# EXTRACTION AND CHARACTERIZATION OF OLEORESIN FROM GINGER

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# DECLARATION

I,Olawuyi Gbemiga Afolarin (2003/17509EH) herby declare that this research project, "Extraction and characterization of ginger oleoresin" Carried out under the supervision of Engr. Aisha Bawa and presented in partial fulfillment of the award of Bachelor of Engineering (B.Eng) Degree in Chemical Engineering has not been presented for any degree elsewhere, to the best of my knowledge.

_____ _____

**OLAWUYI GBEMIGA AFOLARIN** **DATE**

**2003/17509EH**

# DEDICATION

This project is dedicated to God Almighty.

# ACKNOWLEDGEMENT

Firstly I acknowledge God for his favour mercy, protection and provision of my need. I also knowledge the effort of my parent Mr and Mrs Olawuyi for their moral and financial support. And to my nderful loving brother and sisters

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And to my wonderful loving brother and sisters Modupoe,Bose,Grace and last baby Shola you are ll wonderfull indeed, I love u all.

# ABSTRACT

This project researched into the extraction and characterization of ginger oleoresin using ethanol as solvent. Soxhlet apparatus was used for the extraction. The oil content of ginger was found out to be 10.17%. The oil obtained was subjected to physical and chemical analysis .Physically the oleoresin is dark brown in colour ,has spicy odour ,specific gravity of 1.207 at 29°C ,PH of 6.9 at 29°C , refractive index of 1.447at 29°C. The chemical test reveals that free fatty acid content of oleoresin to be 56.4%, acid value as 1.128mgKOH/g,and iodine value as 71.63mg of KOH The saponification value was found to be 17.95 .The ginger oleoresin obtained from this experiment showed encouraging result and very consistent literature values.

Within experimental limitation all parameter obtained stand fit and showed a practical values base on this tropical condition

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# CHAPTER ONE

## 1.0 INTRODUCTION

Ginger has been consider a very important crop and is seriously valued in some country, such as India, Japan, china, Seria Leon, and Nigeria. This is because ginger an it product have a lot of usefulness and wide area of application .This includes confectionary, pharmaceutical and beverage production (Whilley, 1974).

The demand for the crop and it products at local and international market is so encouraging that it was currently rated the $10^{th}$ most important commodity at the world trade level.(Bee croft,1981).

The ginger oleoresin is an extract from ginger and has received much attention in trade, it has many advantages over the dry spice, it amenable to standardization with respect to both strength and quality and it is free from the bacterial associated with dried ginger .On industrial scale, extraction is carried out either in batch or continuous process, ranging from single cylindrical drum extractor or rotary cell. At laboratory scale, researches have been carried out with equipment designed o suit the purpose of finding out ways of improving the efficiency of extraction process.

Ginger oleoresin is extracted by method of solid-liquid extraction otherwise known as leaching i.e. the separation of one or more soluble constituent from solid by extraction with a solvent. Dried powdered rhizomes are extracted by percolation using ethanol as solvent; the extract is cold-distilled at 45-55°C to remove almost all the solvent. Solvent derived from petrochemicals such as hexane, pentane, di and trichloromethane, acetone cannot be use in organic production the international federation of organic movement (ifaom).specifies that only ethanol, water, edible oil and carbon dioxide are allowed.

## 1.1 AIMS/OBJECTIVE

The aim of this research work is to extract and characterize oleoresin using dried ginger. This aim can be actualize through the characterization of ginger oleoresin which is divided into. Namely physical and chemical analysis.

Physical analysis;

(i) Specific gravity

(ii) pH

(iii) Boiling point

(iv) Colour

(v) Viscosity

The chemical analysis consists of;

(i) Free fatty acid

(ii) Ash content

(iii) Iodine value

(iv) Acid value

## 1.2 SIGNIFICANT OF THE STUDY

Since ginger has been reported to have some medicinal values among many other properties. This great uses and application of ginger in food and pharmaceutical industries (most oleoresin). Prompted the need to see the viability of isolating oleoresin from ginger .These will be very important if feasible because it lead to the development of an indigenous technology in process design.

## 1.3 SCOPE AND LIMITATION OF THE STUDY

This work is streamlined to the extraction and characterization of oleoresin It is not intended to carry out the application of oleoresin .since.(Adukadir Ahmed. 1991) carried out extraction of oleoresin. This project thus movies a step further in characterization of oleoresin.

