PROXIMATE ANALYSIS OF GUNA SEED OIL (CITRILLUS VULGARIS) PRODUCED IN GA'ANDA TOWN GOMBI LOCAL GOVERNMENT, ADAMAWA STATE NIGERIA

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ABSTRACT

Guna seed oil was extracted through solvent extraction method. Percentage oil was found to be 57.40%. Saponification value was found to be 112mg/L. Iodine value was also calculated to be 10.16. Free fatty acid was also found to be 5.6%. Peroxide value was calculated to be 7.27mg/kg. Protein content was calculated to be 61%. Moisture content was calculated to be 5.00. More so, the total ash content was found to be 0.73. the crude fiber was found to be 6.67. Crude fats were found to be 12.00 and lastly, Carbohydrate content of guna seed oil was found to be 14.60%. All calculations ware done in accordance with the by the A.O.A.C. 1990,official methods of analysis. Washington DC.

KEYWORDS: *organic fats, organic compounds, animals, vegetables, plants*

INTRODUCTION
OIL:

Any greasy, viscous, flammable or non-flammable substance, liquid at room temperature and insoluble in water but soluble in organic compounds that is derived from plants, animals, or mineral deposit or manufactured artificially and use for food, lubricant or as fuel (Chambers 1996).

Various organic fats are essential constituents of plants and animals life. Every specie of plants and animals produce some quantity of fats and oil during its life cycle. However, only relatively few plants and animals fats and oil are produced in sufficient quantities to become an article of commerce. The most important oil therefore are those that can be obtained in commercial quantity from readily available raw materials (Roslla., 2011).

Sometimes, the terms 'fat' and 'oil' are used interchangeably , but more often, the term fat referred to those fatty materials that are solid at room temperature while oil are liquid under the same condition.

However, some oils such as coconut oil are quiet solid at room temperature. Unlike petroleum or other oil of minerals origin, all animals , vegetables and marine oils are triglyceride or esters of fatty acid, ie, they are chemical combination, of one molecule of glycerol with three molecules of fatty acids. When processes with caustic alkalis, which are salt of fatty acid and are glycerol which is a trihydric

alcohol (Kostianoy, *et al.,* 2014).

The fatty acid portion of the triglyceride structure represent over 92% of the total weight, the balance consisting of the glycerol radical, such as cholesterol, phytosteroids, phosphotides (lecithin), vitamins and water. The total amount of noglycoside components present is usually not over 3% of the weight. Oil and fats has tendency to dry or solidify on exposure to air. The drying is caused by oxidation or chemical combination with oxygen rather than by evaporation of solvents. The rate of drying is measured by number known as iodine number (value). Oils classified as nondrying include coconut, butter, palm oil with iodine numbers from 9- 65. semi- drying oils include olive , peanut, com, cotton seed and sunflower with iodine number between 85-130. Drying oil includes fish oil etc. with iodine number varying from 150 and 200. (Americana Encyclopedia)

OCCURRENCE AND COMPOSITION OF FAT AND OIL

Biochemistry have found it convenient to define one set of bimolecular, the lipids, as substance insoluble in water, that can be extracted from cell by organic solvent at low pH Polarity like ether or chloroform. (Hooper., 2015)

This is a catch of all sort of definition, and lipids including compounds of different kinds. Steroids for example, and trepans of lipids. These compounds are not only important, indeed every compound in organism plays an important role, if only as an available waste product of metabolism, but they are most abundant.

Fats are main constituents of the storage fat cells in animals and plants, and are one of the important food reserves of living organism. We can extract animals and vegetable fat, liquid fat are often referred to as oil.Fats are carboxylic esters derived from the single alcohol, glycerol $HOCH_2CHOHCH_2$ and are known as glycosides, and they are triglycerol (Morison 1987) Fats and waxes belongs to group of natural occurring compounds called lipids (Greek - Lopose = fats) lipids are those constituents of plants/ animals which are soluble in organic solvents such as ether, chloroform, carbon tetrachloride, benzene, hexane but insoluble in water. Lipids which yield fatty acid and alcohol on hydrolysis with aqueous base (saponified) are referred to as simple lipids. These can further be divided as: Fats and oil which yields long chain fatty acids and glycerol upon hydrolysis.Acid waxes, which yields long chain fatly acid and long chain alcohols upon hydrolysis.Fats and oil are the most important lipids found in nature. They are one of the three major factors needed for human body, the other two, being protein and carbohydrates.Fats and oil are widely distributed in tissues, bones and around nervous tissues, kidney, heart and other organs of the body (Human protein, 2017).

