	NDIS
8	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	NDIS	0.010	0.009
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.880	NDIS	NDIS	NDIS	NDIS	0.591	NDIS
20	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	2.623	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	1.240	NDIS	NDIS	NDIS	NDIS	0.374	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.061	0.058	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	49.437	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
9	NDIS	NDIS	NDIS	NDIS	0.002	0.000	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001
10	0.052	0.007	NDIS	NDIS	0.004	0.001	0.000	NDIS	NDIS	NDIS	7.314	NDIS	NDIS	NDIS	NDIS
17	NDIS	NDIS	NDIS	NDIS	NDIS	0.363	NDIS	NDIS	NDIS	0.937	9.553	NDIS	NDIS	NDIS	NDIS
1	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS
21	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.011	NDIS	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	0.004
23	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	9.822	0.210	NDIS	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS
3	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	1.065	0.001	NDIS	NDIS	NDIS	0.155
22	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	0.017	NDIS	NDIS	NDIS	6.923	0.213	NDIS	NDIS	3.235

Name of compounds corresponding to their serial number and the aroma description is presented in Table 4.8; NDIS -Notdetected in sample

C. Percentage of aldehydes in Ofada rice

	Serial Number of Compounds															
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
2	NDIS	NDIS	NDIS	0.395	0.233	0.007	NDIS	0.074	0.041	NDIS	NDIS	NDIS	0.016	0.029	NDIS	0.012
6	0.187	0.727	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	NDIS	NDIS	0.506	NDIS	NDIS	0.060	0.039	NDIS	0.030	NDIS	0.015	NDIS	NDIS	NDIS
24	NDIS	NDIS	NDIS	0.106	0.058	NDIS	NDIS	0.025	0.019	NDIS	NDIS	NDIS	NDIS	0.128	NDIS	0.000
8	0.009	NDIS	NDIS	0.042	0.025	NDIS	NDIS	0.005	0.015	NDIS	NDIS	NDIS	NDIS	NDIS	0.080	NDIS
11	3.080	NDIS	NDIS	0.032	NDIS	NDIS	NDIS	0.003	0.002	NDIS	NDIS	3.312	NDIS	NDIS	33.456	NDIS
20	NDIS	NDIS	NDIS	0.244	NDIS	NDIS	NDIS	0.031	0.013	NDIS	0.006	NDIS	NDIS	NDIS	24.308	NDIS
25	1.853	NDIS	NDIS	0.063	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	27.097	NDIS
18	0.517	NDIS	NDIS	0.033	0.021	NDIS	NDIS	0.021	0.005	NDIS	NDIS	3.184	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	8.701	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	44.774	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	37.720	NDIS
12	NDIS	NDIS	NDIS	0.103	0.061	NDIS	NDIS	0.009	0.012	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	0.039	0.024	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	4.061	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	41.160	NDIS
5	NDIS	NDIS	NDIS	0.245	NDIS	0.005	NDIS	0.055	0.034	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	0.012
9	NDIS	NDIS	NDIS	NDIS	0.013	NDIS	NDIS	0.020	0.006	NDIS	0.001	NDIS	0.001	NDIS	NDIS	NDIS
10	NDIS	NDIS	NDIS	NDIS	0.008	0.016	NDIS	0.015	0.015	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS
17	NDIS	NDIS	NDIS	0.264	0.124	NDIS	NDIS	0.115	0.094	NDIS	NDIS	NDN	NDIS	0.042	NDIS	NDIS
1	NDIS	NDIS	NDIS	NDIS	0.482	0.009	NDIS	0.045	0.054	NDIS	NDIS	3.509	NDIS	NDIS	NDIS	0.006
21	NDIS	NDIS	NDIS	0.382	0.175	NDIS	NDIS	0.157	0.106	0.011	NDIS	NDIS	0.003	0.085	NDIS	0.007
23	NDIS	NDIS	NDIS	0.236	0.151	0.003	0.036	0.159	0.086	0.017	NDIS	NDIS	NDIS	NDIS	NDIS	0.012
4	NDIS	NDIS	NDIS	0.273	0.170	0.004	NDIS	0.065	0.080	NDIS	NDIS	NDIS	NDIS	NDIS	0.059	NDIS
19	NDIS	NDIS	NDIS	1.159	2.280	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	3.802	NDIS	NDIS
3	NDIS	NDIS	0.002	NDIS	0.039	0.005	NDIS	0.055	0.035	0.004	0.002	NDIS	NDIS	NDIS	NDIS	0.004
22	NDIS	NDIS	NDIS	0.104	0.046	0.228	NDIS	0.127	0.030	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	0.016

Name of compounds corresponding to their serial number and the aroma description sis presented in Table 4.9; ND – Not detected

C. Cont'd. Percentage of aldehydes in Ofada rice

	Serial Number of Compounds											
TRT	17	18	19	20	21	22	23	24	25	26	27	28
2	NDIS	NDIS	NDIS	0.018	NDIS	NDIS	NDIS	0.011	0.206	0.121	NDIS	0.025
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS			NDIS	0.012
15	NDIS	0.007	NDIS	0.013	0.014	NDIS	NDIS	NDIS	0.173	0.093	NDIS	NDIS
24	NDIS	0.000	NDIS	0.001	0.001	NDIS	NDIS	NDIS	0.111	0.039	NDIS	NDIS
8	NDIS	NDIS	0.091	NDIS	NDIS	NDIS	NDIS	NDIS	0.018	0.011	NDIS	0.005
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.008	0.005	NDIS	NDIS
20	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	0.119	0.028	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.020	0.013	NDIS	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.048	0.016	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	0.005
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.023
12	NDIS	NDIS	NDIS	0.000	0.002	NDIS	NDIS	NDIS	0.073	NDIS	NDIS	NDIS
14	NDIS	NDIS	44.167	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.016	NDIS	0.001
9	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	0.001	NDIS	0.052	0.007	0.003	0.003
10	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	0.002	0.171	0.016	NDIS	NDIS
17	NDIS	0.142	NDIS	0.015	0.014	NDIS	NDIS	NDIS	2.453	0.136	NDIS	0.010
1	NDIS	0.091	NDIS	0.000	NDIS	NDIS	0.009	NDIS	0.625	0.199	NDIS	NDIS
21	0.003	0.006	NDIS	0.005	NDIS	NDIS	0.036	NDIS	0.637	0.075	NDIS	0.017
23	NDIS	NDIS	0.058	0.003	NDIS	NDIS	0.015	0.010	0.984	0.100	NDIS	0.007
4	NDIS	0.060	NDIS	0.014	NDIS	0.014	NDIS	0.013	1.791	0.081	NDIS	0.036
19	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003
3	0.004	0.003	NDIS	0.002	NDIS	NDIS	0.006	0.003	0.134	0.028	0.002	0.010
22	0.004	0.002	NDIS	0.001	NDIS	NDIS	0.027	0.003	0.016	0.028	NDIS	0.005

Name of compounds corresponding to their serial number aroma description is presented in Table 4.9; NDIS – Not detected in sample

D. Percentages of hydrocarbons in Ofada rice

	Serial Number of Compounds																
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
2	NDIS	0.001	NDIS	2.671	NDIS	NDIS	0.069	0.027	0.032	NDIS	0.013	NDIS	0.010	NDIS	0.050	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	NDIS	NDIS	0.024	NDIS	NDIS	0.023	NDIS	0.016	NDIS	NDIS	0.007	0.018	0.037	NDIS	0.002
24	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	0.008	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	0.021	NDIS	4.638
8	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	0.005	0.003	NDIS	NDIS
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.008	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	NDIS
20	NDIS	0.009	NDIS	NDIS	NDIS	NDIS	0.074	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.329	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	0.010	NDIS	NDIS	NDIS	NDIS	NDIS	0.172	NDIS	NDIS	0.068	0.003	NDIS	NDIS	NDIS	0.035	NDIS	NDIS
14	NDIS	0.013	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.015	0.136	NDIS
7	NDIS	0.008	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	0.034	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.036	NDIS	NDIS	0.002	NDIS	NDIS	0.000	0.026	0.039	NDIS	NDIS
9	NDIS	NDIS	NDIS	NDIS	0.001	0.001	0.002	0.002	0.009	0.004	0.031	NDIS	0.005	NDIS	0.016	NDIS	NDIS
10	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	0.006	0.001	0.008	0.005	0.014	0.000	NDIS	0.049	NDIS	NDIS
17	NDIS	NDIS	0.270	6.221	NDIS	NDIS	0.152	NDIS	NDIS	NDIS	0.008	NDIS	0.003	NDIS	0.105	NDIS	0.015
1	0.003	NDIS	NDIS	0.021	0.051	0.017	0.879	NDIS	0.007	0.003	0.013	NDIS	NDIS	NDIS	0.112	NDIS	NDIS
21	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	0.010	0.011	0.018	0.085	0.061	NDIS	0.149	NDIS	0.008
23	0.078	NDIS	NDIS	NDIS	0.040	NDIS	NDIS	NDIS	0.014	NDIS	0.031	0.002	0.006	NDIS	0.128	NDIS	0.006
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	0.079	0.003	0.001	0.001	0.057	NDIS	0.001
3	NDIS	NDIS	0.428	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.055	0.031	NDIS	0.028	NDIS	0.235	NDIS	NDIS
22	0.000	NDIS	NDIS	NDIS	0.014	0.001	0.023	NDIS	0.002	0.005	0.135	0.014	0.126	NDIS	NDIS	NDIS	0.002

Name of compouds correspoondng to their serial number is presented in Table 4.10a; NDIS – Not detected in sample

D. Cont'd. Percentages of hydrocarbons in Ofada rice

	Serial Number of Compounds															
TRT	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33
2	0.006	0.456	0.162	NDIS	0.022	NDIS	0.415	NDIS	0.041	NDIS	0.048	0.081	0.006	NDIS	0.013	1.668
6	25.557	0.129	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	0.375	NDIS	NDIS	NDIS	NDIS	0.336	NDIS	0.029	0.310	0.054	0.093	NDIS	NDIS	NDIS	0.676
24	0.009	NDIS	0.007	NDIS	NDIS	0.044	NDIS	NDIS	NDIS	NDIS	0.035	0.008	NDIS	NDIS	0.065	0.012
8	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	0.004
20	NDIS	NDIS	NDIS	NDIS	0.011	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.019
25	NDIS	NDIS	0.008	NDIS	NDIS	0.012	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	NDIS	NDIS	0.008	NDIS	0.001	NDIS	0.045	NDIS	NDIS	NDIS	0.015	NDIS	0.002	NDIS	NDIS	0.018
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	0.001	0.162	0.011	0.036	NDIS	NDIS	0.122	NDIS	0.013	NDIS	0.126	0.002	NDIS	NDIS	0.188	0.077
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	0.263	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	0.000	NDIS	0.009	NDIS	0.001	0.026	0.034	NDIS	NDIS	NDIS	NDIS	0.237	NDIS	NDIS	NDIS	0.004
9	NDIS	0.038	0.012	NDIS	0.051	0.003	0.007	NDIS	0.006	0.030	0.058	0.044	NDIS	0.006	NDIS	0.031
10	NDIS	0.078	0.029	NDIS	0.001	NDIS	0.042	0.011	NDIS	0.073	0.128	0.011	NDIS	0.010	NDIS	1.084
17	0.197	0.152	0.020	0.064	0.022	NDIS	0.055	NDIS	NDIS	0.433	0.598	NDIS	NDIS	NDIS	NDIS	0.115
1	NDIS	0.176	NDIS	NDIS	NDIS	NDIS	0.324	0.033	NDIS	0.354	0.221	NDIS	NDIS	NDIS	NDIS	0.321
21	NDIS	0.061	0.013	0.002	0.012	NDIS	NDIS	NDIS	NDIS	0.024	0.151	NDIS	NDIS	NDIS	NDIS	0.051
23	0.011	NDIS	0.024	NDIS	0.018	NDIS	0.019	NDIS	NDIS	NDIS	0.123	0.023	NDIS	NDIS	NDIS	0.049
4	24.027	0.197	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	0.001	0.005	0.016	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	0.009	0.007	0.097	0.015	0.001	NDIS	0.004
3	NDIS	0.011	0.025	0.055	0.103	NDIS	0.042	0.206	NDIS	NDIS	0.201	NDIS	NDIS	NDIS	NDIS	0.026
22	0.006	0.004	0.011	NDIS	0.005	NDIS	NDIS	NDIS	NDIS	0.076	0.007	0.050	2.792	0.002	NDIS	0.042

Name of compoundss corresponding to their serial number is presnerted in Table 4.10b; NDIS – Not detected in sample

D. Cont'd. Percentage of hydrocarbons in Ofada rice

	Serial Number of Compounds																
TRT	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50
2	1.053	NDIS	2.127	0.533	0.241	0.059	2.849	NDIS	0.018	NDIS	NDIS	0.190	0.242	NDIS	0.208	NDIS	0.503
6	NDIS	NDIS	4.063	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	0.271	NDIS	0.694	0.006	0.170	NDIS	0.898	NDIS	NDIS	NDIS	NDIS	NDIS	0.082	NDIS	0.118	NDIS	0.040
24	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.052	NDIS	NDIS	NDIS	NDIS	NDIS	0.019	NDIS	0.001	NDIS	0.008
8	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS	10.514	0.003	0.003	NDIS	NDIS	0.002	NDIS	0.004
20	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.007
18	NDIS	NDIS	0.010	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.028	0.013	0.004	NDIS	NDIS	0.016	0.009
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	5.510	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.030	NDIS	NDIS	NDIS	NDIS	0.012	NDIS
12	0.047	0.009	0.158	0.048	0.008	NDIS	0.662	NDIS	NDIS	NDIS	NDIS	0.228	0.048	NDIS	0.133	NDIS	0.028
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.033	NDIS	NDIS	NDIS	NDIS	0.007	NDIS
7	NDIS	NDIS	NDIS	NDIS	0.033	NDIS	NDIS	NDIS	NDIS	NDIS	0.018	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	0.057	0.003	0.021	NDIS	NDIS	NDIS	0.013	NDIS	0.017	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	0.011	NDIS
9	NDIS	0.000	NDIS	0.014	0.008	NDIS	0.094	0.008	0.000	NDIS	NDIS	NDIS	0.008	0.003	0.039	0.003	0.016
10	0.179	NDIS	0.010	0.041	0.009	NDIS	0.276	NDIS	NDIS	0.879	NDIS	NDIS	0.010	0.026	0.110	NDIS	NDIS
17	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	0.130	0.015	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.058	NDIS	0.014
1	0.199	NDIS	0.053	NDIS	0.349	NDIS	0.052	NDIS	NDIS	5.989	NDIS	0.001	0.073	NDIS	0.172	NDIS	0.087
21	NDIS	NDIS	0.037	NDIS	NDIS	NDIS	0.077	NDIS	NDIS	3.847	NDIS	NDIS	0.014	0.018	0.015	NDIS	0.071
23	NDIS	NDIS	0.023	NDIS	0.551	NDIS	0.090	NDIS	NDIS	0.111	NDIS	NDIS	NDIS	NDIS	0.027	NDIS	0.079
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	0.020	0.011	0.007	0.005	NDIS	0.001	0.010	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	0.018	0.003	NDIS	0.018
3	0.047	NDIS	0.072	NDIS	NDIS	NDIS	0.084	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.061	0.064	NDIS	0.014
22	NDIS	0.005	0.058	NDIS	0.014	0.000	0.039	NDIS	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	0.012	NDIS	0.016

Name of compoundscorresponding to their serial number is presented in Table 4.10c; NDIS – Not detected in sample