# CHAPTER TWO

## 2.0 LITERATURE REVIEW

### 2.1 HISTORICAL BACKGROUND

The native country of ginger is unknown, although it was suppose to be Asia. It was cultivated in tropical region of Asia and America, ginger was known in Europe not later than the first century A.D. It source was not known, but it must have been brought by Arab trader from India (whiley, 1974).The spice was known in Germany France in the ninth century as common in trade in the tenth century .By the thirteenth century, It was nearly as common in trade as pepper .Preserved ginger from China was imported into Europe as sweetmeat as earl as middle age (purseglove, 1975).

The Arabs took the plant from India to east Africa in the thirteenth century and the Portuguese took it to west Africa and other part of the tropics in the sixteen century .As living rhizome of ginger are very easy to transport, the plant was quickly taken throughout the tropic. The Spaniards also introduced ginger to Jamaica from where over 1,000 tones of rhizomes are said to have been exported to Spain in 1547 A.D. It has been transported and introduces to many tropical countries and now cultivated in several parts of the world.

### 2.2.1 BOTANICAL SOURCE OF GINGER

The botanical name of ginger is zingiber officinale roscoe. Ginger belong to the family of zingiberceae, as do many hundreds of other plant , some of them quite important to people .There are different type of ginger that belong to the zingiberaceae, they are zngiber cassumurrar known in India.

It is widely used in India medicine for diarrhea and as a substitute for real ginger (Nelon, 2000.). Another type of ginger, zingiber zerubet is widely cultivated throughout Asia, mostly as a medicine or cough, asthma and stomach aches. In addition, unlike real ginger, it seems to be very popular for skin disease. It tastes and smell like a bitter vessim of ginger but it rhizome and root are gigantic (Nelson, 2000.).

We also have zingiber mioga, are the japans ginger, grow and used on that no one in the spice trade bother to differentiate it botanically from the ginger groom in India and

elsewhere. Zingiber elatum and zingiber chrysanthum are other spice of every aromatic Asia ginger.

## 2.3 PROSPECT OF GINGER CROP PRODUCT IN NIGERIA.

It was asserted that ginger was introduced into Nigeria by Jamaican (Edward,1975.).The major producing area in Nigeria are Kaduna state, Nassarawa state,sokoto state, zamfara state ,Akwa Ibom state, Oyo state, Abia state and Lagos state. However, southern part of kaduna state remain the highest producer of fresh ginger in Nigeria and subsequently has become an attractive market area of ginger product(Areo, 2005).

In southern part of Kaduna state Nigeria, the rural communities of kagarko, kwoi, kagoma, zonkwa, ungwa rimi have been the main producer and chief source of supply of ginger crop for the Nigeria domestic and external market.

The first ginger market in Nigeria began in Gwantu and kachia of southern Kaduna state (Beecroft, 1981) said that shipment to the overseas market was mainly to London and later to U.S.A and Canada .Ginger commodity therefore has very wide market as it produce other by product(Beecroft,1981)listed the bye-product as ginger oil, ginger oleoresin, ginger syrup or juice and ginger flakes.

## 2.4 CHEMICAL COMPOSITION OF GINGER

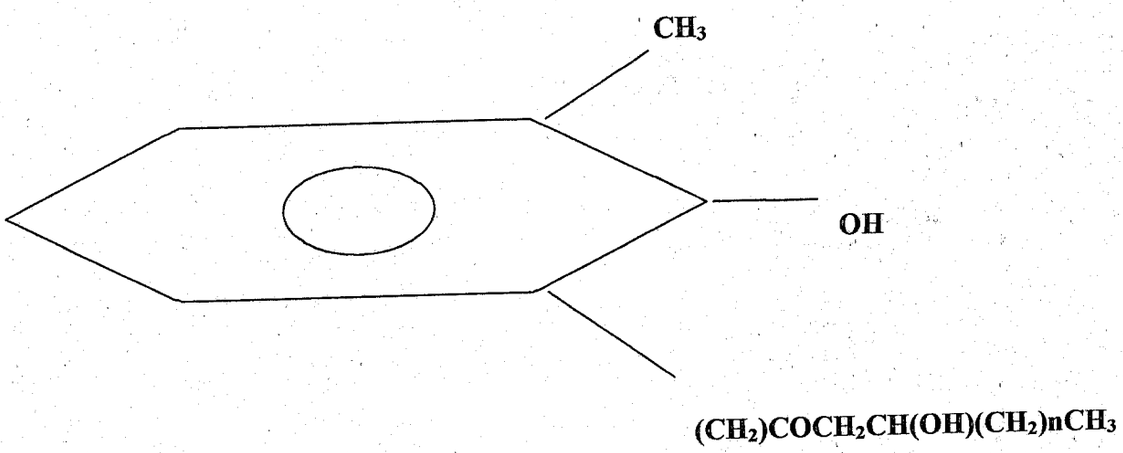
**TABLE 2.1: CHEMICAL COMPOSITION OF FRESH GINGER**

