

## Identification and characterization of the percentage chemical compositions present in oil extracts from two pumpkin cultivars using GCMSD

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**Abstract:** Gas Chromatography Mass Spectrometry Detector (GCMSD) is a method used for the identification and characterization of the chemical constituents/ composition of the oil seeds so as to know the benefit of the oil seeds usage in the area of human consumption and production of soaps, lubricant and paint. This study focused on the identification and characterization of the chemical compositions (using Gas Chromatography Mass Spectrometer Detector) present in oil extracts from two pumpkin cultivars- Ugba (*Telfairiapedata*) and Ugwu (*Telfairia occidentalis*) using two methods, soaking and soxhlet apparatus. The solvent used for the soxhlet extraction was petroleum ether. The identification and characterization of fatty acid methyl ester (FAME) profile was determined by using GCMS. The transesterification process was done using 10g of methanol with 1g of KOH with the two cultivars. The results revealed higher percentage of Methylpalmitate (Palmitic Acid) and 9, 12-octadecadienoic acid (Z,Z) (Linoleic Acid) and Methyl Ester (Stearic Acid) are present in all samples. These compounds are useful in ceramic, cosmetics, paint and health sector.

**Keywords:** Characterization, Spectrometer, Methyl ester, Octadecadienoic, Stearic.

### 1. INTRODUCTION

Plants are recognized for their ability to produce a wealth of secondary metabolites (Cragg *et al.*, 1999). Many of these natural products have been shown to present interesting biological and pharmacological activities and are used as chemotherapeutic agents or serve as the starting point in the development of modern medicines (Verpoorte, 1998). Nigeria is blessed with many medicinal plants whose roots, barks, seeds and leaves are used for the treatment of different diseases. Vegetables are the fresh and edible portions of herbaceous plants, which can be eaten raw, or cooked (Fayemi, 1999; Dhellot *et al.*, 2006) Hassan *et al.*, 2007). Vegetables are valuable in maintaining alkaline reserve of the body. They are valued mainly for their high carbohydrate, vitamin and mineral

contents (Awol, 2014). Vegetables are rich sources of carotene, ascorbic acid, riboflavin, folic acid and minerals like calcium, iron and phosphorus (Fasuyi, 2006). In addition, they contain phyto-chemicals or anti nutrients which reduce their bioavailability (Akindahunsi and Salawu, 2005). According to (Aletor and Adeogun, 1995), some anti nutritional compounds exhibit protective effects, thus making them to serve a dual purpose of reducing some essential nutrients and protecting the body against a number of biochemical, physiological and metabolic disorders. Vegetables may be edible roots, stems, leaves, fruits or seed. Each group contributes to diet in its own way (Chinma and Igyor, 2007). However, there are some inexpensive leafy vegetables whose

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nutritive and anti-nutritive potentials are yet to be adequately studied and utilized. Among these leafy vegetables are the pumpkins.

Fluted pumpkin (*Telfairia occidentalis*) is a tropical plant grown in West Africa as a leafy vegetable and for its edible seeds. The plant is a drought-tolerant, dioecious perennial that is usually grown as trellised (Agatemor, 2005). Fluted pumpkin (*Telfairia occidentalis*) is a tropical crop that belongs to the Cucurbitaceae family (Egbekun *et al.*, 1998). It is reported to be indigenous to the West tropical rain forest areas of Nigeria (Giami and Barber, 2004). Integrated Taxonomy Information System gave the taxonomic hierarchy of *Telfairia occidentalis* as follows: **Kingdom** (Plantae–plantes, Planta, Vegetal, plants), **Subkingdom** (Viridiaeplantae – green plants), **Infrakingdom** (Streptophyta – land plants), **Division** (Tracheophyta – vascular plants, tracheophytes), **Subdivision** (Spermatophytina – spermatophytes, seed plants, phanérogames), **Infradivision** (Angiospermae – flowering plants, angiosperms, plantas com flor, angiosperma, plantes à fleurs, angiospermes, plantes à fruits), **Class** (Magnoliopsida), **Superorder** (Rosanae), **Order** (Cucurbitales), **Family** (Cucurbitaceae – gourds, squashes, citrouilles, gourdes), **Genus** (*Telfairia* Hook.), **Species** (*Telfairia occidentalis* Hook. f.–oysternut; *Telfairia pedata*). **Taxonomic Serial No.:** 505897 (ITIS, 2013). Fluted pumpkin (*Telfairia occidentalis* HOOK. F., Family: Cucurbitaceae) is a creeping vegetative shrub that spreads low across the ground with large lobed leaves, and long twisting tendrils (Horsfall and Spiff, 2005). It probably originated from the south eastern Nigeria, and is widely distributed among the Igbo speaking people, particularly around Imo state, Nigeria (Akoroda, 1990), where it has the widest diversity (variation in pod and seed colour, seed and plant vigour, anthocyanin content of leaves and petioles or shoots, leaf size and their succulence, dioecious or monoecious plants) (Chweya and Eyzaguirre, 1999 ; Chihande *et al.*, 1997).