MATERIALS AND METHODS
MATERIALS USED:

Sexhlet extraction apparatus:- beakers conical flask, heating mantle, water bath, titration apparatus, measuring cylinder, electric weighing balance, phenolphthalein indicator etc.

SOURCE OF RAW MATERIAL

The Gun a seed was purchased from the main market of GA'ANDA Town of Gombi Local Government area, Adamawa state.

SAMPLE PREPARATION:

The Guna seed was manually cleaned, dried and kept in moderate temperature. The cleaned and well stored seed was well pounded using laboratory pestle and mortal.

WEIGHING AND EXTRACTION OF THE OIL

The modem way of processing vegetable oil is by chemical extraction using solvent extract which produce high yield and is quicker and economical. The solvent used is petroleum derived n-HEXANE and this techniques is use for the most of industrial oil such as soybeans and com oil.(Jif and Crisco, 2002).

Another way is the physical extraction which does not involve the use of a solvent extract. It is made the traditional way using different type of mechanical extraction. This method typically produces more traditional oil e.g olive oil which is preferred by most customers. Other methods of extraction of oil include expeller press, the screw press, Decoction method and the Ram Press .

For the purpose of this study, the soxhlet extractor was used. This is because it produces high yield and it is quicker and more economical. It is only limited to the extraction of lipids required where the desired compound has a limited solubility in a solvent and the impurity is soluble in the solvent (Cumpson and Sano, 2013).

EXTRACTION OF OIL

100ml extraction flask was weighed empty, and l00g of the grinded Guna seed was **folded in a thick filter paper and inserted into an extraction thimble. It was** placed in the soxhlet extractor, the flask was connected to n- hexane 60-80° C was filled into the extractor and the solvent siphoned over and over until it was colorless then the solvent was gently boiled under reflex. At the end, the solvent was evaporated by direct healing on water bath **(69°C), Then,** the flask **containing** the **oil** was even dried 30-40°C for at least 30 minutes. It was allowed to cool and was weighed to find its mass (Jensen, 2007).

DETERMINATION OF PERCENTAGE OIL EXTRACTION

Mass of sample = l00.0g, Mass of empty beaker = 86.0g, Mass of o flask + oil = 142.2
Mass of oil = (Mass of flask + oil) - (mass of empty flask)

= 142.20 -86 = 57.40g

The percentage Gun a seed oil extracted was found to be 57.40%

CHARACTERISTICS OF GUNA SEED OIL
SAPONIFICATION VALLE

Saponification value (SV) relates to the molecular weighted of the number of milligram . 1 gram of oil that will neutralize the free fatty acid Combined as glycerol The saponification equivalent is the amount of oil or fat saponified by one gram (Igm) equivalent of potassium hydroxide and equal to 56108 (sv) (A.O. A C 1990),

PRINCIPLE

Saponification value is determined by complete saponificaton the oil with known amount of KOH, the excess of which is determined by titration.

MATERIALS AND REAGENTS

0.5MHC1,0.5MKOH, **96%** ethanol ,1% solution of phenolphthalein in 96% ethanol, 250ml conical flask, condenser, weighing balance , and heating mantle.

PROCEDURE

0.5g of the oil was weighed into 250ml conical flask fitted with an air condenser. It was dissolved in 10ml alcohol and 10ml of 2.5 KOH Solution. The flask was refluxed on a heating mantle for about 2 hrs, it was cooled and 2 drops of phenolphthalein was added and was titrated with standard 1 m oxalic acid until the pink color disappears The same procedure and the same reagents was performed without the sample to determine the titre value for the blank Weight of oil w= 0.5 g

Volume of 1m oxalic acid for blank V1.= 12.60mls.

Volume of lm oxalic acid for sample V2 = 10.60mls.