| *COMPONENTS* | *PRECENTAGE COMPOSITION(%)* |
|---|---|
| WATER | 80.2 |
| PROTEIN | 2.3 |
| FAT | 1.0 |
| STARCH | 12.3 |
| FIBRE | 2.4 |
| ASH | 1.2 |
| VOLATILE OIL | 1-2 |
| RESINOUS MATTER | 5-8 |

Source: (Areo, 2005)

The pungent principle of ginger is due to gingerol (Zingeron) $C_{11}H_{14}O_3$ which is present in the Oleoresin (solvent extract).

Fig 2.1 Gingerol.




(where n = 3, 4 or 5)

Ginger has a number of gingerols described as 6-gingerol, 10-gingerol and possibly 16 carbon, three gingerols compose about one-third (1/3) of ginger oleoresin. The pungency of ginger is destroyed by boiling with 2 % Potassium hydroxide (KOH) to 70°C.

## 2.4 MEDICINAL VALUE OF GINGER

A study publicized in the journal of Ethropharmacology (Grueriwald, 1998) revealed that ginger significantly inhibit the growth of both gram-positive and gram-negative bacteria.

Ginger is a stimulant that when chewed, it increase flow of saliva. When swallowed, it act as stimulating tonic, stomachic and increase the secretion of gastric juice exactly the excitability of the alimentary muscular system and dispelling gases accumulated in the stomach and bowels.

Other studies and analysis reveals that, ginger has pronounced antioxidant activity, reduced inflammation much like an analgesic, and may even help in arresting narcotic addition (Fulder, 1996).

It appears this spicy aromatic has something to offer everyone. From flavor to fitness. Ginger is effective not only for indigestion, but also in preventing the symptoms of motion sickness.

Researcher, (J. Backin, 1996) has studied ginger effect on human health for over a decade and has become convinced through numerous trials that the aromatic spice offer a wide range of potential health benefits.

He believes that, because ginger is such a potent thronboxane synthesis inhibitor and prostacylin suppressor, it has theraperatic capabilities in alcohol withdrawal, in recovery from serious burns, in treating peptic ulcer as an anti depressant in preventing ageing penile vascular changes and impotency and as an analgesic in dysmenorrheal (painful menstruation).

### 2.4.1 SOME BIOLOGICALLY ACTIVE COMPONENTS IN GINGER

**TABLE 2.2: BIOLOGICALLY ACTIVE COMPONENT IN GINGER**

| *SUBSTANCE* | *EFFECT* |
|---|---|
| Asparegines | Promote urination |
| Borneol | Analgesic anti-inflammatory, protect liver |
| Chavicol | Kills fungi |
| Cincole | Lower blood pressure, anticeptic clear throat |
| Citral | Antilislamine antibiotic |
| Cumen | Narcotic |
| Cymen | Kill visniser, kill fungi, kill insect |
| Geranoil | Anticandida, kill insects |
| Gingerol | Analgesic stimulate, circulation, lower blood pressure |
| Gingerdiorie | Inhibits prostaglandins |
| Limonene | Can irritate skins and deters insects |
| Myrecene | Kill bacteria and insects |
| Neral | Kill bacteria |
| Piriene | Remove philedun, kills insects |

| | |
|---|---|
| Shogoal | Analgesic, lower blood fever, coustric blood |
| Zingerone | Raise blood pressure |

Source: Foulder, 1996

## 2.5 STARCH CONTENT OF GINGER

Starch is the chief food reserve of plant and is converted as required into sugar. It may be stored in the stem as in the palm, in the tuber as in cassava or in rhizome as in ginger. On microscopic examination, starch from various plant sources is found to consist of small granule, shape and size of which are peculiar to the plant from which they have been obtained.