E. Percentage of Nitrogenous compounds in Ofada rice

	Serial Number of Compounds												
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13
2	1.507	NDIS	NDIS	NDIS	NDIS	NDIS	0.064	NDIS	0.042	NDIS	0.305	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	NDIS	NDIS	NDIS	0.041	0.054	NDIS	NDIS	0.005	NDIS	NDIS	NDIS
24	NDIS	NDIS	NDIS	NDIS	NDIS	0.011	0.054	NDIS	0.005	0.010	NDIS	NDIS	NDIS
8	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS
20	NDIS	NDIS	NDIS	0.020	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
25	NDIS	NDIS	NDIS	0.015	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	0.006	0.000	0.011	0.001	NDIS	NDIS	0.008	NDIS	0.009	NDIS	NDIS	NDIS	0.000
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	3.244
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.698
12	NDIS	NDIS	NDIS	NDIS	NDIS	0.010	NDIS	NDIS	0.009	NDIS	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	2.720
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	0.009	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	0.009
9	NDIS	0.053	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	0.001	0.005	NDIS	NDIS	NDIS
10	NDIS	0.390	0.030	NDIS	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	0.001	NDIS	0.000
17	NDIS	0.002	NDIS	0.057	0.525	NDIS	0.106	0.012	0.018	NDIS	NDIS	NDIS	NDIS
1	NDIS	0.001	NDIS	NDIS	NDIS	0.011	0.069	0.019	0.015	NDIS	NDIS	0.001	NDIS
21	NDIS	NDIS	NDIS	0.023	NDIS	NDIS	NDIS	0.013	0.154	NDIS	NDIS	0.109	NDIS
23	NDIS	1.746	NDIS	0.022	0.004	NDIS	NDIS	0.011	NDIS	NDIS	NDIS	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	0.139	3.702	NDIS
3	NDIS	NDIS	NDIS	0.026	NDIS	NDIS	0.008	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
22	NDIS	0.004	0.004	0.018	0.002	NDIS	NDIS	0.004	0.004	NDIS	0.005	0.070	0.006

Name of compounds corresponding to their serial number is presented in Table 11; NDIS – Not detected in sample

F. Percentages of ketones in Ofada rice

	Serial Number of Compounds																
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
2	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.020	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.215	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS
15	NDIS	NDIS	NDIS	0.030	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
24	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.012	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006
8	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.725	NDIS
20	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	9.382	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	1.419	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.013	0.160	NDIS	NDIS	NDIS	0.085	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	1.609	NDIS
9	0.001	0.002	NDIS	0.020	NDIS	0.001	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
10	NDIS	0.007	NDIS	NDIS	0.003	NDIS	0.023	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
17	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
1	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.009	NDIS	NDIS	NDIS	NDIS	NDIS	0.309	NDIS	NDIS	NDIS
21	0.009	NDIS	0.010	NDIS	NDIS	NDIS	NDIS	0.017	0.020	NDIS	NDIS	NDIS	NDIS	NDIS	0.013	NDIS	NDIS
23	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	NDIS	0.026	0.023	NDIS	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	0.000	NDIS	0.002	0.002	0.002	0.001	NDIS	0.000	NDIS	NDIS	NDIS	0.021	0.042	0.016	NDIS	NDIS	NDIS
3	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
22	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	NDIS	0.077	NDIS	NDIS	0.022	NDIS	NDIS	0.213	0.002	NDIS	0.012

Name of compounds correspondiong to therir seraial number is presented in Table 4.12a; NDIS – Not detected in sample

F. Cont'd. Percentages of ketones in Ofada rice

	Serial Number of Compounds																		
TRT	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
2	NDIS	0.031	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS	0.111	NDIS	0.020	NDIS	0.015	NDIS	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS	0.171	NDIS
15	NDIS	0.026	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NONE4	NDIS	0.170	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS
24	0.002	NDIS	NDIS	NDIS	0.002	NDIS	0.000	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS
8	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	2.914	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
20	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	1.118	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	1.552	NDIS
25	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.152	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	1.355	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	8.931	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	0.026	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.070	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.103	NDIS	0.001	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
9	0.001	0.004	NDIS	NDIS	0.093	NDIS	NDIS	NDIS	NONE0	NDIS	0.001	NDIS	NDIS	0.001	NDIS	0.000	NDIS	NDIS	NDIS
10	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	0.050	NDIS	0.005	0.003	NDIS	NDIS
17	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
1	0.016	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NONE8	NDIS	NDIS	NDIS
21	NDIS	0.014	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NONE9	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003
23	NDIS	NDIS	0.001	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	0.004	NDIS	NONE4	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	0.001	0.012	0.001	NDIS	NDIS	0.000	NDIS	0.011	NDIS	NDIS	0.002	0.067	0.002	NDIS	0.001	0.006	0.002	0.002	0.001
3	NDIS	0.021	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.206	NDIS	NDIS	0.006	NDIS	NDIS	NDIS	NDIS
22	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	0.003	NDIS

Name of compounds correspondiong to therir seraial number is presented in Table 4.12; NDIS – Not detected in sample

G. Percentage of heterocyclics in Ofada rice

	Serial Number of Compounds														
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
2	0.015	0.036	0.018	NDIS	NDIS	0.007	0.040	NDIS	NDIS	0.383	NDIS	NDIS	NDIS	NDIS	NDIS
6	NDIS	0.761	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.029	3.244	NDIS	NDIS	NDIS	NDIS	NDIS
15	0.025	0.064	0.014	NDIS	NDIS	0.017	NDIS	NDIS	NDIS	0.377	NDIS	0.045	NDIS	0.039	NDIS
24	NDIS	0.008	0.005	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	0.026	NDIS	NDIS	NDIS
8	NDIS	0.041	0.003	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS	NDIS
11	NDIS	6.272	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	0.000	NDIS	NDIS
20	NDIS	7.042	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS
25	NDIS	3.020	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS
18	NDIS	8.110	NDIS	NDIS	NDIS	2.125	NDIS	NDIS	NDIS	NDIS	NDIS	0.046	0.002	NDIS	NDIS
13	NDIS	13.100	NDIS	NDIS	NDIS	2.548	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	5.856	NDIS	NDIS	NDIS	8.750	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	0.016	NDIS	0.001	NDIS	NDIS	0.017	NDIS	NDIS	0.002	NDIS	NDIS
14	NDIS	13.557	NDIS	NDIS	NDIS	8.143	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	8.209	NDIS	NDIS	NDIS	7.676	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	NDIS
9	0.001	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	0.094	0.003	0.001	0.013	0.001	NDIS	0.001
10	0.002	NDIS	NDIS	0.029	NDIS	NDIS	NDIS	NDIS	0.027	NDIS	NDIS	0.054	0.000	NDIS	0.029
17	0.010	NDIS	NDIS	0.014	NDIS	NDIS	0.372	NDIS	NDIS	NDIS	0.014	NDIS	0.000	0.082	NDIS
1	0.027	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.072	NDIS	0.244	0.000	NDIS	NDIS
21	0.030	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	0.280	NDIS	0.045	NDIS
23	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.410	NDIS	NDIS	NDIS	0.019	NDIS	NDIS	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
19	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.399	0.009	0.006	0.047	0.000	NDIS	0.002
3	0.051	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS
22	0.010	0.004	NDIS	NDIS	0.051	NDIS	NDIS	NDIS	NDIS	0.051	0.017	0.063	0.001	NDIS	NDIS

Name of compounds corresponding to their serial number and aroma description is presented in Table 4.13; NDIS – Not detected in sample

H Percentage of esters in Ofada rice

	Serial Number of Compounds																
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
2	NDIS	NDIS	NDIS	NDIS	NDIS	0.067	0.011	0.005	NDIS	NDIS	NDIS	NDIS	0.119	0.000	NDIS	0.007	NDIS
6	0.194	NDIS	NDIS	NDIS	0.376	0.751	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	NDIS	NDIS	0.013	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	0.008	NDIS	0.011	NDIS	NDIS
24	NDIS	NDIS	NDIS	NDIS	NDIS	0.025	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	0.007	NDIS	0.031	NDIS	NDIS
8	NDIS	NDIS	0.032	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	0.001	NDIS	0.450	NDIS	NDIS	0.046	NDIS	0.029	0.066	NDIS	NDIS	NDIS	0.103	NDIS	NDIS	NDIS	NDIS
20	NDIS	NDIS	4.13	NDIS	NDIS	0.044	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.033	NDIS	NDIS	NDIS	NDIS
25	NDIS	NDIS	NDIS	NDIS	21.861	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	0.001	NDIS	0.130	NDIS	NDIS	0.031	0.008	NDIS	0.016	0.107	0.005	0.001	0.028	NDIS	0.002	NDIS	NDIS
13	0.002	NDIS	2.632	NDIS	0.823	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	NDIS	NDIS	NDIS	NDIS	0.027	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	NDIS	0.029	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.013	NDIS	0.155
14	NDIS	NDIS	0.037	NDIS	0.01	0.155	NDIS	NDIS	0.017	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
9	NDIS	NDIS	0.172	0.033	0.013	0.464	NDIS	NDIS	0.120	0.043	NDIS	NDIS	0.074	0.069	0.003	0.002	NDIS
10	NDIS	NDIS	0.008	NDIS	0.074	0.08	NDIS	NDIS	0.117	NDIS	NDIS	NDIS	0.200	NDIS	NDIS	0.022	0.026
17	NDIS	NDIS	0.263	NDIS	0.202	NDIS	NDIS	NDIS	NDIS	7.780	NDIS	NDIS	NDIS	NDIS	0.368	0.136	0.082
1	NDIS	NDIS	0.974	NDIS	NDIS	0.049	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	NDIS	NDIS
21	NDIS	NDIS	0.226	NDIS	0.082	NDIS	NDIS	NDIS	1.193	NDIS	NDIS	NDIS	0.033	0.002	0.094	NDIS	NDIS
23	NDIS	0.046	NDIS	NDIS	NDIS	NDIS	NDIS	0.051	0.389	NDIS	NDIS	NDIS	0.345	0.045	NDIS	NDIS	NDIS
4	0.006	0.010	0.140	0.003	NDIS	0.127	0.004	NDIS	NDIS	NDIS	0.004	0.011	0.025	NDIS	NDIS	5.562	NDIS
19	0.040	0.024	0.304	0.000	0.338	0.051	NDIS	NDIS	NDIS	NDIS	NDIS	0.012	0.072	0.005	0.003	NDIS	NDIS
3	0.000	0.014	0.455	0.018	0.182	0.058	0.104	NDIS	0.026	NDIS	0.117	0.002	0.057	0.001	0.080	0.287	0.053
22	0.022	NDIS	6.315	0.036	0.041	0.171	0.030	NDIS	0.062	NDIS	NDIS	0.018	0.061	NDIS	0.012	NDIS	NDIS

Name of esters corresponding to their serial number is presented in Table 4.14; NDIS – Not detected in sample

H. Cont'd. Percentage of esters in Ofada rice

	Serial Number of Compounds															
TRT	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33
2	0.052	0.121	0.065	NDIS	0.027	0.029	0.016	NDIS	0.869	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	0.713	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.024	NDIS	NDIS	NDIS
15	NDIS	NDIS	0.091	NDIS	0.012	NDIS	NDIS	NDIS	0.011	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	NDIS
24	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
8	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	0.007	NDIS	NDIS	NDIS	NDIS	NDIS	0.117	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
20	NDIS	0.004	0.010	NDIS	NDIS	NDIS	NDIS	NDIS	0.022	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
25	NDIS	0.031	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	NDIS
18	0.015	NDIS	0.037	NDIS	0.014	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.025	0.043	0.012	0.008	0.006
13	NDIS	0.011	0.014	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	0.002	NDIS	NDIS	0.006
16	NDIS	0.013	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.042	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	NDIS	NDIS	NDIS	0.003	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	0.295	NDIS	0.030	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	0.028	NDIS	0.074	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
9	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	NDIS	0.194	NDIS	NDIS	NDIS	NDIS	0.016	NDIS	NDIS	NDIS
10	NDIS	NDIS	0.032	NDIS	NDIS	0.145	0.006	NDIS	0.095	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
17	NDIS	NDIS	0.202	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
1	NDIS	NDIS	0.112	NDIS	0.003	NDIS	0.004	0.006	NDIS	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS
21	0.009	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
23	0.057	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
4	NDIS	0.024	0.023	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	0.052	NDIS	NDIS	NDIS	NDIS	NDIS	0.011
19	NDIS	NDIS	0.095	0.004	0.009	NDIS	NDIS	NDIS	NDIS	0.171	NDIS	NDIS	0.574	NDIS	0.060	0.012
3	NDIS	NDIS	0.054	NDIS	0.008	NDIS	0.129	NDIS	NDIS	0.040	NDIS	NDIS	0.042	0.004	0.032	0.003
22	NDIS	NDIS	0.074	NDIS	0.022	NDIS	NDIS	NDIS	NDIS	0.109	NDIS	0.049	NDIS	0.011	NDIS	0.003

Name of compounds correpsonidng to their serial number is presented in Table 4.14; NDIS – Not detected in sample

I. Percentage phenolic compound in Ofada rice

	Serial Number of Compounds											
TRT	1	2	3	4	5	6	7	8	9	10	11	12
2	0.040	0.395	NDIS	0.059	0.052	0.002	NDIS	NDIS	0.000	0.233	0.090	0.120
6	NDIS	0.030	NDIS	1.142	NDIS	NDIS	NDIS	0.097	NDIS	NDIS	0.774	NDIS
15	0.021	0.310	NDIS	0.132	0.019	0.064	NDIS	NDIS	0.002	0.196	0.086	NDIS
24	NDIS	0.106	NDIS	0.075	0.030	NDIS	NDIS	NDIS	NDIS	NDIS	0.028	0.030
8	NDIS	NDIS	NDIS	0.017	NDIS	NDIS	NDIS	0.026	NDIS	0.003	NDIS	NDIS
11	NDIS	NDIS	NDIS	3.654	NDIS	NDIS	NDIS	NDIS	NDIS	0.595	5.785	0.011
20	NDIS	NDIS	NDIS	18.943	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	21.447	0.018
25	NDIS	NDIS	NDIS	6.115	NDIS	NDIS	NDIS	NDIS	NDIS	2.642	9.275	NDIS
18	NDIS	NDIS	NDIS	9.357	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	11.542	0.011
13	NDIS	NDIS	NDIS	3.728	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	19.293	NDIS
16	NDIS	NDIS	NDIS	5.885	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	7.670	NDIS
12	0.029	NDIS	NDIS	0.019	NDIS	NDIS	0.027	NDIS	NDIS	NDIS	0.012	0.011
14	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	25.587	NDIS
7	NDIS	NDIS	NDIS	13.594	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	13.785	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	NDIS	NDIS	0.001	NDIS
9	0.008	0.008	NDIS	0.003	NDIS	NDIS	3.026	NDIS	0.000	NDIS	0.003	0.004
10	0.020	NDIS	0.007	0.010	NDIS	NDIS	4.305	NDIS	0.000	NDIS	0.006	0.006
17	NDIS	NDIS	NDIS	0.086	0.096	NDIS	NDIS	NDIS	NDIS	NDIS	0.062	0.114
1	0.049	0.321	NDIS	0.029	0.039	NDIS	NDIS	NDIS	NDIS	NDIS	0.028	0.091
21	NDIS	NDIS	NDIS	0.055	0.006	NDIS	NDIS	NDIS	0.002	NDIS	0.027	0.146
23	NDIS	0.236	NDIS	0.079	0.025	0.037	NDIS	NDIS	NDIS	NDIS	0.066	0.166
4	NDIS	NDIS	NDIS	NDIS	NDIS	0.034	NDIS	0.029		NDIS	NDIS	0.064
19	NDIS	NDIS	NDIS	0.009	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.087	NDIS
3	NDIS	NDIS	NDIS	0.013	0.002	0.004	NDIS	NDIS	0.000	NDIS	0.005	0.021
22	NDIS	NDIS	NDIS	0.017	0.019	NDIS	NDIS	NDIS	0.001	NDIS	0.022	0.130

Name of compounds corresponding to their serial number and aroma description is presented in Table 4.16; NDIS – Not detected in sample