There are two main varieties in Nigeria: Ugu-ala (succulent, broad leaves, small black seeds about 12 g, a thick vine and slow growth); Ugu-elu (high growth rate, large brown coloured seeds of 20 g or more, fast emergence, thin stems and small leaves) (Chweya and Eyzaguirre, 1999). Harvesting of fluted pumpkin takes place between 120-150 days, after sowing. The seed contains 13% oil (Okoli *et al.*, 1998) and is used for cooking (Horsfall and Spiff, 2005), marmalade manufacturing (Egbekun *et al.*, 1998) and cookie formulations (Giami and Barber, 2004).

Fluted pumpkin seed oil (FPSO) has been reported to possess some essential properties (vitamin A, tannins, linoleic acid, oleic acid and alkaloids) which suppress lipid peroxidation, hence, improving testicular function (Akintayo, 1997; Bensoussan *et al.*, 1998; Pearson, 1976). The seed contains oil which is used for cooking (Okoli *et al.*, 1998; Horsfall and Spiff, 2005). The oil has saponification value that is beyond the range for most oils of plants origin and less than that of palm oil. Meaning that, the oil has larger molecular weight than the common oils (Pearson, 1976). It also has high iodine value compared to palm oil, indicating that it has high content of unsaturated fatty acids relative to palm oil. It may be used for cooking or manufacturing of margarine. It has a high specific gravity compared to commonly known vegetable oil. Its low acid value also indicates that the oil is edible (Agatemor, 2005). The main applications of mechanical expression are in the extraction of oils and juices. Expression is often combined with size reduction to maximize the yield of product. Components are extracted from plant parts either for direct use or for use in subsequent processing such as refining. In oil-bearing seeds, the oil is found inside cells in small droplets (10–80 µm) in diameter (Fellows, 1998). However, a single type of equipment is not suited to all oilseeds owing to variation in oil content, moisture content, porosity and solidity of the material, applied pressure, heating temperature, heating duration, particle size and shape, storage and handling practices, and the proportions of hulls in different oilseeds are factors influencing yield and quality of vegetable oil expressed (Weiss, 2000).

Expression is achieved either in two stages (size reduction to produce pulp or slurry, followed by separation in a press) or in a single stage, which both rupture the cells and express the oil. In general, the single-stage operation is more economical, permits higher throughputs and has lower capital and operational cost but not suitable for hard nuts as the two stages of expression are more effective. The degree of effectiveness varies with the kind of oilseed and method of oil expression (Akinoso, 2006). (Akubugwo and Ugbogu, 2007) and other numerous researchers have carried out a lot of analytical works on the seed primarily because of extensive and increasing demands for them both for human consumption and for numerous industrial applications. While the proximate analyses to estimate the oil content and other food values of the seed preceded the work of parameter effect on the oil yield, the physico-chemical analysis of the extracted

oil was carried out later as a guide to the uses and applications of the oil. Many researchers in the past have worked in this area of the effects of process conditions on the yield of oil from local seeds (Igwe and Mbah, 2011).