The size of starch granule measure along their longest axis varies from $2 \times 10^{-4}$cm – $15 \times 10^{-2}$cm (Brain and Allan, 1970).

The size of starch granule may influence the properties of starch because larger granular gelatinize more easily than granules.

Starch does not dissolve in cold water due to closer and more orderly packing of starch particles at the surface of the granule than in it interior. If a suspension of starch in water is heated, water diffuses through the walls of the granules and cause swelling. This been at 60°C and by 85°C the volume of the granules has increase by about five times, and the suspension will be very viscous, on above this temperature, the starch granule burst giving a gel of starch in water. At this point, sedimentation of starch (more than 50%) in ginger cannot be overemphasized. It is therefore paramount to sediment the starch granules and decant the mother liquor (Brain and Allan, 1970).

## 2.6 WATER SOLUBLE COMPONENT OF GINGER

The water soluble components of ginger include the following:-

i) Amino acid

ii) Mineral

iii) Ketones

iv) Fatty acid which includes stearic acid, oleic acid , linolic acid

v) Vitamins particularly Vitamin B & C

(Bardger, 1984)

### 2.6.1 INDUSTRIAL PROCESSING AND COMMERCIAL FORMS OF GINGER

In general ginger is processed for international market in the form of its three (3) primary products; these are fresh ginger and dried ginger.

### 2.6.2 FRESH GINGER

Sometime known as green ginger, is produced in large number of countries in Asia, in the Pacific region and in the Indian Ocean Islands, In Australia and most West African countries notably Nigeria, Sierra Leone and Kenya.

### 2.6.3 PRESERVED GINGER

Preserved ginger is preferred in certain ginger growing countries like China, Hong Kong, Australia, and India. The two forms of processed ginger at the market are:

(a) Preserved ginger in sugar syrup

(b) Crystallized ginger. This is sugar which has been impregnated into sugar syrup dried and coated with crystalline sugar. (Mosley et al, 1979).

### 2.9.3 GINGER OIL

Ginger oil is distilled from fried spice mainly in the major spice importing countries of Western Europe and North America.

This product possesses the aroma and flavour of the spice but lacks the pungency. (Mosley et al 1979)

This oil is located in the cells immediately under the epiderssmis, peeling of the rhizome will ineciatably result in a loss of essential oil (Musa, 1989)

There is no production of ginger oil in Nigeria, while oil is produce in small scale in Jamaica, India, China and Japan (Haruna, 1989)

### 2.10 GINGER OLEORESIN

Ginger oleoresin is obtained by solvent extraction by either alcohol or acetone of dried ginger and is treated both in industrialized Western countries and some of the spice producing countries mostly Australia and China (Abdulkadir 1991).

Ginger oleoresin is extracted from various types of ginger but the majority of all ginger oleoresin are derived from Nigeria and Jamaican ginger. Ginger oleoresin contain gingerol which is responsible for it pungency. Other constituent of oleoresin include Shongoal 6,8,10 (paradol) $C_{17}H_{24}O_3$, 4, 6,8, 10 (gingediol) 6-methyl gingedide, 4 and 6 gingediacetate and 4, 6 hexahydrocircunin. It is intresting to note that when stored gingerol gradually changes to shogoal, which is not very different from gingerol. This is why ground pungency gradually drops as the spice sits aging at the back of the kitchen cupboard. (Cornel and Sutherland, 1969).

Oleoresin, commercially called Gingerin contains Pungent principles, Gingerol and Shogoal.