J. Percentage of miscellaneous compounds in Ofada rice

	Serial Number of Compounds														
TRT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
2	0.022	0.004	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.040	0.054	NDIS	0.015	NDIS	NDIS
6	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
15	NDIS	NDIS	NDIS	NDIS	0.018	NDIS	NDIS	NDIS	NDIS	0.021	NDIS	NDIS	0.008	NDIS	0.003
24	NDIS	NDIS	NDIS	NDIS	0.010	NDIS	NDIS	NDIS	0.002	0.006	NDIS	NDIS	0.002	NDIS	NDIS
8	0.005	NDIS	0.026	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	NDIS
11	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	NDIS	NDIS	0.001	NDIS	NDIS
20	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	0.008	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003
25	NDIS	NDIS	4.952	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.006	NDIS	NDIS	NDIS	NDIS	NDIS
18	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	24.821	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
13	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	41.059	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
16	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	NDIS	0.011	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	0.029	NDIS	NDIS	0.016	NDIS	NDIS
14	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
7	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	NDIS	NDIS	0.012	NDIS	0.007
9	NDIS	NDIS	NDIS	0.004	NDIS	NDIS	NDIS	0.001	0.001	0.008	NDIS	0.001	0.018	0.001	NDIS
10	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.005	0.003	0.020	NDIS	0.009	0.028	0.000	NDIS
17	NDIS	NDIS	NDIS	NDIS	NDIS	0.912	NDIS	NDIS	0.017	0.006	NDIS	NDIS	0.274	NDIS	NDIS
1	NDIS	0.014	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	0.049	NDIS	NDIS	0.135	NDIS	NDIS
21	0.085	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	0.003	NDIS	0.024	NDIS	NDIS	NDIS	NDIS	NDIS
23	NDIS	NDIS	NDIS	0.000	NDIS	NDIS	NDIS	NDIS	0.007	0.010	NDIS	NDIS	0.158	NDIS	NDIS
4	NDIS	NDIS	NDIS	NDIS	0.341	NDIS	NDIS	NDIS	0.008	0.032	0.043	NDIS	0.068	NDIS	NDIS
19	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	2.298	NDIS	NDIS	NDIS	NDIS
3	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.001	0.002	0.004	NDIS	0.004	0.020	NDIS	NDIS
22	0.004	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	0.001	0.002	0.007	NDIS	0.001	0.066	0.004	0.004

Name of compounds corresponding to the serial number is presented in Table 4.16a; NDIS – Not detected in sample

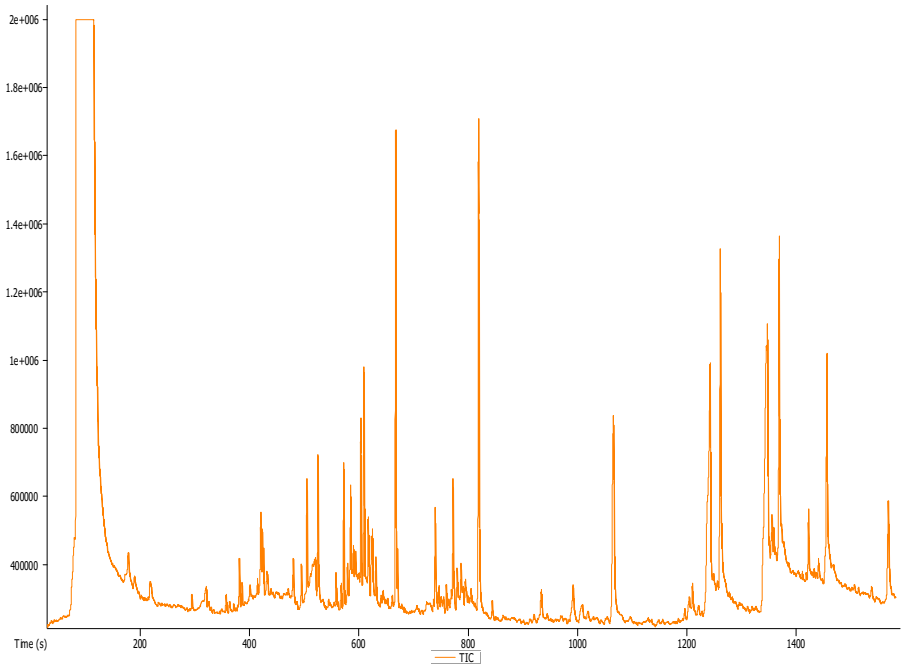
J Cont'd. Percentage of miscellaneous compounds in Ofada rice

	Serial Number of Compounds										
TRT	16	17	18	19	20	21	22	23	24	25	26
2	0.007	0.005	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.014	NDIS	NDIS
6	0.211	NDIS	NDIS	NDIS	NDIS	0.079	NDIS	NDIS	NDIS	NDIS	0.182
15	0.044	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.007	NDIS	NDIS
24	0.013	0.051	NDIS	NDIS	0.002	0.008	NDIS	NDIS	NDIS	0.112	0.058
8	0.019	0.022	0.028	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.045	0.000
11	3.098	0.003	3.256	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS
20	4.513	0.024	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.002	2.444
25	1.614	0.003	2.884	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
18	7.573	0.008	9.971	NDIS	0.001	NDIS	NDIS	NDIS	NDIS	NDIS	0.249
13	6.308	2.005	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	0.000
16	28.076	0.000	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
12	0.001	0.144	NDIS	NDIS	0.014	0.048	NDIS	NDIS	NDIS	0.049	NDIS
14	7.480	0.020	NDIS	NDIS	NDIS	0.002	NDIS	NDIS	NDIS	NDIS	NDIS
7	3.241	0.015	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
5	0.003	0.033	NDIS	0.009	0.015	0.032	NDIS	NDIS	0.008	NDIS	NDIS
9	0.000	0.027	NDIS	0.008	0.001	0.017	NDIS	0.007	NDIS	NDIS	NDIS
10	NDIS	0.047	NDIS	NDIS	0.011	0.024	NDIS	0.027	NDIS	0.066	NDIS
17	0.014	0.107	NDIS	NDIS	NDIS	0.403	NDIS	NDIS	0.051	NDIS	NDIS
1	0.004	0.100	NDIS	0.001	0.015	0.315	NDIS	0.028	NDIS	0.201	0.161
21	0.011	NDIS	NDIS	0.011	0.001	0.655	NDIS	0.028	NDIS	0.013	NDIS
23	NDIS	NDIS	NDIS	0.065	0.006	0.234	NDIS	0.030	0.010	NDIS	NDIS
4	0.001	0.166	NDIS	0.075	0.000	0.149	0.003	0.067	0.023	NDIS	NDIS
19	NDIS	0.179	0.031	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS	NDIS
3	0.001	NDIS	NDIS	NDIS	0.016	0.030	0.001	0.023	0.010	NDIS	NDIS
22	NDIS	NDIS	NDIS	NDIS	0.028	0.129	0.000	0.029	NDIS	NDIS	NDIS

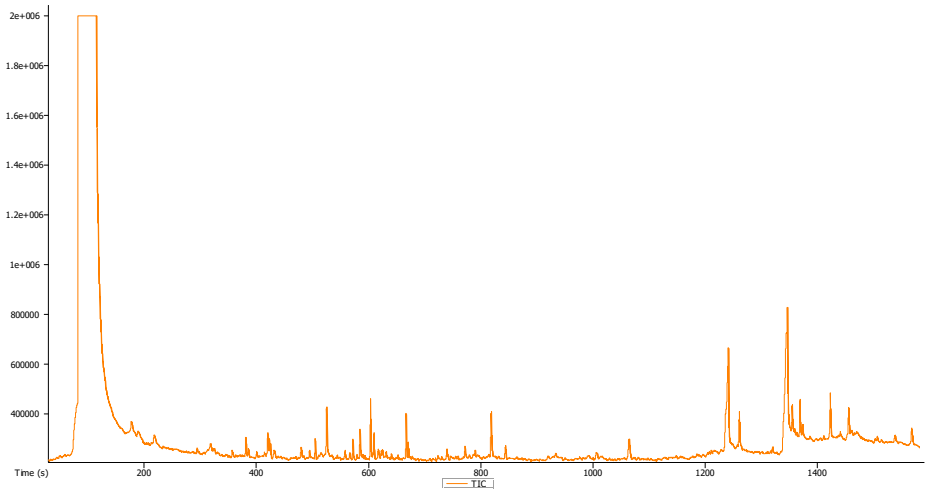
Name of compounds corresponding to the serial number is presented in Table 4.16; NDIS – Not detected in sample

