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**PROXIMATE COMPOSITIONS AND OLEORESIN
CONTENTS OF FIVE NIGERIAN INDIGENOUS SPICES**

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ABSTRACT

A study of proximate compositions and oleoresin contents of five Nigerian indigenous spices, namely African Black Pepper, African Nutmeg, Alligator Pepper, Negro Pepper and Pepper Fruit, was carried out. The proximate compositions were determined using the standard AOAC method. The oleoresins were extracted using four different standard solvents: acetone, diethyl ether, ethanol and hexane, and the oleoresin contents determined by weighing. The results of the proximate analyses showed that Negro pepper had the highest moisture content of 12.45% while African black pepper had the lowest (9.88%). The fat content ranged from 4.64% for Pepper fruit to 10.87% for African black pepper. Protein ranged from 8.47% Pepper fruit to 12.94 African nutmeg; Ash 2.73 for African nutmeg to 8.95 African black pepper; crude fibre 6.24% African nutmeg to 34.91 Pepper fruit while carbohydrate ranged from 34.70% Pepper fruit to 58.03% African nutmeg. The oleoresin contents were in the decreasing order of Negro pepper (11.24%), Alligator pepper (10.81%), African nutmeg (9.41%), Pepper fruit (8.68%) and African black pepper (7.83%). Diethyl ether proved to be the most suitable solvent in oleoresin extraction.

Keywords: Proximate Analysis, compositions, oleoresin contents, indigenous spices, solvents

INTRODUCTION

Flavour is one of the most important attributes of food and is detected by the senses of taste and smell. Flavouring agents have been used from earliest times to improve the attractiveness and taste of food. These agents include both natural and synthetic flavouring substances such as herbs and spices.^{1,2}

Spices have been defined as aromatic vegetable products of tropical origin that are used in crushed or pulverized state, primarily for seasoning, garnishing foods and beverages (Kochhar, 1986). Spices can also be defined as aromatic flowers, bark of tropical plants or other plant parts which can be used in foods, medicine, embalming, perfumes and as incense. The word “spice” is derived from Latin “specie ‘aromatic’”, meaning, “aromatic fruits of earth.”³ The United States Drug and Food Administration (USDA) describes a spice as “an aromatic vegetable substance in whole; broken or ground form whose function is for seasoning rather than nutritional”.⁴

The essential difference between herbs and spices is that a herb is a whole plant, used for medicine or flavouring food whereas a spice is any aromatic pungent part of a plant (be it leaf, fruit, seed, flowers, stem or root, either in natural or powdered form) used for flavouring food.

A “seasoning” is also differentiated from a spice in that the former is any substance, whether natural or synthetic, which is used in flavouring foods. “Condiments”, on the other hand, are other flavorings usually added to food after cooking.

The story of spices, condiments and other flavoring materials is a fascinating story in the history of vegetable products as they are connected with many important events such as international trade. Spices were among the first items of trade between the East and the West.⁵ There are about 70 species of spices cultivated in different parts of the world⁶; examples are African black pepper (Uziza), Ginger, Garlic, Turmeric, Onion, Alligator pepper, Scent leaf (Nchuanwu), Clove, Nutmeg and Cinnamon.

The five spices under study are indigenous to Nigeria and they are African black pepper, African nutmeg, alligator pepper, negro pepper and pepper fruit. African black pepper (*Piper guineense*) is severally referred to as Ashanti pepper, West African pepper, Benin pepper Guinea pepper and false cubeb. The plant is a perennial woody vine belonging to the family “Piperaceae”. It is a close relative of cubeb pepper but the fruit is smaller than that of cubeb pepper; it is also elliptical whereas cubeb pepper fruit is spherical. Both the fruit and the leaves of African black pepper are used as spice but it is the fruit that is used in this study. African nutmeg (*Monodora; myristica*) commonly known as calabash nutmeg, Jamaican nutmeg and African orchid nutmeg is a tropical shrub of the “Annonaceae” or “Custard apple” family. The fruit is a nearly spherical drup about 20cm in diameter containing numerous oblongoid pale brown aromatic seeds embedded in a yellow pulp. Each seed measures about 1.5cm in diameter. The seed is called “Ehuru” or “Ehiri” in Igbo, Arineol in Yoruba and Jedemia in Hausa. The odour and taste of African nutmeg are like those of “nutmeg” and hence, the name.