## 2.11 SOME COMMON PROPERTIES OF GINGER OLEORESIN

**Table 2.4**

| ***Colour and appearance*** | ***Dark brown viscous liquid*** |
|---|---|
| Odour description | Characteristic odour of ginger |
| Odour type | Spicy |
| Acid value | 0.98mgKOH/g |
| Volatile oil content | 35% |
| Residual solvent | <10ppm |
| Solubility | Soluble in oil |
| Free fatty acid | 60% |
| Flash point | 168.00°F (75.56° C) |
| Specific gravity | 0.882 – 0.89 |

Source:(www.niir.org)

## 2.12 OLEORESIN PRODUCT FORMULATION

Most of the world's ginger processed into concentrates (oleoresin) suitable for the manufacture of ready to serve ginger drink e.g ginger ale and ginger bear (langer et al, 1998)

### 2.12.1 GINGER ICE CREAM

The recipe recommended is 16litres of ice cream mixes with 5g of ginger powder, 40ml of oleoresin and a shade of vanilla colouring agent. Although, the resultant ice cream is hot, it was found acceptable to a large number of people. (Steward,1953).

### 2.12.2 GINGER TONIC

This is a mixture of oleoresin and pure honey. The proportion of oleoresin that is being added to the honey before mixing depends on how ash is the oleoresin.

### 2.12.3 GINGER INSTANT TEA

These also have the same procedure with the tonic only that in this case a blender is use to blend oleoresin with brown sugar. 10kg of brown is mixed with 40ml of oleoresin also the quantity of oleoresin dosage also depend on it purngency.

## 2.13 APPLICATION OF OLEORESINE

### 2.13.1 AS FLAVOURANT :

Oleoresin is primarily used as a food flavoring in soft drinks like ginger ale, bitters, cordials and liquors, as a spice in bakery product, confectionary, pickles, sauces and preserves .et.c.

### 2.13.2 PHARMACEUTICAL USES:

The pharmaceutical use are; carminative, rubefaccient, stimulant, in alcoholic gastritis, dyspepsia e.t.c.

#### 2.13.2.1 VETERINARY USED:

As stimulant and carminative, in indigestion of horses and cattle.

#### 2.13.2.2 USED IN PERFUMERY

(www.niir.org).

### 2.13.3 SPECIE GINGER CONCENTRATE

This concentrate is used in the manufacturing of ginger flavoured sweet and pepper mint extract e.t.c. It is made from ginger oleoresin extract mixed with orange oil.(Steward, 1953).

### 2.13.4 CHEMICAL TEST

These examine the physiochemical properties of oleoresin. They are;

Saponification value: This is the number of milligram of potassium hydroxide to neutralize fatty acid resulting from complete hydrolysis of 1g of the oleoresin sample.

Acid value: is the milligram of KOH require to neutralize free acid in 1g of oleoresin sample.

### 2.13.5 PHYSICAL TESTS

This examines the physical properties of the oleoresin. They are:

Specific gravity: This is the ratio of the weight of substance to the weight of the same volume of water at a specific temperature.

Boiling point: This is the temperature at which it vapour pressure becomes equal to the atmospheric pressure.

**pH:** This is used to express the degree of acidity and alkalinity of a substance others include melting point, freezing point, viscosity, e.t.c.

## 2.14 SOLVENT EXTRACTION

This process employs a solvent to lead out the oleoresin and it is the only practical method recovering oleoresin from tissues having a relative low portion of oleoresin. Gasoline, Benzene, petroleum ether, ethanol, acetone, trichloro ethane or dichloroethane, although the latter two are non carcinogenic and ethyl acetate or hexane is preferred.

Dried powdered rhizome are extracted by percolation and the extract is then cold distilled at (45 – 50) ° C to remove all the solvent while assuring integrity of gengerols by not overheating. (www.fao.org)

Hydrophilic solvent such as ethanol and acetone also extract water soluble gums which may need to be further separated by centrifugation. However water soluble solvent may be preferred to prepare extractive to be used by the beverage industry to assure water solubility.

### 2.14.1 THE CHOICE OF SOLVENT

The choice of separation agent is the most important and any particular liquid having the following properties: solubility, selectivity, density, viscosity, interfacial tension, recoverability e.t.c should be considered for certificate organic production,

synthetic solvent are not allowed. Therefore solvent derived from pharmaceutical such as hexane, pentane, di and tri-chloromethane.(www.fao.org).

**Density:** A difference in density of the saturated liquid phase is necessary both for stagnise and continuous-contact equipment operation. The larger this differences the better system.

**Viscosity**: This has to be low for each handling and storage.

***Selectivity***: The effectiveness of solvent B for separating a solution of A and C into it component is measured by comparing the ratio of C and A in the B rich phase to that in the A-rich phase at equilibrium.