Appendix 3

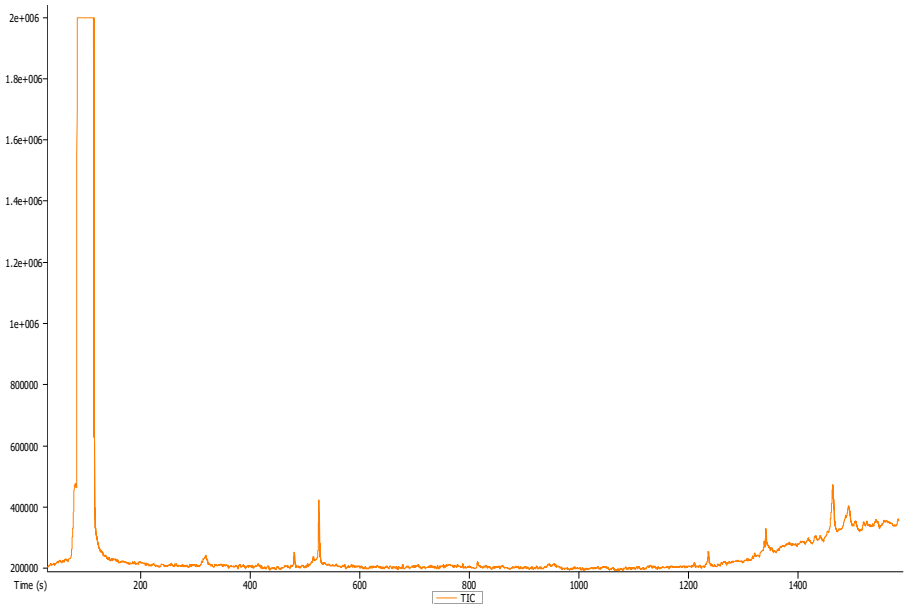
GCMS Chromatographs for rice extracts



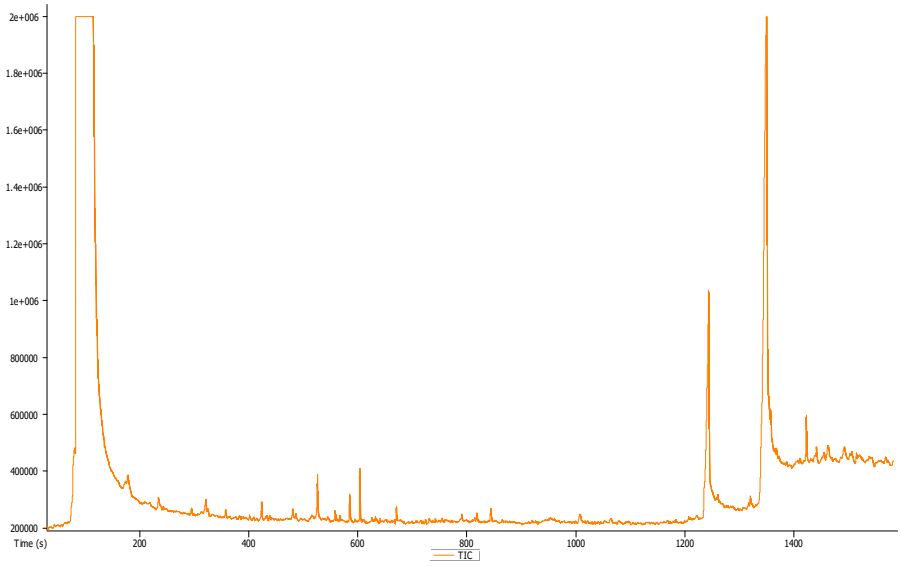
Chromatograph for treatment 1



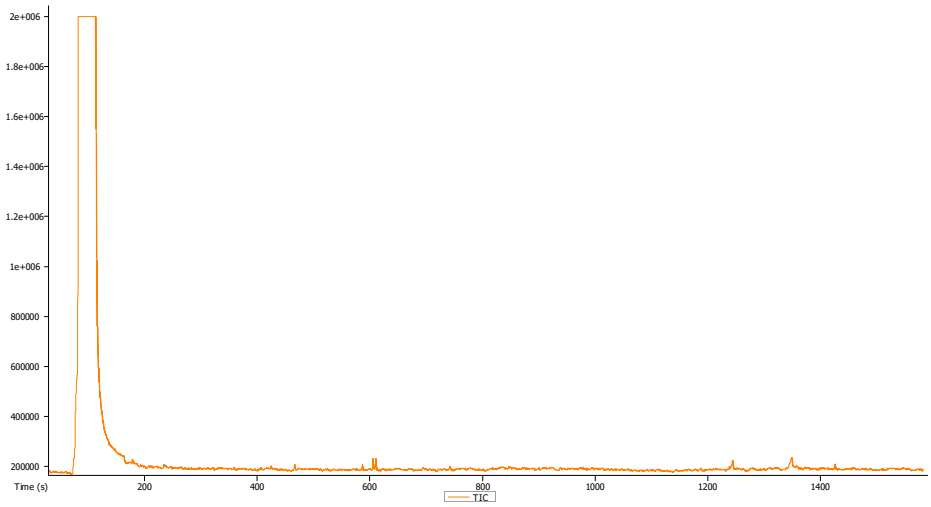
Chromatograph for treatment 2



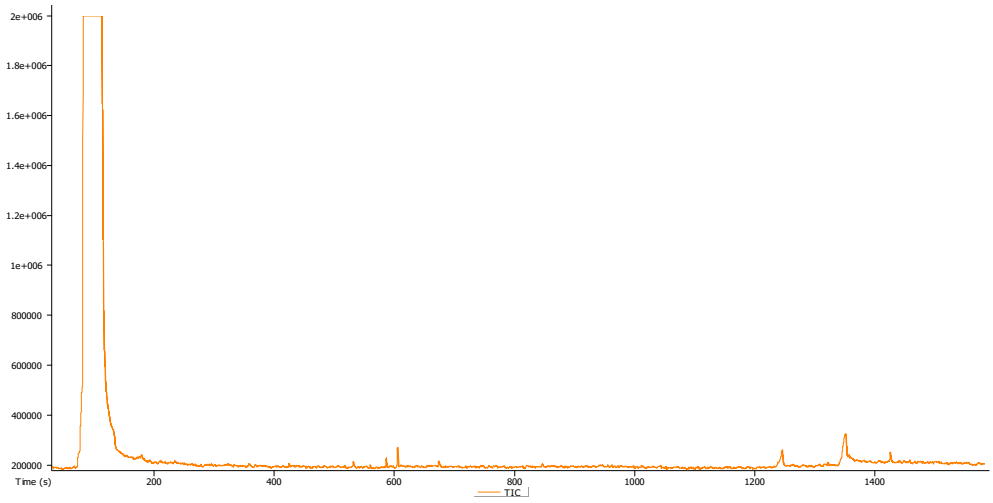
Chromatograph for treatment 3



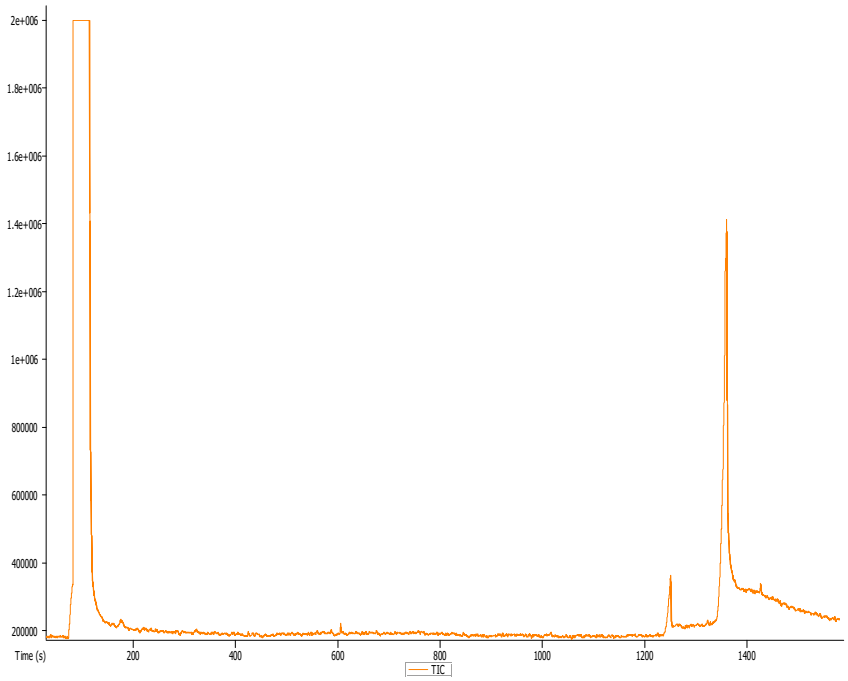
Chromatograph for treatment 4



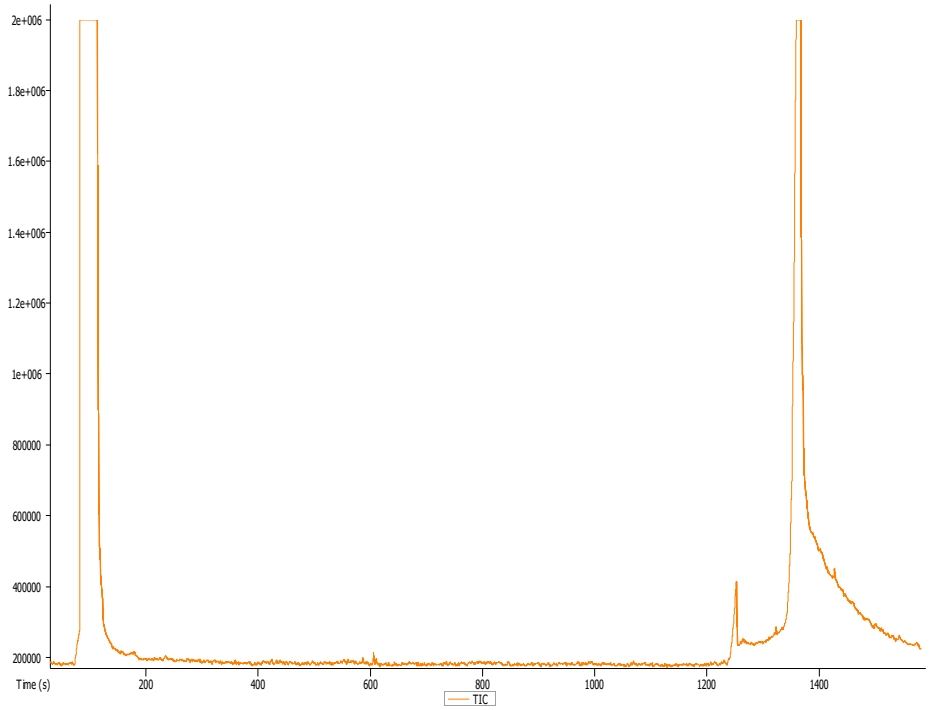
Chromatograph for treatment 5



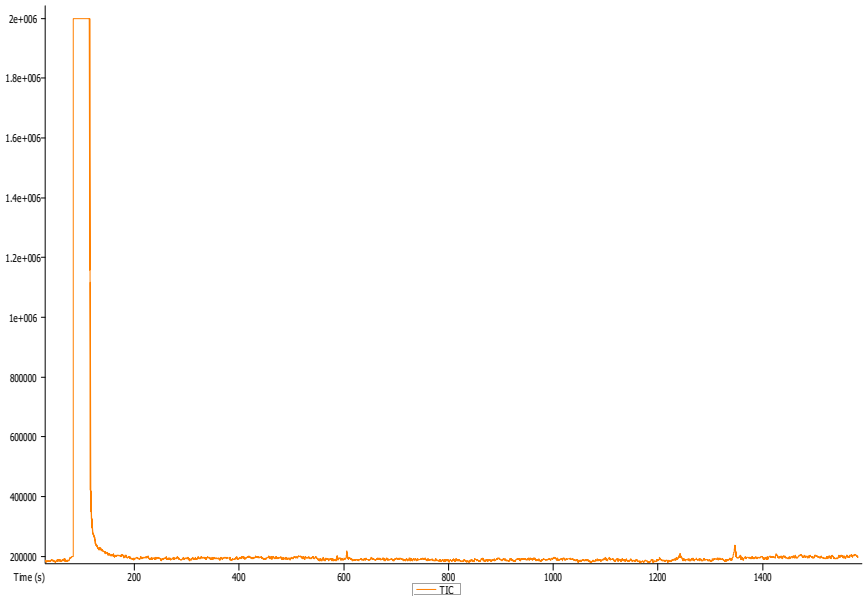
Chromatograph for treatment 6



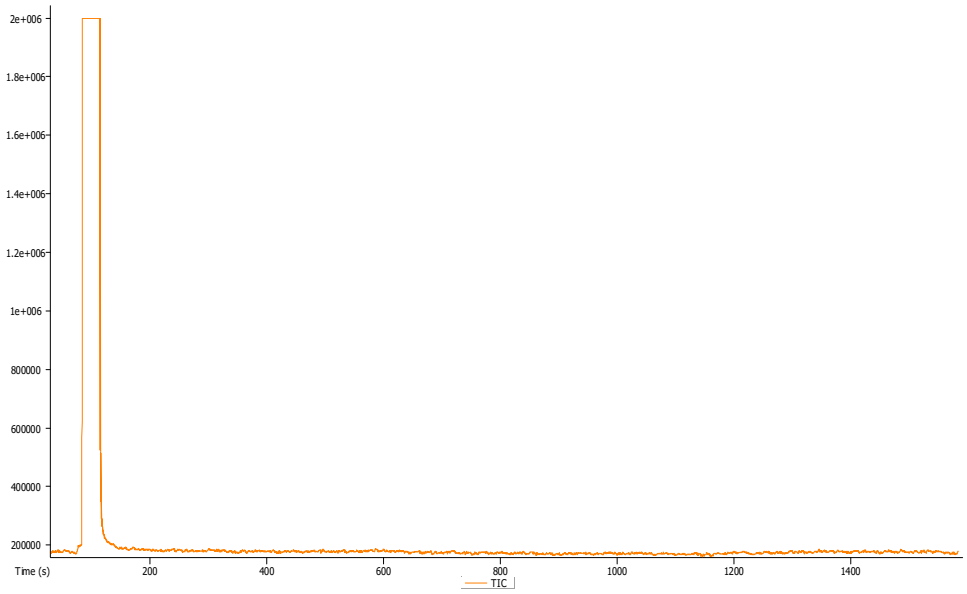
Chromatograph for treatment 7



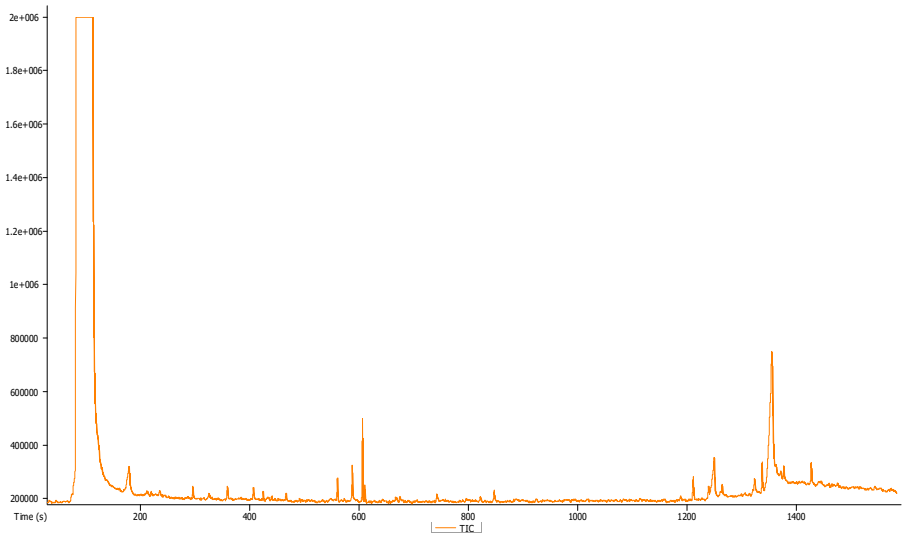
Chromatograph for treatment 8



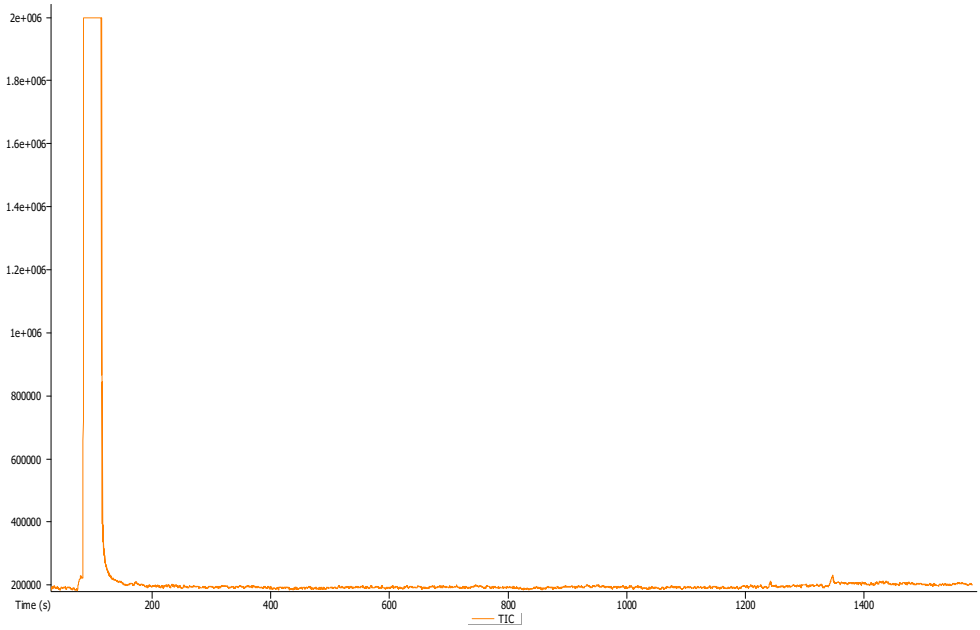
Chromatograph for treatment 9



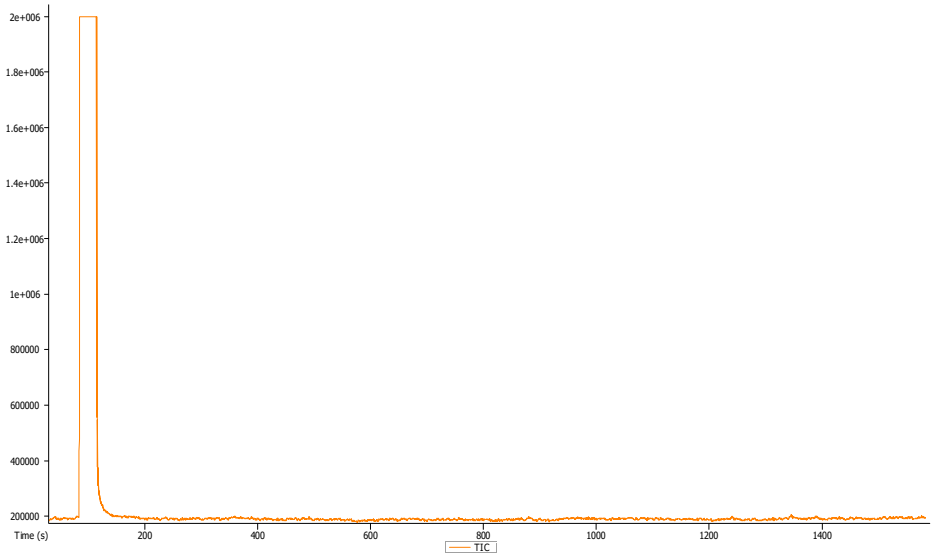
Chromatograph for treatment 10



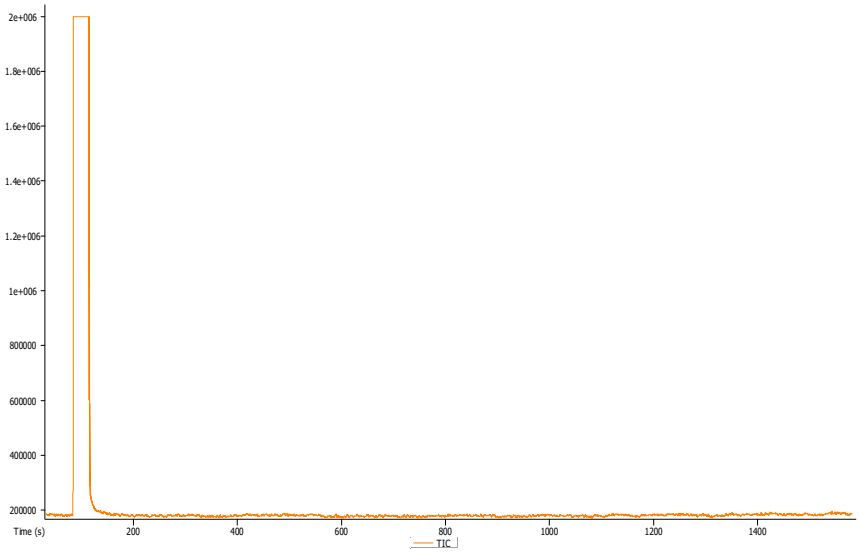
Chromatograph for treatment 11



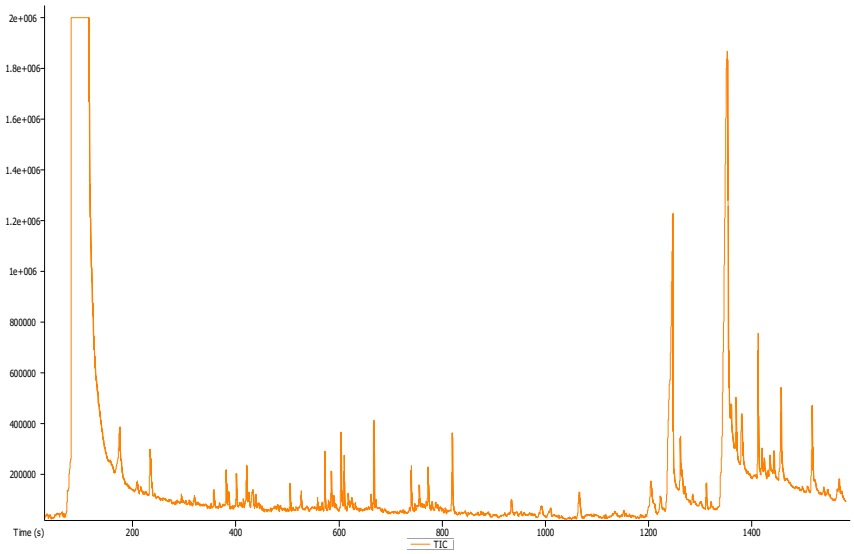
Chromatograph for treatment 12



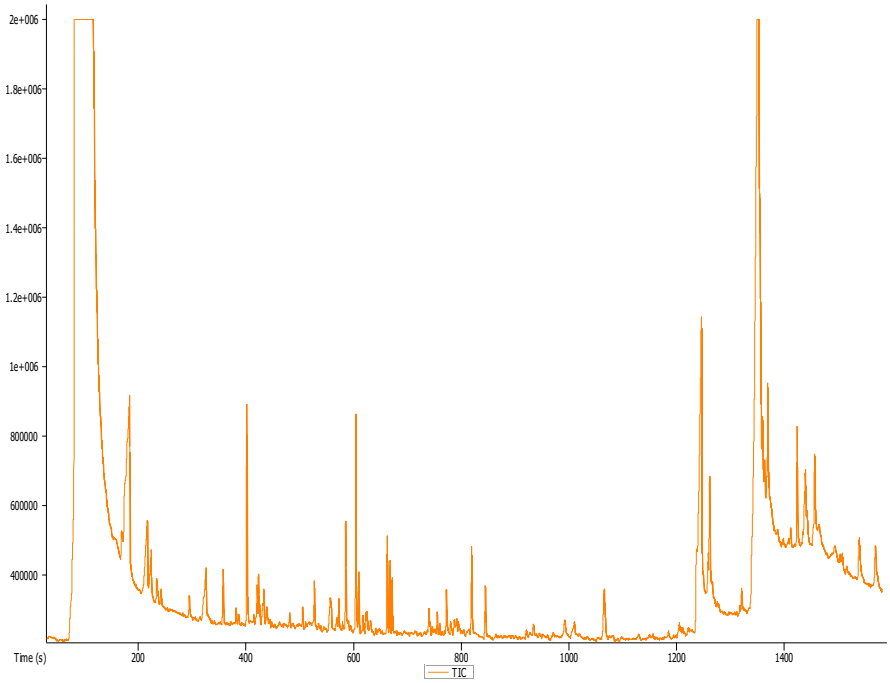
Chromatograph for treatment 13



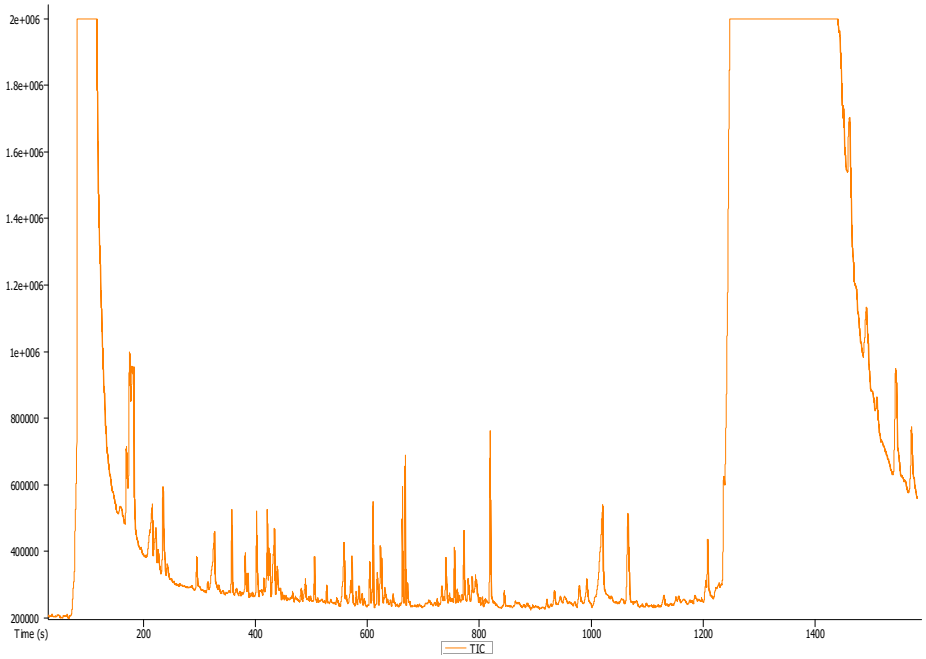
Chromatograph for treatment 14



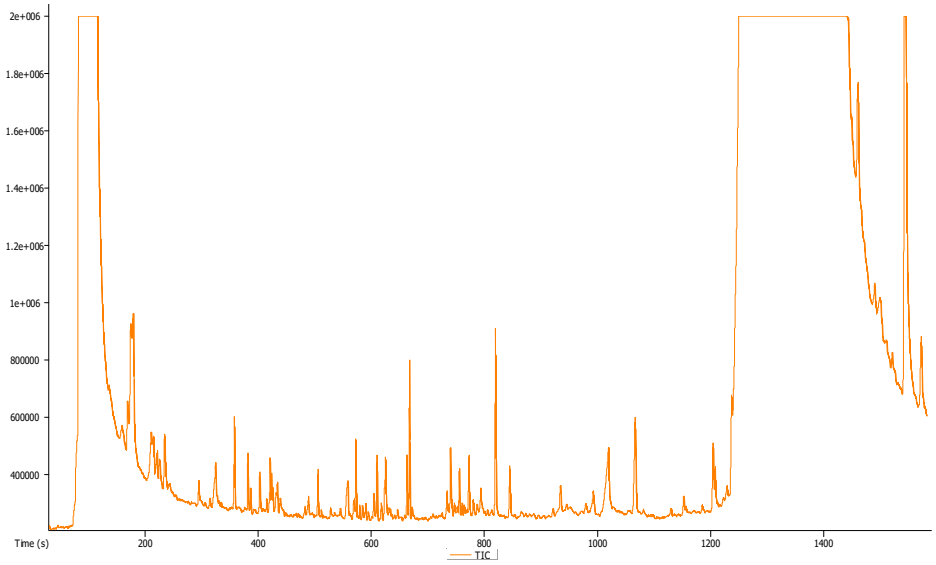
Chromatograph for treatment 15



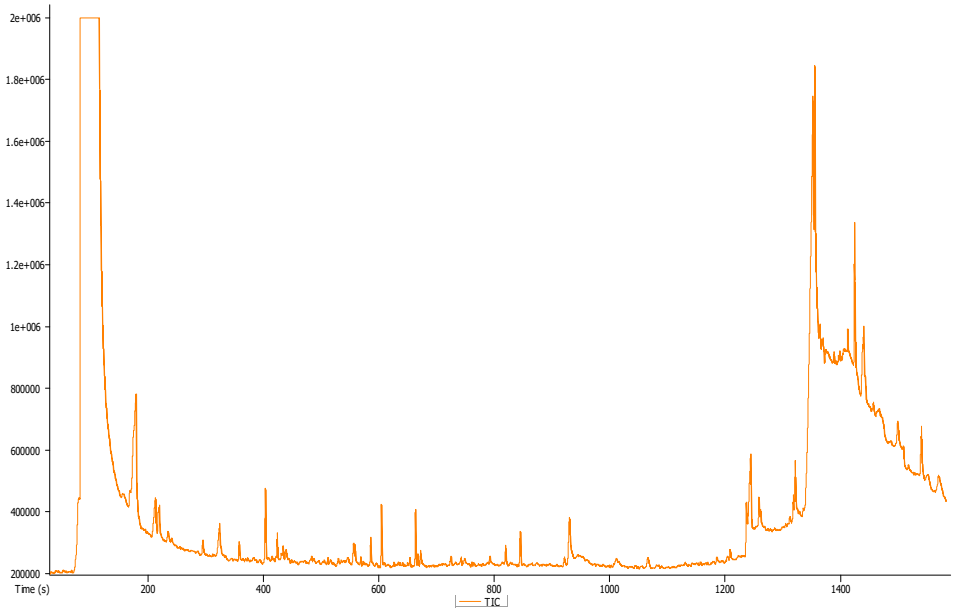
Chromatograph for treatment 16



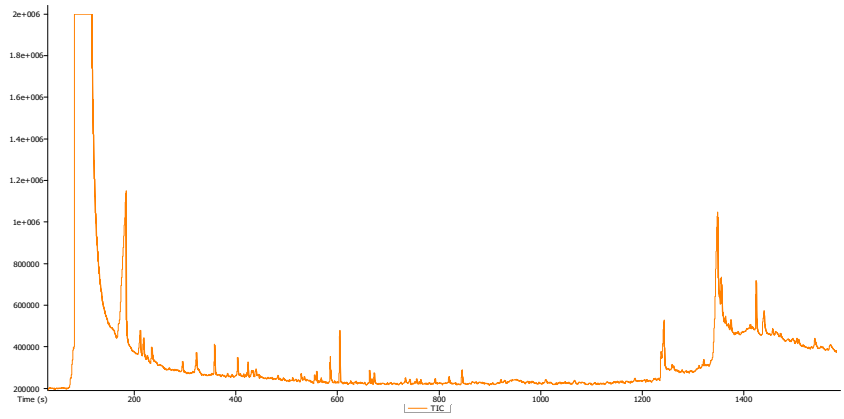
Chromatograph for treatment 17



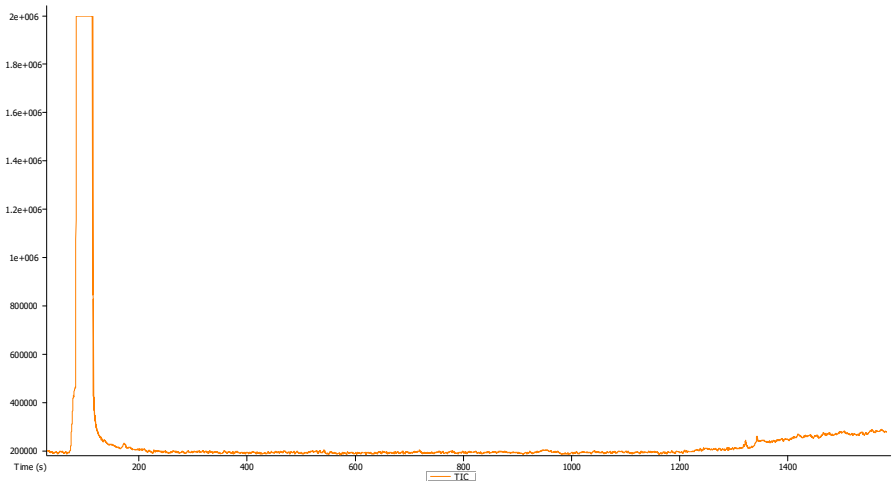
Chromatograph for treatment 18



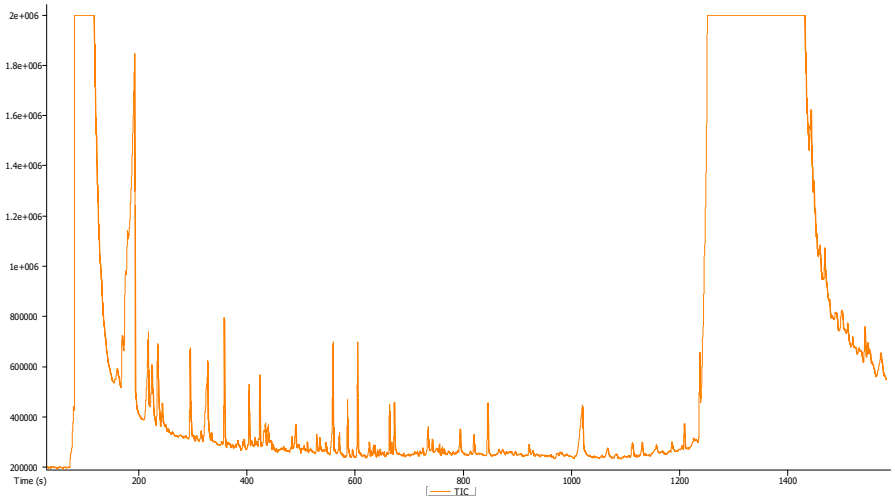
Chromatograph for treatment 19



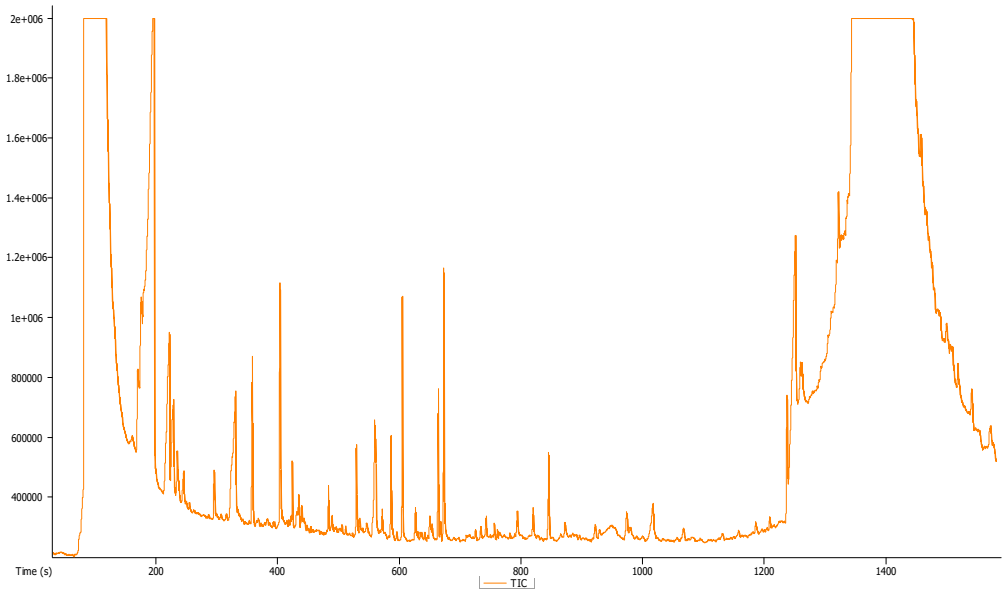
Chromatograph for treatment 20



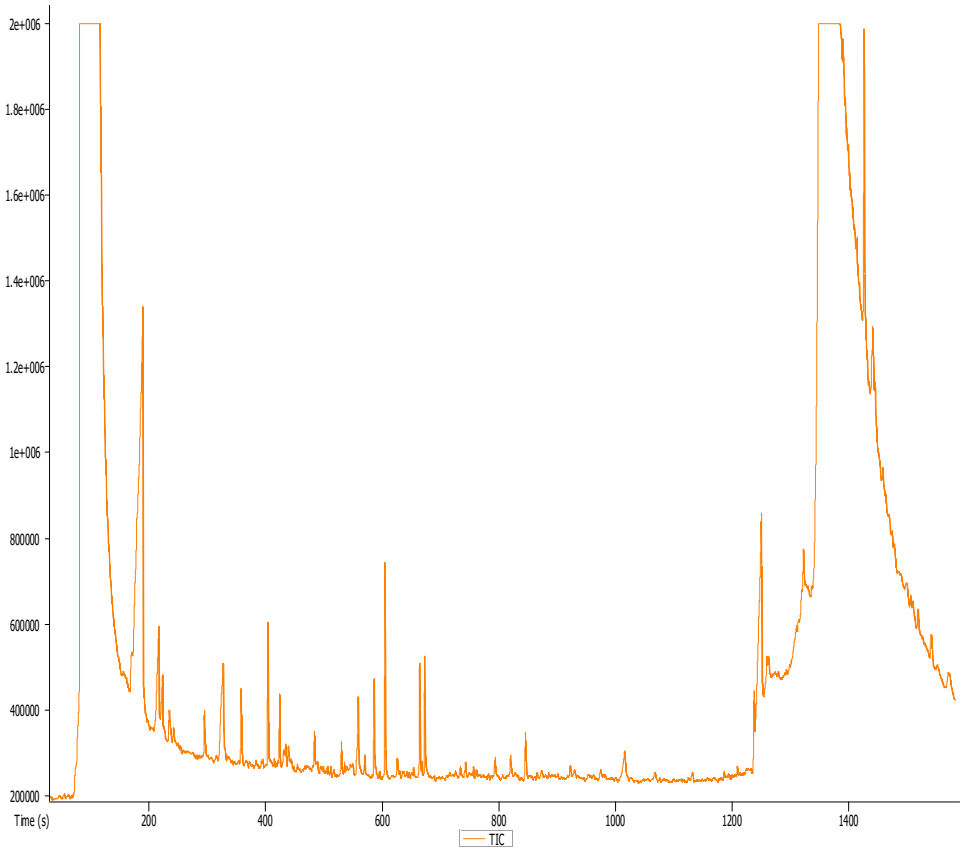
Chromatograph for treatment 21



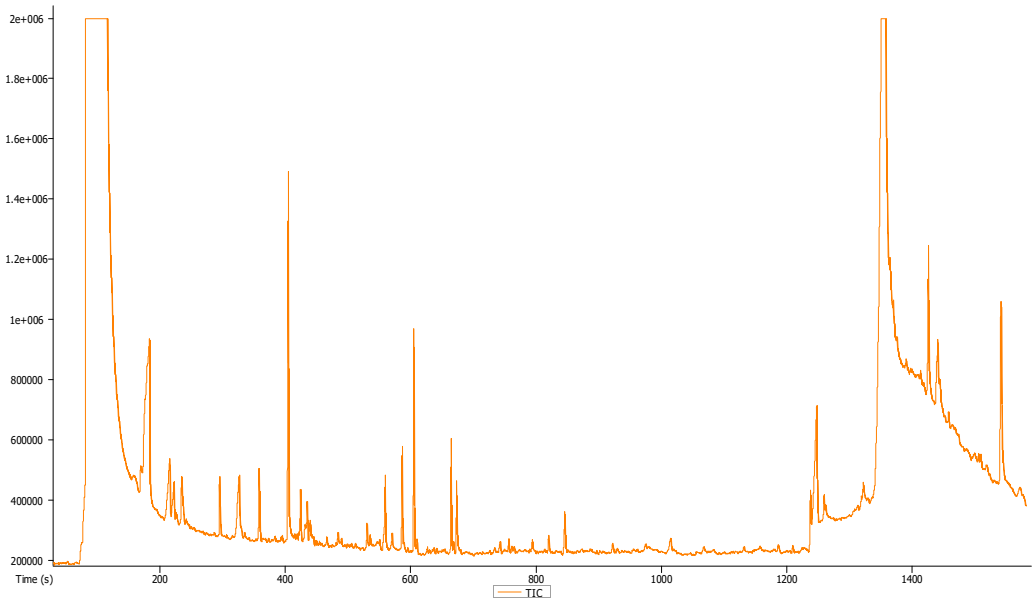
Chromatograph for treatment 22



Chromatograph for treatment 23



Chromatograph for treatment 24



Chromatograph for treatment 25

APPENDIX 4

Percentage Bacteria phylum occurrence in Ofada rice Paddy Fermenting soak-water

Initial soaking Temperature	Soaking Duration (Days)	1	2	3	4	5
	Microbial Community	Occurrence (%)				
30 °C	Proteobacteria	99.10	83.82	88.38	86.80	84.19
	Actinobacteria	0.55	10.35	0.06	0.09	0.19
	Acidobacteria	0.14	0.00	0.00	0.00	0.00
	Chloroflexi	0.07	0.00	0.00	0.00	0.00
	Bacteria_unclassified	0.04	0.00	0.00	0.00	0.00
	Bacteroidetes	0.04	0.11	0.15	1.24	2.33
	Elusimicrobia	0.04	0.00	0.00	0.00	0.00
	Firmicutes	0.04	5.72	11.42	11.88	13.29
	Minor Phyla	0.00	0.00	0.00	0.00	0.00
65°C	Proteobacteria	56.30	28.18	81.49	79.64	81.90
	Actinobacteria	27.06	16.22	0.09	0.06	0.80
	Acidobacteria	0.00	0.04	0.00	0.01	0.00
	Chloroflexi	0.00	0.03	0.00	0.00	0.00
	Bacteria_unclassified	0.00	0.04	0.00	0.00	0.00
	Bacteroidetes	0.29	0.06	0.002	0.01	0.04
	Elusimicrobia	0.00	0.01	0.00	0.00	0.00
	Firmicutes	16.28	55.33	18.42	20.29	17.26
	Minor Phyla	0.07	0.14	0.00	0.00	0.00
100 °C	Proteobacteria	0.40	0.07	0.04	0.13	0.03
	Actinobacteria	0.015	0.01	0.00	0.00	0.00
	Acidobacteria	0.00	0.00	0.00	0.00	0.00
	Chloroflexi	0.00	0.00	0.00	0.00	0.00
	Bacteria_unclassified	0.00	0.00	0.00	0.00	0.00
	Bacteroidetes	0.00	0.00	0.00	0.00	0.00
	Elusimicrobia	0.00	0.00	0.00	0.00	0.00
	Firmicutes	99.58	99.92	99.96	99.87	99.96
	Minor Phyla	0.00	0.00	0.00	0.00	0.00

APPENDIX 5

Analysis of variance output

ONEWAY Moisture Ash Fat protein Crudefibre Carbohydrate Energy Freefattyacid Amylose Amylopectin aminobutyric Nicotinicacid Panthote

nic Malic Pyruvic Lactic citric succinic fumaric Orotic asparagine cysteine Asparticacid Glu tamicacid serine glycine

Histidine Arginine Threonine Alanine Proline Tryptophan Methionine Phenylalaline BY sa mple

/STATISTICS DESCRIPTIVES

/MISSING ANALYSIS

/POSTHOC=DUNCAN ALPHA(0.05).

Oneway

Notes

Output Created		18-Mar-2019 17:26:17
Comments		
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	Filter	<none>
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	Split File	<none>
	N of Rows in Working Data File	75
Missing Value Handling	Definition of Missing	User-defined missing values are treated as missing.