Alligator pepper (*Aframomum melegueta*) also known as “mbongo spice, hepper pepper, grains of paradise or guinea grains belongs to the family of “Zingiberaceae” or Ginger family. It is known as “ose oji” or “orewa” in Igbo, “Atare” in Yoruba and “chila” or “Ehila” in Hausa. Alligator plant is a perennial herb commonly found in tropical regions, particularly in West Africa. The seeds which are used as the spice are borne in 5-7cm long grayish-brown pods or capsules which are oval in shape.⁷ Each seed which measures 3-4mm in diameter is reddish brown in colour and has a pungent aroma.⁸ Negro pepper refers to the seed of a shrubby tree, *Xylophia aethiopica*, found in Africa. The tree is 8-10m high with a short trunk branching almost from the base with relatively dense crown.⁹ Negro pepper is severally referred to as African pepper, killi pepper, Guinea pepper, Ethiopian pepper or grains of salim. It is known as “uda” in Igbo, “Eeru” in Yoruba and “Kimba” in Hausa. It belongs to the family Annonaceae. The fruits of *X. aethiopica* which grow in clusters, cylindrical and dark brown in colour measure 2.5 to 5.0cm in length and 4 to 6mm in thickness. Each pod

contains 5 to 8 kidney-shaped seeds that are approximately 5mm in length. The plant parts used as spice are primarily the fruit and the bark.

Pepper fruit is the fruit of the plant *Dennittia tripetala*. *D. tripetala* is a woody plant of at least 2m high. It also belongs to the family of "Annonaceae" *D. tripetala* is known as "Mmimi" in Igbo, "Ata" in Yoruba, "Nkarika" by the Ibibios and Efiks, and "Imako" by the urhobos. The plant can grow up to a height of 18m and is about 60cm in girth. It is found in Ivory Coast, southern Nigeria and Western Cameroon.¹⁰ The parts of the plant used as spice are the leaves, fruits, seeds, roots and stem.¹¹ However, the fruits are the parts used for this study.

Oleoresins are substances responsible for the flavour and aroma characteristics of spices. An oleoresin is made of two parts: the volatile component called the "essential oil" or "volatile oil" and non volatile component, the "resin". The aim of this study is to determine the proximate compositions and the oleoresin contents of five Nigerian indigenous spices, African black pepper, African nutmeg, Alligator pepper, Negro pepper and Pepper fruit.

MATERIALS AND METHODS

The five Nigerian indigenous spices namely African black pepper (AB) fruit, African nutmeg (AN) seeds, Alligator pepper (AP) seeds, Negro pepper (NP) fruit and pepper fruit, (PF) were purchased from markets in Owerri, Imo state, Nigeria. They were sorted, cleaned, ground and stored in polythen sacks ready for use.

Proximate Analyses

Moisture content was determined by measuring directly with sartorius moisture analyzer model MA35 to take care of the volatiles in the spices. The fat, protein ash, crude fibre and carbohydrate contents were determined using the standard AOAC (2005) methods.¹²

Oleoresin Content Determination

Oleoresin was extracted from each of the spices by the Soxhlet extraction method using separately 4 different solvents, namely (i) acetone, b.p. 56°C (ii) Hexane, bp. 69°C (iii) Diethyl ether b.p. 35°C and (iv) Ethanol b.p. 79°C. A 350g amount of each ground spice was weighed into a thimble and loaded into the main chamber of the Soxhlet extractor. Each solvent (400mls) was measured into the round bottom boiling flask. The whole apparatus was connected, including the mantle and the reflux condenser. The extraction process was carried out for 6-8 hours at a condensation rate of at least 3-6 drops per second. At the end of the process the solvent, together with the extracted oleoresin, was poured into a beaker. The diethyl ether was allowed to evaporate at room temperature for 24 hours, leaving the oleoresin inside the beaker. However, for acetone, ethanol and hexane, the evaporation was effected by the use of a rotary evaporator. Percentage oleoresin was calculated based on the masses of the spice used and the oleoresin extracted.