Gas chromatography–mass spectrometry (GC-MS) is an [analytical](#) method that combines the features of [gas-chromatograph](#) and [mass spectrometry](#) to identify different substances within a test sample. Gas chromatography mass spectrometry (GC/MS) is an instrumental technique, comprising a gas chromatograph (GC) coupled to a mass spectrometer (MS), by which complex mixtures of chemicals may be separated, identified and quantified. This makes it ideal for the analysis of the hundreds of relatively low molecular weight compounds found in environmental materials. In order for a compound to be analysed by GC/MS it must be sufficiently volatile and thermally stable. In addition, functionalised compounds may require chemical modification (derivatization), prior to analysis, to eliminate undesirable adsorption effects that would otherwise affect the quality of the data obtained. Samples are usually analysed as organic solutions consequently materials of interest (e.g. soils, sediments, tissues, etc.) need to be solvent extracted and the extract subjected to various 'wet chemical' techniques before GC/MS analysis is possible. Applications of GC-MS include [drug](#) detection, [fire](#) investigation, environmental analysis, [explosives](#) investigation, and identification of unknown samples. GC-MS can also be used in airport security to detect substances in luggage or on human beings. Additionally, it can identify [trace elements](#) in materials that were previously thought to have disintegrated beyond identification. GC-MS has been widely heralded as a "[gold standard](#)" for [forensic](#) substance identification because it is used to perform a specific test. A specific test positively identifies the actual presence of a particular substance in a given sample. A non-specific test merely indicates that a substance falls into a category of substances. Although a non-specific test could statistically suggest the identity of the substance, this could lead to [false positive](#) identification.

Thus, this present research work examines the percentage oil yields as well as identifying and characterizing the percentage chemical compositions using GCMSD present in oil extract from two pumpkin cultivars, fluted gourd (*Telfairia occidentalis*) and oysternut (*Telfairia pedata*) using

soxhlet apparatus and soaking method with the view of knowing the benefit of the oil seeds usage in the area of human consumption, production of cosmetics, lubricant and paint.

## 2. MATERIALS AND METHODS

### 2.1 Collection of Seed Samples

The fresh Fluted Pumpkin pods (Ugba and Ugwu) were gotten from a market in Kwara State. The pods were opened to remove the seeds; the seeds were sun dried for 24 hours. The seeds were then pounded and sieved with a 2mm sieve.

### 2.2 Extraction of Oil and Solvent Extraction

The Extraction of Oil was done by two methods namely; Soxhlet apparatus and soaking method.

The Solvent extraction was done using the soxhlet apparatus in the chemical engineering laboratory. 200ml of petroleum ether was put in the conical flask, 100g of the seed was weighed after grounding and put in a muslin cloth and the muslin cloth placed in the extraction tube, then the extraction tube fixed into the conical flask and the condenser tube was also fixed into the condenser tube. The whole set up then placed in the heating mantle and left to run for two (2) hours at a temperature of 60°C. After the oil was obtained, the conical flask was placed in an oven dryer at a temperature of 70°C and weighed at an interval of 3 minutes until constant weight was achieved.

### 2.3 Procedure for Soaking Method

The seeds from which the oil was to be extracted were first weighed. 100g of the seed was weighed for size reduction using a blender. 200ml of petroleum ether was measured into a plastic container, the ground seeds sample was poured into the petroleum ether, and the container covered tightly to avoid escape of the petroleum ether. The mixture was shaken at an interval of 4 hours and left for 72 hours (3 days). The mixture was filtered using filter paper, separating funnel and a flask. The filtrate was heated to obtain the oil with the solvent (petroleum ether) heated off in an oven dryer at a temperature of 70°C. The procedure was repeated for each of the varieties.

### 2.4 Determination of Percentage Chemical Compounds in the Oil Seeds

In the determination of Chemical Compounds, Gas Chromatography Mass Spectrometry Detector (GCMSD) is used. The need to carry out Transesterification process on the Oil is necessary for the GCMSD to be able to identify the compounds.

### 2.5 Procedure for Transesterification

3.7g of the oil was put in a conical flask and 0.04g of KOH was weighed and added to the oil, 5ml of

methanol was also added and mixed thoroughly. The magnetic stirrer was then put inside the flask and the flask placed on the hot plate for 30 minutes to transesterify. The Biodiesel was then taken to the GCMSD for further analysis of the Oil's Chemical Composition.

Plates 1 and 2 show the Gas chromatography mass spectrometer detector and Transesterification set up respectively.