	Cases Used	Statistics for each analysis are based on cases with no missing data for any variable in the analysis.

Syntax		ONEWAY Moisture Ash Fat protein Crudefibre Carbohydrate Energy Freefattyacid Amylose Amylopectin aminobutyric Nicotinicacid Panthotenic Malic Pyruvic Lactic citric succinic fumaric Orotic asparagine cysteine Asparticacid Glutamicacid serine glycine Histidine Arginine Threonine Alanine Proline Tryptophan Methionine Phenylalaline BY sample /STATISTICS DESCRIPTIVES /MISSING ANALYSIS /POSTHOC=DUNCAN ALPHA(0.05).
Resources	Processor Time	00:00:31.044
	Elapsed Time	00:00:34.208

\

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Moisture	Between Groups	120.941	24	5.039	40.858	.000
	Within Groups	6.167	50	.123		
	Total	127.108	74			
Ash	Between Groups	1.094	24	.046	22.410	.000
	Within Groups	.102	50	.002		
	Total	1.195	74			
Fat	Between Groups	5.081	24	.212	13.541	.000
	Within Groups	.782	50	.016		
	Total	5.862	74			
protein	Between Groups	20.227	24	.843	85.825	.000
	Within Groups	.491	50	.010		
	Total	20.718	74			
Crudefibre	Between Groups	6.385	24	.266	68.067	.000
	Within Groups	.195	50	.004		
	Total	6.580	74			
Carbohydrate	Between Groups	147.482	24	6.145	98.321	.000
	Within Groups	3.125	50	.062		
	Total	150.607	74			
Energy	Between Groups	1715.866	24	71.494	36.477	.000
	Within Groups	98.000	50	1.960		
	Total	1813.866	74			
Free fatty acid	Between Groups	50.764	24	2.115	125.158	.000

	Within Groups	.845	50	.017		
	Total	51.609	74			
Amylose	Between Groups	218.046	24	9.085	4.635	.000
	Within Groups	98.000	50	1.960		
	Total	316.046	74			
Amylopectin	Between Groups	218.046	24	9.085	4.319	.000
	Within Groups	105.167	50	2.103		
	Total	323.213	74			
aminobutyric	Between Groups	28.789	24	1.200	19.193	.000
	Within Groups	3.125	50	.062		
	Total	31.914	74			
Nicotinic acid	Between Groups	14.257	24	.594	26.402	.000
	Within Groups	1.125	50	.022		
	Total	15.382	74			
Panthotenic acid	Between Groups	.682	24	.028	11.361	.000
	Within Groups	.125	50	.002		
	Total	.807	74			
Malic acid	Between Groups	14.546	24	.606	94.703	.000
	Within Groups	.320	50	.006		
	Total	14.866	74			
Pyruvic acid	Between Groups	.001	24	.000	45.927	.000
	Within Groups	.000	50	.000		
	Total	.001	74			
lactic acid	Between Groups	35.069	24	1.461	584.490	.000

	Within Groups	.125	50	.002		
	Total	35.194	74			
citric acid	Between Groups	149.158	24	6.215	4.316	.000
	Within Groups	72.000	50	1.440		
	Total	221.158	74			
succinic acid	Between Groups	1.527	24	.064	25.449	.000
	Within Groups	.125	50	.002		
	Total	1.652	74			
fumaric acid	Between Groups	5.607	24	.234	5.840E4	.000
	Within Groups	.000	50	.000		
	Total	5.607	74			
orotic acid	Between Groups	.000	24	.000	239.158	.000
	Within Groups	.000	49	.000		
	Total	.000	73			
asparagine	Between Groups	20.914	24	.871	21.785	.000
	Within Groups	2.000	50	.040		
	Total	22.914	74			
cysteine	Between Groups	.020	24	.001	90.954	.000
	Within Groups	.000	50	.000		
	Total	.020	74			
Asparticacid	Between Groups	26.860	24	1.119	21.156	.000
	Within Groups	2.645	50	.053		
	Total	29.505	74			
Glutamicacid	Between Groups	919.305	24	38.304	17.024	.000

	Within Groups	112.500	50	2.250		
	Total	1031.805	74			
serine	Between Groups	7.956	24	.331	132.595	.000
	Within Groups	.125	50	.003		
	Total	8.081	74			
glycine	Between Groups	9.700	24	.404	10.104	.000
	Within Groups	2.000	50	.040		
	Total	11.700	74			
Histidine	Between Groups	.007	24	.000	7.258E3	.000
	Within Groups	.000	50	.000		
	Total	.007	74			
Arginine	Between Groups	.006	24	.000	61.561	.000
	Within Groups	.000	50	.000		
	Total	.006	74			
Threonine	Between Groups	24.328	24	1.014	11.263	.000
	Within Groups	4.500	50	.090		
	Total	28.828	74			
Alanine	Between Groups	1258.903	24	52.454	23.313	.000
	Within Groups	112.500	50	2.250		
	Total	1371.403	74			
Proline	Between Groups	1014.652	24	42.277	29.359	.000
	Within Groups	72.000	50	1.440		
	Total	1086.652	74			
Tryptophan	Between Groups	.001	24	.000	30.211	.000

	Within Groups	.000	50	.000		
	Total	.001	74			