RESULTS

Proximate Compositions of the Spices

The results of the proximate analyses of the five spices are presented in Table I.

Table I: Proximate compositions (%) of the five spices

	SPICES				
	AB	AN	AP	NP	PF
Moisture	12.16±0.39 ^a	10.39±0.40 ^a	9.88±0.63 ^a	12.45±0.52 ^a	11.56±0.12 ^a
Fat	10.87±1.29 ^a	9.67±0.75 ^a	7.86±0.06 ^a	6.93±0.48 ^a	4.64±1.50 ^a
Crude Protein	11.96±0.18 ^a	12.94±0.62 ^a	12.24±0.30 ^a	12.11±0.25 ^a	8.47±1.38 ^a
Ash	8.95±1.66 ^a	2.73±1.12 ^a	4.12±0.50 ^a	4.63±0.27 ^a	5.72±0.22 ^a
Crude Fibre	20.60±0.05 ^b	6.24±6.48 ^c	21.41±0.31 ^b	20.45±0.12 ^b	34.91±6.35 ^a
Carbohydrate LSD = 12.05	35.46±3.46 ^b	58.03±6.63 ^a	44.39±0.53 ^b	43.43±0.10 ^b	34.70±80 ^b

Values are mean ± SD

Key: AB = African black pepper
AN = African nutmeg
AP = Alligator Pepper
NP = Negro pepper
PF = Pepper fruit

Values bearing different superscripts along the same row are significantly different from each other (p<0.05).

Oleoresin Contents of the Spices

The results of the oleoresin determination are presented in Tables II and III.

Table II: Oleoresin yields (%) from the 5 spices

SOLVENTS	SPICES				
	AB	AN	AP	NP	PF
Acetone	7.20±0.81 ^b	9.28±0.12 ^{ab}	10.47±0.65 ^a	11.25±0.67 ^a	7.26±0.78 ^b
Diethyl Ether	7.83±0.95 ^b	9.41±0.24 ^{ab}	10.81±0.38 ^a	11.24±0.58 ^a	8.68±0.57 ^b
Ethanol	5.29±1.19 ^b	8.78±0.38 ^a	9.86±0.86 ^a	7.88±0.03 ^a	7.89±0.02 ^a
Hexane	4.18±1.21 ^c	7.40±0.23 ^b	6.28±0.27 ^b	9.81±1.31 ^a	6.74±0.06 ^b
Means LSD = 2.206	6.125 ^c	8.718 ^{ab}	9.355 ^{ab}	10.045 ^a	7.643 ^{bc}

Values are mean ± SD

Key: AB = African black pepper
AN = African nutmeg
AP = Alligator Pepper
NP = Negro pepper
PF = Pepper fruit

Values bearing different superscripts along the same row are significantly different from each other (P<0.05).

Table III: Oleoresin (%) as extracted by different solvents

	SOLVENTS		SPICES			MEANS
	AB	AN	AP	NP	PF	
Acetone	7.20±0.54 ^a	9.28±0.28 ^a	10.47±0.56 ^a	11.25±0.60 ^a	7.26±0.19 ^a	9.012 ^{ab}
Diethyl Ether	7.83±0.85 ^a	9.41±0.64 ^a	10.81±0.73 ^a	11.24±0.60 ^a	8.68±0.52 ^a	9.954 ^a
Ethanol	5.29±0.42 ^b	8.78±0.03 ^{ba}	9.86±0.25 ^a	7.88±0.08 ^b	7.89±0.12 ^a	7.940 ^{bc}
Hexane	4.18±0.97 ^b	7.40±0.66 ^b	6.28±1.54 ^b	9.81±0.12 ^a	6.74±0.45 ^{ba}	6.882 ^c

LSD = 1.838

Values are mean ± SD

Key: AB = African black pepper
AN = African nutmeg
AP = Alligator Pepper
NP = Negro pepper
PF = Pepper fruit

Values bearing different superscripts within the same column are significantly different from each other (P<0.05).