Using the formula Sap Value = $\frac{56 (V1 – V2) \times 100}{2000 \times W}$

$= \frac{56 (VI-V2)}{2000 \times 0.5}$

$=112$ mg/L

DETERT AMI NATION IODINE VALUE

I_2+ 2NaS$_2$03 -------------- ► 2 Nal + Na$_2$S40$_6$

The mixture was kept inthedark

METHOD.

2.00g of the oil and 25ml of chloroform was weight into 500ml conical flask. 30ml of wij's solution was also added. (about 75% remain unreacted). The flask was shaked and was allowed in the dark for 20-30 minutes with occasional shaking 30ml 15% KI and 100m H$_2$0 was added. The librated iodine was titrated with standard 0.1m sodium thiosulphate solution using starch as indicator (to find the V$_2$. the experiment for the blank was done by the same procedure (to find the Vi)

Weight of sample w = 2g

Polarity of sodiumthosulphate - = 0.1 m

Volume of sodiumthrosuphate for blank - Vi = 12.00ml

Volume of sodiumthrophate for sample = V$_2$ = 4.00ml

Using the formular: iodine value = $\frac{(V1-V2) \times M \times 127 \times 100}{W \times 1000}$

$- \frac{(12.00-4.00)0.1 \times 127 \times 100}{2.0 \times 1000}$

$= \frac{8 \times 0.1 \times 127}{20}$

$=10.16$

DETERMINATION OF FREE FATTY ACID (PERCENTAGE)

Acid value is expressed as the percentage fatty acid calculated as oleic acid.(A.O. AC 1990)

METHOD AND MATERIALS

2.00g of Gun a seed oil was weighed into 250ml conical flask, 25ml 96% alcohol was added and 1 ml phenolphthalein was added (indicator) and was shaked

The solution was titrated with 0.1M NaOH, with constant shaking until pink color was obtained , but the pink color disappeared in less than 20 seconds.

Acidvalue=titifml)x5,61Wtofpe

F.F.A is usually calculated as oleic acid (Mr. =282g)

lml 0.1M NaOH --------------------------- * 0.028g

Then, 2.0gm lm NaOH _____ $p \times (AV)$

X= 0.028 X 2.00 =0.056 FFA = (A.V) X 2

= 0.056 X 2 =0.112 **%** FFA = FFA X100

$\frac{0.112 \times 100}{2.00}$

=5.6%

DETERMINATION OF PEROXID VALUE

Peroxide value is determined by subjecting potassium iodide at room temperature. Thus the liberated iodine is titrated with sodiumthiosulphate.

METHOD AND MATERIAL

1.10g of Guna seed oil was weighed into 250ml conical flask, 25ml of a solvent (n- Hexane), 1ml 15% KI solution was added and was allowed in the dark for a minute. 35ml of distilled water and 3ml of starch solution was added. The solution was titrated with 0.1M sodium thiosulphate. The same procedure was taken for the blank le without the ample.

The reading was taken for 3 times to obtain die average titre Number of moles of sodiumthiosulphate, N = 0 1 weight of the oil used, w = 1. l0g

volume of solution used for blank, V1 = 7.80

volume of solution used for the sample, V$_2$ = 7.00

Using the formula.

Peroxide value = $100 \frac{(V1 – V2)N}{W}$

$= \frac{100(7.80 - 7.00) 0.1}{0.1}$

$= \frac{100 \times 080 \times 0,1}{1.10}$

$= 7.27$ mg/kg

DETERMINATION OF PROTEIN IN GUNA SEED OIL;

100g of Guna oil was placed in as Kjedahl flask, 25cml Cone. H_2SO_4, 2 5 m l _2m K_2SO_4 and 2m $CuSO_4$ was added The flask was gently heated on a heating mantle in an incline position until the brown color disappeared The flask was cooled and its content diluted with distilled water. The dilute solution was transferred into round bottom Bask and tilted with *a* condenser, the condenser depend in excess 10ml 0.2M H_2SO_4,the liquid heated and titrated The excess acid was determined by titrating the liquid with 0.1M NaOH using phenolphthalein as indicator.