Methionine	Between Groups	32.073	24	1.336	3.341E3	.000
	Within Groups	.020	50	.000		
	Total	32.093	74			
Phenylalaline	Between Groups	.002	24	.000	17.629	.000
	Within Groups	.000	50	.000		
	Total	.002	74			

Moisture

Duncan

sample	N	Subset for alpha = 0.05									
		1	2	3	4	5	6	7	8	9	10
F	3	4.19067									
K	3		6.62134								
R	3			7.68867							
H	3			8.04600	8.04600						
G	3			8.20267	8.20267	8.20267					
B	3				8.32267	8.32267					
L	3				8.60200	8.60200					
M	3					8.68867					
I	3					8.73767	8.73767				
A	3					8.77967	8.77967	8.77967			
Q	3					8.83367	8.83367	8.83367	8.83367		
C	3						9.34134	9.34134	9.34134	9.34134	
J	3							9.38167	9.38167	9.38167	
W	3							9.41200	9.41200	9.41200	

Y	3							9.41467	9.41467	9.41467	
P	3							9.42334	9.42334	9.42334	
N	3								9.44600	9.44600	
X	3								9.45867	9.45867	
O	3									9.69867	9.69867
V	3									9.77600	9.77600
U	3									9.77934	9.77934
E	3									9.81334	9.81334
S	3										1.01167E1
D	3										1.02080E1
T	3										1.02080E1
Sig.		1.000	1.000	.096	.082	.061	.059	.056	.066	.175	.131

Means for groups in homogeneous subsets are displayed.

Ash

Duncan

sample	N	Subset for alpha = 0.05							
		1	2	3	4	5	6	7	8
T	3	.18083							
L	3	.24333							
M	3		.39833						
D	3		.40333						
Y	3		.40583						
P	3		.41333						
U	3		.42833	.42833					
R	3		.46833	.46833	.46833				
Q	3			.50833	.50833	.50833			
V	3			.50833	.50833	.50833			
W	3				.53833	.53833	.53833		
B	3					.55833	.55833	.55833	
E	3					.57333	.57333	.57333	
I	3					.57333	.57333	.57333	
J	3					.57333	.57333	.57333	

O	3					.57333	.57333	.57333	
N	3					.58083	.58083	.58083	
S	3					.58333	.58333	.58333	
X	3					.58833	.58833	.58833	
K	3					.59083	.59083	.59083	
A	3					.59333	.59333	.59333	
G	3						.60583	.60583	
F	3							.63583	.63583
C	3							.64333	.64333
H	3								.71333
Sig.		.096	.101	.051	.088	.060	.133	.060	.051

Means for groups in homogeneous subsets are displayed.

protein

Duncan

sample	N	Subset for alpha = 0.05											
		1	2	3	4	5	6	7	8	9	10	11	12
O	3	7.40800											
A	3	7.51667	7.51667										
Y	3		7.58333										
R	3			8.08967									
J	3			8.18667	8.18667								
L	3			8.24000	8.24000								
S	3				8.30100	8.30100							
Q	3					8.42067	8.42067						
B	3					8.44600	8.44600						
U	3					8.46100	8.46100						
P	3						8.49433	8.49433					
C	3						8.49867	8.49867					
V	3						8.50767	8.50767					
N	3						8.55700	8.55700	8.55700				

K	3						8.59000	8.59000	8.59000	8.59000			
H	3						8.60800	8.60800	8.60800	8.60800			
M	3							8.65733	8.65733	8.65733			
E	3								8.73467	8.73467	8.73467		
G	3									8.75867	8.75867		
I	3										8.86400		
X	3										8.87833		
F	3										8.90933		
T	3											9.36767	
D	3											9.42267	9.42267
W	3												9.57433
Sig.		.185	.414	.084	.189	.076	.053	.086	.054	.067	.058	.500	.067

Means for groups in homogeneous subsets are displayed.

Carbohydrate

Duncan

sample	N	Subset for alpha = 0.05

		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
D	3	78.0700														
E	3	78.3100	78.3100													
T	3		78.5400	78.5400												
X	3		78.7000	78.7000												
P	3			78.7700												
W	3			78.9300	78.9300											
S	3				79.2300	79.2300										
N	3				79.2700	79.2700	79.2700									
I	3					79.4400	79.4400	79.4400								
U	3					79.6600	79.6600	79.6600	79.6600							
V	3					79.6800	79.6800	79.6800	79.6800							
C	3						79.7000	79.7000	79.7000							
G	3							79.8900	79.8900	79.8900						
M	3								79.9500	79.9500						
J	3									80.2100	80.2100					
H	3										80.4400	80.4400				

Q	3										80.4600	80.4600				
A	3											80.8100	80.8100			
O	3											80.8500	80.8500			
L	3												80.9600	80.9600		
Y	3												80.9600	80.9600		
B	3													81.3000		
R	3														81.9500	
K	3														81.9600	
F	3															84.6500
Sig.		.245	.076	.086	.122	.053	.064	.053	.214	.145	.255	.071	.510	.122	.961	1.000

Means for groups in homogeneous subsets are displayed.