DISCUSSION

Proximate Composition of the Spices

The results of the proximate analysis of the spices showed that they contained an average moisture content of 11.3% with negro pepper having the highest yield (12.45%) while alligator pepper had the least value (9.88%). African black pepper contained the highest amount of fat (10.87%) while pepper fruit contained the least amount (4.64%). African nutmeg (AN) contained the highest amount of protein (12.94%) while pepper fruit (PF) also contained the least (8.47%). African Black pepper contained the highest amount of ash (8.75%) while African Nutmeg contained the least (6.24%). African Nutmeg contained the highest percentage of carbohydrate (58.03%) while African Black pepper contained the least (3.546%). Pepper Fruit had the highest amount of fibre (34.91%) followed by Alligator Pepper (21.41%), then African Black pepper (20.60%) closely followed by Negro Pepper (20.45%). African Nutmeg had small amount of fibre (6.24%).

The values are in agreement with results obtained by Achinewhu *et.al.*¹³ which showed that spices and herbs contain 4.6 to 22.1% protein, 2.5 to 36.0% fat and 34.6 to 71.8% total carbohydrate. The highest amount of nutrients contained by the spices is carbohydrate (35.46-58.03%) while the least is ash (2.73-8.95%).

Analysis of variance shows that at 5% level of probability, significant difference did not exist in the moisture, fat, protein and ash contents of the spices. Significant difference also did not exist ($p \leq 0.05$), in the fibre contents of African black pepper, Alligator pepper and Negro pepper; however significant difference existed between this group, African nutmeg and Pepper fruit.

Oleoresin Yields from the Spices

Tables II and III give the results of oleoresin extraction from each of the spices: Table II highlights the amount of oleoresin extracted from each spice while Table III highlights the quantity of oleoresin extracted with each of the 4 solvents.

According to Table II, Negro pepper gave the highest mean oleoresin yield of 10.045%, followed by Alligator pepper (9.355%), then African nutmeg (8.718%), pepper fruit (7.643%), while African black pepper gave the least mean yield of 6.125%. Analysis of variance showed that there was no significant difference in oleoresin contents of African nutmeg, pepper fruit and African black pepper ($p \leq 0.05$) as extracted with acetone. There was the same trend in oleoresin contents as extracted with diethyl ether. There was significant difference in oleoresin yields by African black pepper and those of the other four spices as extracted with ethanol. As for the extraction with hexane, there were significant differences except between alligator pepper, pepper fruit and African nutmeg.

On the average, there was no significant difference in oleoresin yields of Negro pepper, Alligator pepper and African nutmeg, and between pepper fruit and African black pepper.

In general, the oleoresin yields by the spices are within the range expected of spices (1.0% to 20.0%), according to Hulse.

Table III shows the oleoresin yield as extracted with each solvent. Diethyl ether gave the highest mean oleoresin yield (9.954%), followed by acetone (9.012%); ethanol with 7.954% mean yield while hexane gave the lowest mean yield (6.882%). Analysis of variance showed that there was no significant difference in oleoresin yield with acetone and diethyl ether for African black pepper, African nutmeg, alligator pepper and negro pepper, where as with ethanol and hexane, there were significant differences ($P \leq 0.05$). On the average, there was no significant difference in oleoresin yield as extracted with acetone and diethyl ether, and with ethanol and hexane. Diethyl ether proved to be the best solvent for extraction of the oleoresins. It has the lowest boiling point among the four solvents (35°C), while the boiling points of the other three are 56°C (acetone) 79°C (ethanol) and 69°C (hexane).

Extraction with diethyl ether was at room temperature which must have given an added advantage that much of the volatile components were not lost to the atmosphere during the evaporation stage of the extraction.

RECOMMENDATIONS

There is need to investigate the proximate compositions and oleoresin contents of abounding spices other than the five worked upon in this study, both indigenous and non-indigenous. Furthermore, further research is recommended to find out the percentage of the oleoresin which is volatile oil and which percentage is resin in each case.

CONCLUSION

The study has demonstrated the fact that, apart from flavour impartation of spices to foods, some vital nutrients can still be obtained from them. Alligator pepper and pepper fruit are rich in crude fibre which provides roughage and lower cholesterol level in the blood, incidence of colon cancer and insulin requirements of diabetes. The oleoresin contents of the five spices in decreasing order of magnitude are Negro pepper > Alligator pepper > African nutmeg > African black pepper > pepper fruit. This shows the trend of flavour impartation of the spices in food.

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