Plate 1: Gas Chromatography Mass Spectrometer Detector



Plate 2: Transesterification Set Up

### 3. RESULTS AND DISCUSSIONS

Tables 1- 4 show the result for the oil analysis using GCMSD. The result revealed that higher percentage of Methylpalmitate (Palmitic Acid) and 9, 12-octadecadienoic acid (Z,Z) (Linoleic Acid) and Methyl Ester (Stearic Acid) are present in all samples.

**Table 1. GCMS Analysis of Ugwu (*Telfairia occidentalis*) Seed Oil for Soaking Method**

S/No.	Suggested Compound	Time	Percentage
1	Methyl Palmitate	31.90	14.85
2	9,12-octadecadienoic acid (Z,Z)-, methyl ester	35.11	36.40
3	Methyl Stearate	35.68	17.53
4	Methyl 9- Cis-, 11. Trans. t, 13. trans-octadecatrienoate	37.54	16.80
5	Bicyclo-[10.1.0] tridec-1-ene	37.94	1.34
6	7-Tetradecyne	38.04	2.90
7	Oxiraneoctanoic acid, 3-octyl-, methyl ester	38.33	4.58
8	Squalene	46.26	3.11

**Table 2. GCMS Analysis of Ugba (*Telfairia pedata*) Seed Oil for Soaking Method**

S/No.	Suggested Compound	Time	Percentage
1	Methyl Palmitate	31.88	12.91
2	9,12-octadecadienoic acid (Z,Z)-, methyl ester	35.04	31.99
3	Methyl Stearate	35.63	12.95
4	Methyl 9- cis-, 11. trans. 1, 13. trans-octadecatrienoate	37.65	40.90
5	Squalene	46.27	1.25

**Table 3. GCMS Analysis of Ugwu (*Telfairia occidentalis*) Seed Oil for Soxhlet Apparatus**

S/No.	Suggested Compound	Time	Percentage
1	Hexadecanoic acid, methyl ester	31.86	10.63
2	9,12-octadecadienoic acid, methyl ester (E,E)	34.95	20.010
3	9-octadecenoic acid (Z), methyl ester	35.09	33.36
4	Methyl Stearate	35.58	10.81
5	-9-octadecenoic acid (E)-oleic acid	35.83	3.07
6	Methyl 9-cis., 11 trans. T,13,trans Octadecatrienoate	37.46	11.22
7	Cis-13-octadecenoic acid	41.21	1.75
8	-9-octadecenal, (Z) -	44.38	7.20

**Table 4. GCMS Analysis of Ugba (*Telfairia pedata*) Seed Oil for Soxhlet Apparatus**

S/No.	Suggested Compound	Time	Percentage
1	Methyl Palmitate	31.89	13.94
2	9,12-octadecadienoic acid (Z,Z)-, methyl ester	35.09	30.43
3	Methyl Stearate	35.66	13.59
4	Methyl 9- cis-, 11. trans. 13. trans-octadecatrienoate	37.65	39.39
5	Squalene	46.26	2.63

The oil samples are edible due to the presence of palmitic acid that helps to reduce the risk of developing cardiovascular diseases (WHO, 2003). Also, the high percentage in linoleic acid finds application in making of quick-drying oils which are useful in oil paints and varnishes. Furthermore, the compound also finds popular in the beauty products industry because of its beneficial properties to the skin. In addition, the compound has anti-inflammatory and moisture retentive properties when applied on the skin (Dizel *et al.*, 1993; Letawe *et al.*, 1998; Darmstadt *et al.*, 2002). Moreso, the stearic acid has its application as common lubricant during injection moulding and pressing of ceramics (Tsenga *et al.*, 1999). Stearic acid also finds application clinical and epidemiological studies and lower LDL cholesterol (Hunter *et al.*, 2009).

#### 4. CONCLUSIONS

From the analysis drawn out from the two varieties of the fluted pumpkin seed oil, the following results were obtained:

1. For the GCMS analysis for the soaking method, the highest composition was observed to be Ugwu 36.4% of 9,12-octadecadienoic acid (Z,Z), methyl ester and Ugba 40.90% of methyl 9-cis-11trans 13.trans–Octadecatrienoate.
2. However for the GCMS analysis for the soxhlet apparatus, the highest composition was observed to be Ugwu 33.36% of 9-octadecanoic acid (Z) methyl ester and Ugba 39.39% of methyl 9-cis-11 trans 13.trans-octadecatrienoate.

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