Appendix 6

Estimated Coefficient of the model showing the effect of processing on antioxidant property of Ofada rice

Factor	TPC mgGAE/g	TFC mgQuercetin/g	TAC mgGAE/g	FRAP mgTrolox/g	DPPH I50 (µg/mL)
Intercept	49.86	0.74	0.98	26.22	746.67
A-Soaking Duration	-8.28	0.06	-0.26	0.54	-38.40
B-Initial Soaking Temperature	-1.17	-0.02	-0.64	0.68	63.48
C-Drying Temperature	1.84	-0.01	-0.04	0.97	13.03
D-Parboiling Temperature	1.58	-0.07	0.14	0.84	-26.60
A2	-9.98	0.01	0.79	-2.10	-98.76
B2	-1.96	0.02	1.19	-0.44	79.00
C2	-4.27	0.07	-0.98	-2.27	35.11
D2	9.93	0.10	-0.26	4.31	80.33
AB	-1.40	0.03	0.47	2.34	-142.56
AC	-0.14	0.02	-0.21	0.63	-103.17
AD	2.18	-0.05	-0.04	-0.33	67.90
BC	2.14	0.11	-0.37	-0.97	81.72
BD	2.58	0.13	-0.44	-0.08	52.96
CD	1.18	0.10	0.54	0.52	-49.41

Appendix 7:

ANOVA for response surface quadratic model of the effect of processing on antioxidant property of Ofada rice

	TPC mgGAE/g	TFC mgQuercetin/g	TAC mgGAE/g	FRAP mgTrolox/g	DPPH I50 (µg/mL)
R-Squared	0.54	0.89	0.72	0.65	0.50
Adj R-Squared	-0.09	0.73	0.34	0.16	-0.20
Pred R-Squared	-2.22	-0.03	-1.37	-5.73	-4.88
Adeq Precision	3.21	8.11	5.23	3.91	3.84
P-value	0.62	0.005	0.16	0.33	0.73

Appendix 8

Optimisation of processing conditions for optimum antioxidant properties of Ofada rice

Number	Soaking time	Initial Soaking Temperature	Parboiling Temperature	Drying Temperature	DPPH	TPC	FRAP	TFC	TAC	Desirability	
1	1	35	80	38.14	743.65	58.39	26.57	1.14	2.74	0.64	Selected
2	1	35	80.22	39.84	745.47	58.44	26.75	1.11	2.84	0.64	
3	5	100	120	58.15	766.34	44.69	32.07	1.04	1.74	0.56	
4	2.77	35	80	46.36	899.90	60.99	28.53	1.08	1.72	0.56	
5	4.75	100	120	60.18	808.25	48.13	32.11	1.06	1.44	0.55	
6	5	100	120	65.61	760.28	44.66	31.49	1.15	1.27	0.55	
7	4.99	100	118.66	54.87	757.23	42.68	31.53	0.98	1.90	0.55	
8	4.93	100	80	46.27	604.40	31.17	31.04	0.93	2.73	0.54	
9	4.59	100	80	48.05	677.76	36.52	31.20	0.90	2.37	0.53	
10	1.12	35	120	46.43	532.40	54.19	29.97	0.73	3.80	0.52	

Appendix 9

Estimated Coefficient of the model showing the effect of Processing on Amino acids Profile of Ofada rice

Factor	Asparagine %	Cysteine %	Aspartic Acid %	Glutamic acid %	Serine %	Glycine %	Histidine %
Intercept	3.018	0.028	1.674	14.151	1.234	1.529	0.016
A-Soaking Duration	-0.063	-0.002	-0.281	-0.987	-0.051	0.121	0.004
B-Initial Soaking Temperature	0.185	0.009	0.216	1.291	0.058	0.005	0.004
C-Drying Temperature	-0.128	-0.001	0.121	1.348	-0.052	-0.105	0.001
D-Parboiling Temperature	0.039	0.001	0.308	2.477	0.028	-0.017	-0.004
A2	0.080	-0.017	1.174	4.232	-0.055	-0.309	-0.011
B2	0.015	0.012	0.578	2.340	-0.043	-0.235	0.002
C2	0.433	-0.005	0.415	0.244	0.248	0.331	-0.007
D2	-0.294	-0.008	-1.341	-4.766	0.095	0.205	0.008
AB	-0.021	-0.001	-0.149	-1.462	-0.097	-0.105	0.004
AC	0.203	0.000	-0.048	0.174	0.137	0.122	-0.001
AD	-0.304	-0.002	-0.278	-1.924	0.029	-0.019	-0.006
BC	0.076	0.001	-0.066	-0.717	0.125	0.125	-0.003
BD	-0.037	0.003	-0.077	-0.013	-0.118	-0.188	-0.006
CD	-0.012	0.001	0.095	0.717	-0.117	-0.158	0.001

Appendix 10:

ANOVA for response surface quadratic model of the effect of processing on Amino acid profile of Ofada rice

	Asparagine	Cysteine	Aspartic Acid	Glutamic acid	Serine	Glycine	Histidine
R-Squared	0.471	0.432	0.841	0.644	0.694	0.812	0.869
Adj R-Squared	-0.269	-0.363	0.617	0.145	0.265	0.55	0.686
Pred R-Squared	-3.056	-9.549	-1.407	-5.424	-2.477	-0.345	-0.556
Adeq Precision	3.478	3.166	9.143	5.455	4.591	6.941	10.27
P-value	0.787	0.856	0.02	0.348	0.225	0.039	0.009

	Arginine	Threonine	Alanine	Proline	Tryptophan	Methionine	Phenylalanine
R-Squared	0.620	0.684	0.798	0.563	0.695	0.508	0.568
Adj R-Squared	0.087	0.241	0.516	-0.048	0.268	-0.180	-0.038
Pred R-Squared	-0.839	-2.571	-0.110	-3.262	-3.944	-9.911	-1.616
Adeq Precision	4.180	5.277	6.627	3.770	5.979	3.246	3.315
P-value	0.413	0.247	0.052	0.567	0.222	0.707	0.556

Appendix 11

Optimisation of processing conditions for maximum Yield of amino acid profile

Number	Soaking time	Soaking Temperature	Parboiling Temperature	Drying Temperature	Asparagine	Cysteine	Aspartic Acid	Glutamic acid	Serine	Glycine	Histidine	Desirability	
1	2.06	100	89.84	69.98	3.42	0.04	2.84	17.36	1.70	1.75	0.01	0.56	Selected
2	2.63	100	105.08	30	3.76	0.04	2.90	18.26	1.49	1.62	0.01	0.54	
3	2.05	100	93.44	67.66	3.41	0.04	3.04	18.74	1.58	1.58	0.01	0.54	
4	2.44	99.94	101.02	30	3.79	0.04	3.03	18.55	1.50	1.61	0.01	0.54	
5	1.88	35	115.08	30	3.67	0.02	2.25	13.98	1.83	2.07	0.01	0.46	
6	1.92	35	114.56	30.55	3.64	0.02	2.25	14.04	1.80	2.03	0.01	0.46	
7	2.37	35	111.32	30	3.58	0.02	2.19	13.53	1.75	2.04	0.01	0.46	
8	3.88	35	103.79	70	3.14	0.02	2.80	18.89	1.28	1.49	0.01	0.44	
9	3.79	35	104.29	70	3.13	0.02	2.77	18.74	1.28	1.49	0.01	0.44	
10	3.78	35	112.49	30	3.27	0.02	2.00	12.92	1.72	2.12	0.00	0.43	

Appendix 11 Contd.

Optimisation of processing conditions for maximum Yield of amino acid profile

Number	Soaking time	Initial Soaking Temperature	Parboiling Temperature	Drying Temperature	Arginine	Threonine	Alanine	Proline	Tryptophan	Methionine	Phenylalanine	Desirability	
1	2.06	100	89.84	69.98	0.01	3.30	22.66	13.86	0.01	2.28	0.01	0.56	Selected
2	2.63	100	105.08	30	0.02	3.05	20.30	12.57	0.01	2.12	0.00	0.54	
3	2.05	100	93.44	67.66	0.01	3.10	20.72	12.86	0.01	2.11	0.01	0.54	
4	2.44	99.94	101.02	30	0.02	3.05	20.19	12.66	0.01	2.17	0.00	0.54	
5	1.88	35	115.08	30	0.01	3.62	23.22	13.55	0.00	1.92	0.01	0.46	
6	1.92	35	114.56	30.55	0.01	3.56	22.85	13.30	0.00	1.90	0.01	0.46	
7	2.37	35	111.32	30	0.01	3.49	23.39	13.37	0.00	2.04	0.01	0.46	
8	3.88	35	103.79	70	0.01	2.74	20.60	10.76	0.01	2.07	0.01	0.44	
9	3.79	35	104.29	70	0.01	2.74	20.53	10.66	0.01	2.07	0.01	0.44	
10	3.78	35	112.49	30	0.01	3.50	24.82	13.96	0.01	2.27	0.01	0.43	

Appendix 12

Estimated Coefficient of the model showing the effect of Processing on organic acids Profile of Ofada rice

	2-aminobutyric acid %	Nicotinic acid %	Panthotenic acid %	Malic acid %	Pyruvic acid %	Lactic acid %	Citric acid %	Succinic acid %	Fumaric acid %	Orotic acid %
Intercept	1.554	1.739	0.355	0.587	0.004	-0.171	6.454	0.343	0.165	0.002
A-Soaking time	-0.277	-0.045	-0.036	-0.212	-0.001	-0.299	0.417	-0.021	-0.141	0.000
B-Initial Soaking Temperature	0.101	0.199	0.021	0.137	0.000	-0.324	1.221	0.017	0.092	-0.001
C-Drying Temperature	-0.108	-0.021	0.032	0.049	0.001	0.235	0.424	0.025	0.013	-0.001
D-Parboiling Temperature	0.051	0.078	0.000	0.206	0.002	-0.095	0.419	0.052	0.136	0.001
A2	0.091	0.243	0.114	0.379	0.000	0.817	1.655	0.040	0.212	0.001
B2	0.226	-0.048	0.086	0.097	0.002	0.605	0.436	-0.042	0.028	-0.001
C2	-0.167	0.294	0.002	-0.380	-0.002	-0.167	-0.234	-0.125	-0.221	-0.001
D2	-0.092	-0.137	-0.156	-0.294	0.002	-0.702	-2.113	-0.022	-0.143	-0.001
AB	0.078	0.014	0.004	-0.206	-0.001	0.294	-0.467	-0.072	-0.108	0.000
AC	-0.033	-0.145	-0.028	0.063	0.000	-0.262	-0.535	0.040	0.046	0.001
AD	-0.348	-0.282	-0.021	-0.126	-0.002	0.287	-0.421	-0.044	-0.076	-0.001
BC	-0.053	-0.046	-0.048	-0.049	0.000	-0.191	-0.681	-0.005	-0.008	-0.001
BD	0.157	-0.177	0.010	0.205	0.001	0.119	0.185	0.030	0.119	0.001
CD	-0.307	0.311	0.032	0.038	0.000	0.176	0.854	-0.014	0.017	0.001

Appendix 13

ANOVA for response surface quadratic model of the effect of processing on Amino acid profile of Ofada rice

	2-aminobutyric acid	Nicotinic acid	Panthotenic acid	Malic acid	Pyruvic acid	Lactic acid	Citric acid	Succinic acid	Fumaric acid	Orotic acid
R-Squared	0.352	0.764	0.703	0.676	0.537	0.839	0.833	0.525	0.642	0.269
Adj R-Squared	-0.556	0.433	0.287	0.221	-0.110	0.615	0.600	-0.140	0.141	-0.868
Pred R-Squared	-7.472	-0.796	-4.389	-3.407	-6.752	-1.876	-0.914	-7.872	-3.083	-7.514
Adeq Precision	2.195	7.450	6.422	6.069	3.940	8.802	7.124	3.695	5.796	1.713
P-value	0.949	0.094	0.204	0.267	0.636	0.021	0.025	0.666	0.352	0.992

Appendix 14

Optimisation of processing conditions for maximum yield of organic acid profile

No	Soaking time	Initial Soaking Temperature	Drying Temperature	Parboiling Temperature	2-aminobutyric acid	Nicotinic acid	Panthotenic acid	Malic acid	Pyruvic acid	Lactic acid	Citric acid	Succinic acid	Fumaric acid	Orotic acid	Desirability	
1	1	100	46.690	109.680	2.460	2.192	0.583	1.807	0.010	0.571	9.697	0.507	0.871	0.002	0.575	Selected
2	1	100	49.190	107.530	2.377	2.207	0.597	1.781	0.010	0.719	9.879	0.495	0.850	0.002	0.567	
3	1	99.96	41.860	106.860	2.417	2.185	0.592	1.725	0.009	0.582	9.645	0.480	0.819	0.003	0.567	
4	1	46.63	54.850	111.140	2.077	2.207	0.517	1.006	0.008	1.283	6.974	0.353	0.415	0.003	0.487	
5	5	35	51.460	95.590	1.508	1.663	0.496	0.943	0.006	0.940	8.172	0.384	0.290	0.002	0.391	
6	5	38.73	57.900	96.840	1.408	1.692	0.495	0.899	0.005	0.835	8.277	0.390	0.267	0.003	0.383	
7	5	68.44	52.070	101.410	1.333	1.915	0.432	0.764	0.003	0.353	8.528	0.366	0.244	0.003	0.319	

Appendix 15

Estimated Coefficient of the model showing the effect of Processing on chemical properties of Ofada rice

Factor	Moisture content	Ash	Fat	Acid value	Free fatty acid	Amylose	Amylopectin
Intercept	9.865944	0.538325	1.369374	3.033672	1.525937	21.53988	78.46012
A-Soaking time	0.575127	0.009039	-0.01509	-0.1011	-0.05085	0.961335	-0.96134
B-Initial Soaking Temperature	0.287965	-0.0264	0.097591	0.733918	0.369161	0.032247	-0.03225
C-Drying Temperature	-0.90464	0.017498	-0.01715	-0.92498	-0.46527	0.108916	-0.10892
D-Parboiling Temperature	-0.00502	-0.00082	0.133477	-0.43983	-0.22124	0.015317	-0.01532
A2	-1.36665	0.090892	-0.26153	0.170923	0.085974	-2.40723	2.407232
B2	-0.4243	-0.0439	-0.03473	1.304763	0.656296	0.034018	-0.03402
C2	-0.35191	0.001344	-0.13722	1.186829	0.596975	1.100675	-1.10067
D2	1.100787	-0.08449	0.033001	-2.39516	-1.20477	-0.91047	0.910466
AB	-0.07412	0.032903	-0.18852	0.01042	0.005241	0.063343	-0.06334
AC	0.742527	-0.01707	0.037184	-0.82091	-0.41292	0.003297	-0.0033
AD	0.159335	-0.00456	-0.05696	-0.61303	-0.30836	-0.00166	0.001657
BC	0.206112	0.058315	-0.00869	0.116237	0.058467	0.139729	-0.13973
BD	0.10399	0.018852	0.031298	-0.14583	-0.07335	0.55119	-0.55119
CD	-0.277	-0.02157	-0.06286	0.873147	0.439193	0.105591	-0.10559

Appendix 16:

ANOVA for response surface quadratic model of the effect of processing on chemical properties of Ofada rice

	Moisture	Ash	Fat	Acid value	Free fatty acid	Amylose	Amylopectin
R-Squared	0.922258	0.545699	0.717484	0.801788	0.801788	0.905143	0.905143
Adj R-Squared	0.81342	-0.09032	0.321962	0.524291	0.524291	0.772343	0.772343
Pred R-Squared	-0.4384	-6.18739	-1.10688	-2.00554	-2.00554	-0.98047	-0.98047
Adeq Precision	12.704	3.418063	6.279951	6.401542	6.401542	7.898296	7.898296
P-value	0.0009	0.6141	0.1735	0.0487	0.0487	0.0022	0.0022