Titre value, 0.35ml = v Weight of sample used, w = 1.0g Using the formula

% Nitrogen = $\underline{14 \times V \times N \times 100}$
                             1000

= $\underline{1.4 \times 0.35 \times 0.2}$
1.00   = 0.098

The value 0.098 is multiplied by 6.25 ie 0.098 x 6.25 = 0.6125

The percentage protein was calculated thus; 61.0%

DETERMINATION OF MOISTURE CONTENT

2.00g of the oil was placed in a watch glass and oven dried for 31/2 hours. It was cooled in a desiccator and weighed the process continued until a constant weigh was obtained. The % moisture was calculated using the formula below:

Weight of sample used , w = 2.00g,
Weight of watch glass 54 .40
Weight of watch glass + oil = 54.80 + 2.00 = 56.40
wight after 1st drying = 56.20.
             2nd drying = 56.10
             3rd drying = 56.10.
             Weight lost= 56.20 - 56.10 = 0 10

% moisture = $\underline{\text{weight lost} \times 100}$
                        Weight of sample 1

= $\underline{0.10}$ x $\underline{100}$
    2.0   1

= 5.00

TOTAL ASH DETERMINATION

15g of Guna seed oil was placed into a clean weighed porceline crucible and was put on a hot plate until a dark mass was obtained. The crucible and its content was cooled in a desiccator's the weight of the reduce was expressed as %ash.

Weight of sample used - w = 1 5g.
Weight of crucible = 54.80.
Weight of crucible + ash = 55.90

% ash = $\underline{\text{weight of crucible + ash - weight crucible x}}$
                    100   Weight of sample
= $\underline{55.90 - 54.80}$
      1.5
= 0.73

DETERMINATION OF CRUDE FIBRE

1.5 g of oil sample was defected with diethyl ether for about 6 hrs. the residue was boiled under reflux for 30 minutes with 200ml of 1.25% $H2SO_4$. It was filtered by a cloth on a funnel. The residue was washed until not longer acidic the residue was boiled again under reflux with 200ml 1.25% NaOH solution for 30 minutes It was filtered with a piece of cloth on a funnel, it was washed with warmed water in a crucible of known 54.80 g and was allowed to cool in a desicator and weighed as the C2 % crude fiber was calculated as

% crude fiber = $\underline{C_2 - C_3}$
                              W

Weight of sample - w = 1.50g
Weight after 1st cooling -= C2 = 56.10
Weight 2nd cooling - C_3 56.00
% crud fibre ,
= $\underline{56.10-56.00 \times 100}$
          1.50
= 6.67

DETERMINATION OF CRUDE FAT

1.5 g Gun a seed oil was placed in a fat extraction finable and covered with cotton wool. It was set a place to the soxhlet apparatus and the fat extracted with petroleum ether for 3-4 hours.

Extracted fat was dried on a heating mantle for about an hour. The flask was then cooled m a desiccator and weighed.

Weight of flask =86.00
Weight of oil = 1.5
The % fat was determined by the formular;

% fat = $\underline{\text{weight of flask + oil - weight of flask x 100}}$
                           Weight of sample

$\underline{86.18 \, 86.00 \times 100}$
          1.50
=12.00

CARBOHYDRATE DETERMINATION

The percentage carbohydrate was determined by subtracting the known i.e- crude protein, crude fat, total ash, crude fibre and moisture content out of 100, the difference **is** the % carbohydrate.

Ie. % CHO = (100 - (Protein + fat + ash + fibre + moisture)
= 100- (61.00 + 12 + 0.73+ 6.67 + 5.00)
= 100 - 85.40 = 14.60
% carbohydrate was found to be 14.60%

Table 1
Characterization of the Guna Seed Oil

S/N	CHARACTER	RESULT
1	Saponification value	112mglL
2.	Iodine value	10.20
3.	Acid value	0.112
4.	Free fatly acid (%)	5.6
5.	*Peroxide* value	7.27 mg/kg
6.	Protein value (%)	61.00
7.	Moisture content	5.00
8.	Ash content	0.73
9.	Crude fibre	6.67
10.	Crude fat	12.00
11.	Carbohydrate content	14.60

CONCLUSION

In conclusion, guna seed (Citrillus Vulgaris) contains high amount/Percentage of oil compared to some seeds, it is found to have very low acid value and so it can hardly be saponified. It is a drying oil that can be used for industrial manufacture of cosmetics, margarine and can as well be used as a large and small scale industrial lubricant.

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