Appendix 17

Optimisation of processing conditions for chemical properties of Ofada

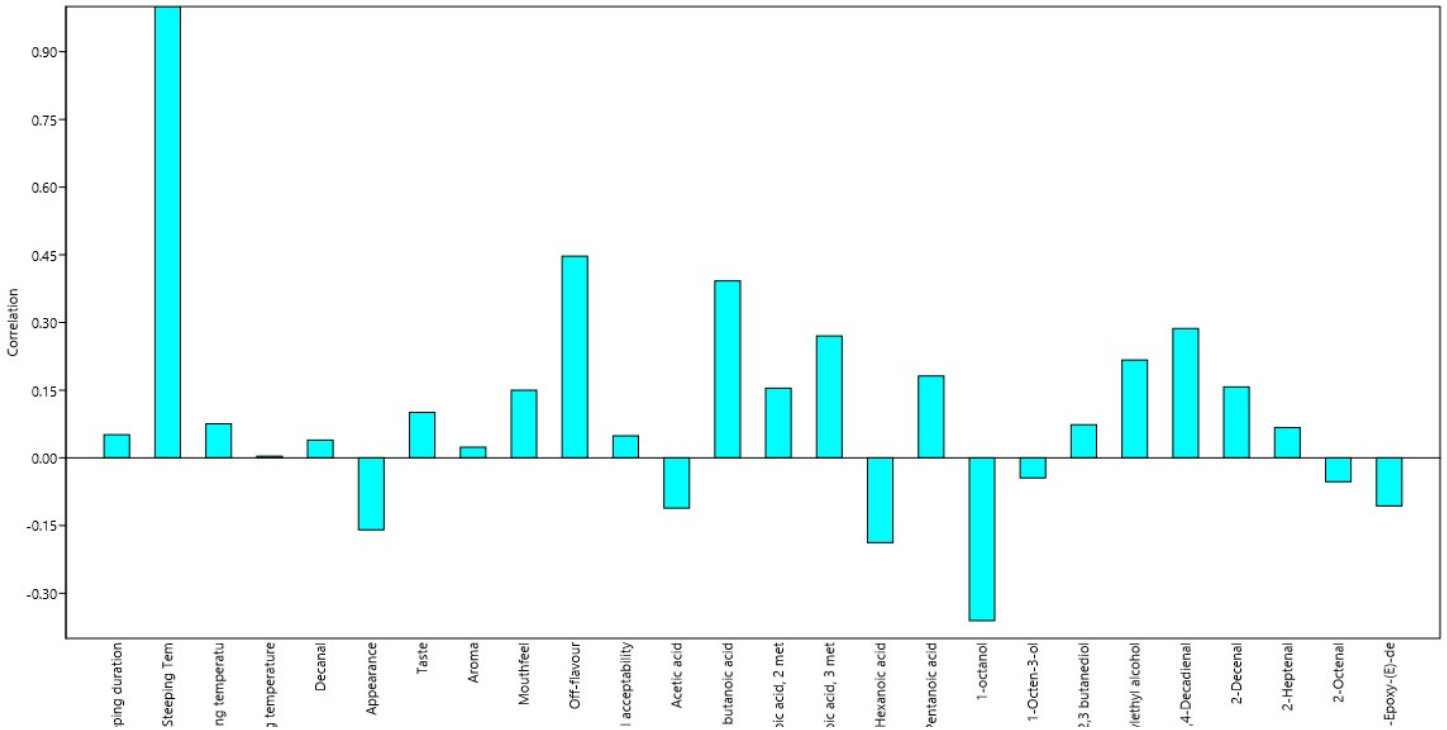
Number	Soaking time	Initial Soaking Temperature	Drying Temperature	Parboiling Temperature	Moisture	Ash	Fat	Acid Value	Free fatty acid	Amylose	Amylopectin	Desirability	
1	3.33	35.13	70	85.99	8.68	0.47	1.11	2.08	1.05	22.55	77.45	0.67	Selected
2	3.32	35.87	69.98	86.24	8.68	0.47	1.12	2.10	1.05	22.55	77.45	0.67	
3	3.23	36.74	70	86.63	8.64	0.47	1.11	2.15	1.08	22.55	77.45	0.67	
4	3.62	35	70	86.84	8.69	0.47	1.13	2.19	1.10	22.55	77.45	0.67	
5	3.21	52.04	70	111.88	8.61	0.48	1.21	2.51	1.26	22.34	77.66	0.67	
6	3.25	52.29	70	109.81	8.55	0.50	1.21	2.70	1.36	22.47	77.53	0.67	
7	3.25	54.36	70	109.39	8.58	0.50	1.21	2.72	1.37	22.52	77.48	0.67	
8	3.49	45.62	70	110.58	8.48	0.47	1.19	2.63	1.32	22.34	77.66	0.67	

Appendix 18
Summary of principal component result

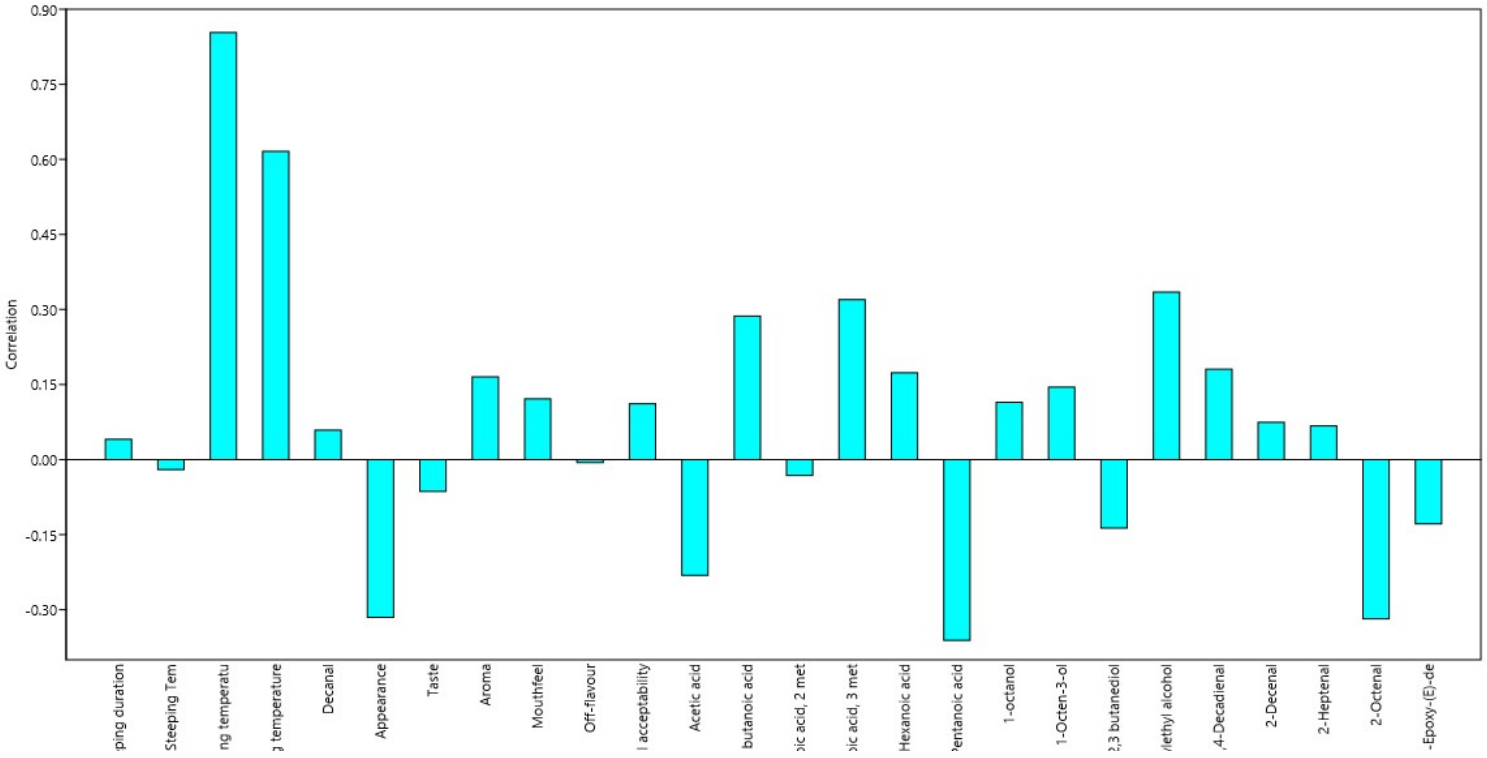
PC	Eigenvalue	% variance
1	1023.01	63.634
2	320.367	19.928
3	247.578	15.4
4	5.59196	0.34783
5	3.22316	0.20049

Appendix 19

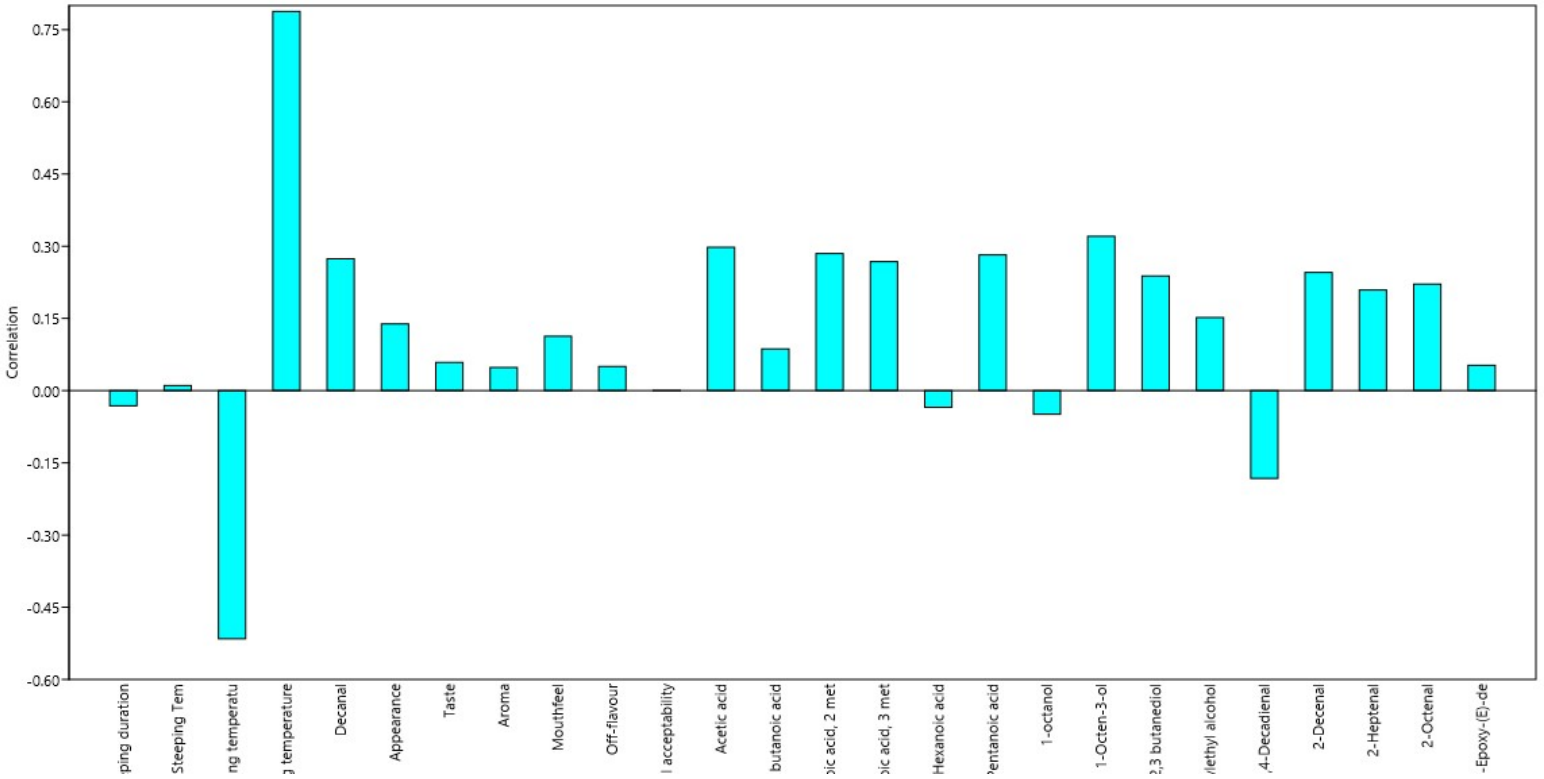
Loading plot for principal component



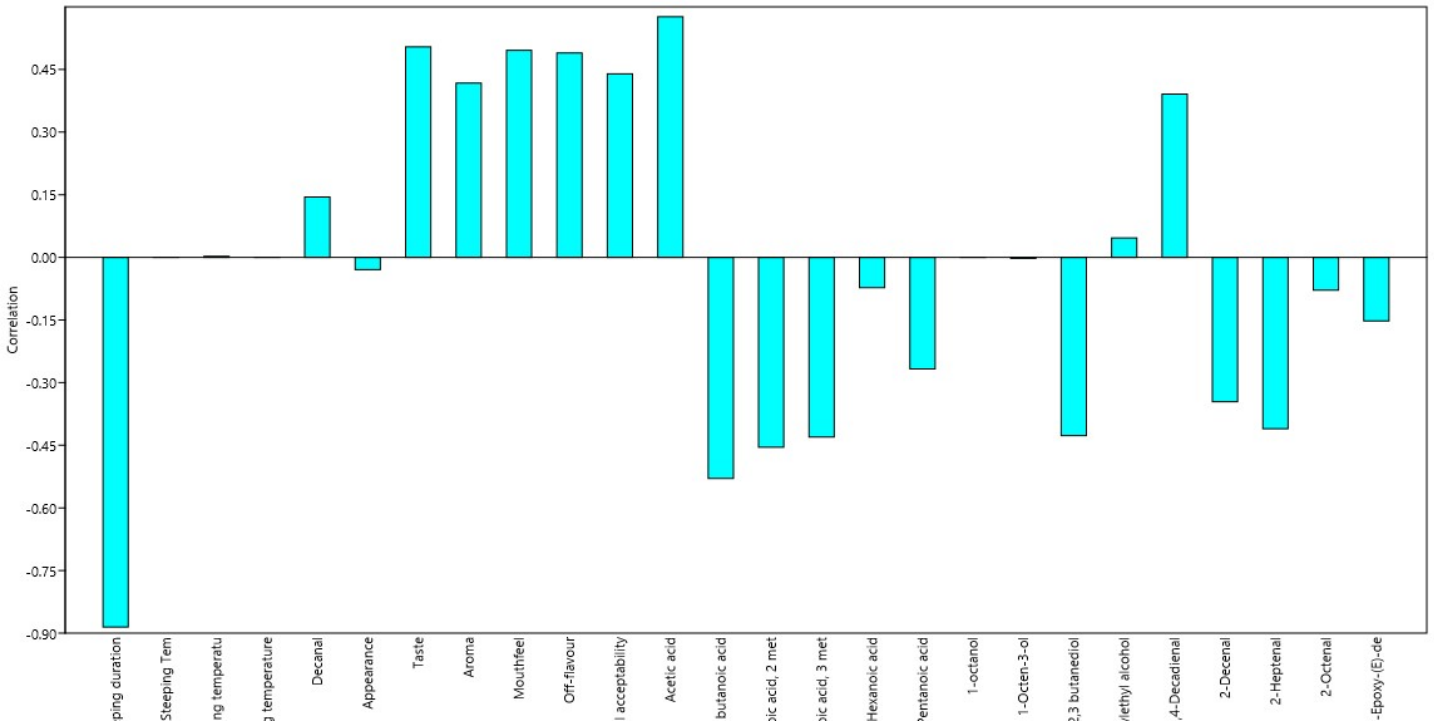
Loading plot for principal component 1



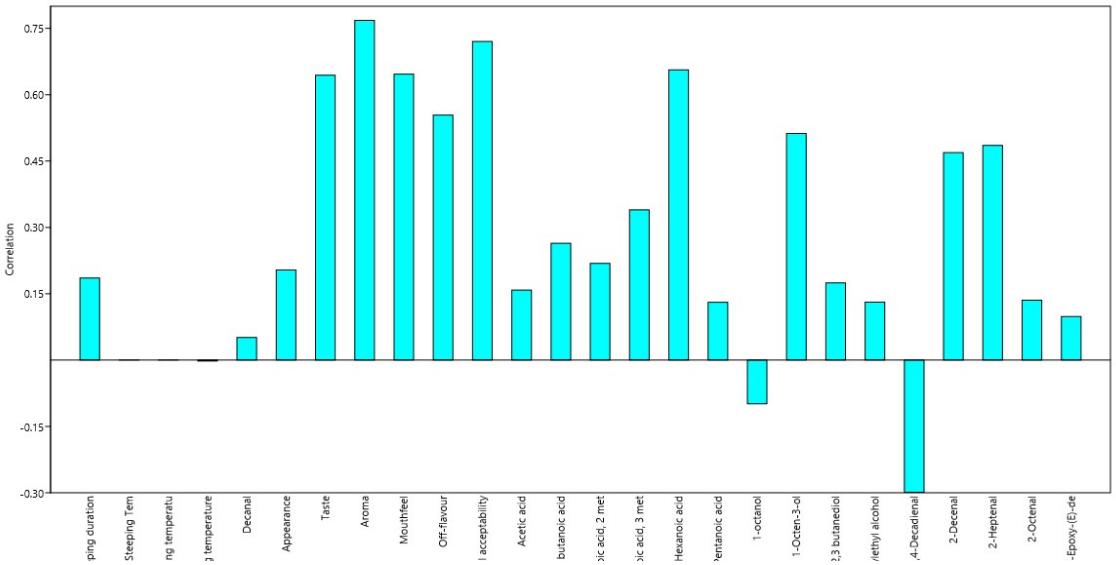
Loadings plot for principal component 2



Loadings plot for principal component 3



Loadings plot for Principal component 4



Loadings plot for principal component 5