The ratio of the separation factor or selectivity B is analogous to the relative volatility of distillation, if E and R are the equilibrium phase.

$$B = \frac{\{\text{wt fraction c in E}\}/\{\text{wt fraction A in E}\}}{\{\text{wt fraction C in R}\}/\{\text{wt fraction A in R}\}}$$

***Interfacial tension***: the larger the interfacial tension, the more readily will coalescene of emulsion occur, but the more difficult will dispersion of one liquid in the other be.

***Recoverability***: It is always necessary to recover the solvent for reuse, and another of the mass transfer operation, most frequently distillation must ordinarily do this. If is to be used, the solvent should form zero type with the extract solute and mixture should show relative volatility for low cost recovery.

- The solvent should have low boiling point to allow simple distillation, but for solute as well as solvent purification. There is little point in maximizing leaching of the solute is impossible or difficult to recover Boiling point of solvent is very important in this extraction process the boiling of the solvent is expected to be low because of the constituent of ginger oleoresin are sensible to temperature.
- The boiling point of the solvent should be low otherwise evaporated losses may be high. In the case of toxic solvent almost all organic solvent are in close system or careful ventilation. If the threshold limit value (TLV) is not exceeded, the TLV is used in legislation for safety in the work place. TLV is the concentration to which it is believed

the average worker could be exposed to day-by-day for 8hours a day, 5days a week, without suffering harm from toxic materials. (Adesiyan, 2001).

- A similar restriction should be in place on inflammable solvent, if possible the solvent should be non-inflammable, but where this is not possible, the boiling point and flash point should be as high as possible.
- Preferable the solvent should be non-toxic.
- The solvent should be cheap so that cost of the initial inventory is reduced as it associated cost of solvent replacement.
- The solvent should be chemically stable and inert towards the material of construction.
- The solvent should be readily available.

## 2.15 SOLID-LIQUID EXTRACTION PROCESS IN GINGER

This is a case where soluble constituent present either as a solid or liquid is removed from a solid or liquid by the use of a solvent. It is a mass transfer phenomenon of the extraction from a soluble component by means of a solvent. Liquid-solid extraction is an industrial operation use in large scale chemical technology (*Howitz, 1970*).

This operation can be sub-divided into 2types:

- Extraction that occur because of the solubility of the solute in or it miscibility with the solvent. E.g. ginger extraction, oil extraction and oleoresin extraction e.t.c (*Howitz, 1970*).
- Extraction where solvent must react with constituent of the solid material in order to produce a compound soluble in the solvent e.g. extraction of metals from metalliferous ores. (*Howitz, 1970*).

The above two categories are subject to different constraints. The first being rate controlled by diffusional phenomena, where as, the second is more frequently governed by the kinetic of the chemical reaction producing the solute these difference reflected in different techniques use to analyse and carry out the operation (*Areo,2005*).

Usually, it is not possible to completely separate the liquid phase from the solid phase. In practice thus, the steam from the second step above consist of liquid phase which do not contain any solid known as overflow and remainder consisting of solid plus adhering solution known as underflow. Liquid extraction on ideal stage is define as a stage in which the solution - leaving overflow of the same composition as solution retained by the solid in the underflow. Most solid extraction systems are considered to consist of 3 components:

- Solute
- The inert solid component
- The solvent component

Solid – liquid extraction can be used when the extracted component is required to remove the solid residue often more associated with purification such as washing of filterfor the extraction of oil from vegetable seed. This mixture is often called Micella and leached solid marc.

In general, the extraction of vegetable oil is quite difficult especially if high quality product is required in high yield. The overall process involves several stages:

- Cleaning and Dehulling
- Reduction
- Cooking
- Pressing
- Extraction by solvent.

The solvent method of extraction or leaching of oil is the most efficient method for the recovery of oil from oil-seeds. The success of leaching and technique to be used will vary frequently depending upon any prior treatment, which may be giving to the solids.

Also, choice of extraction method depends on the seed to be processed and the equipment available.

The solvent recovery from micelle and marc is an essential part of the leaching process. Typically the filtrated micelle is passed to an evaporated for removal of the solvent.

# CHAPTER THREE

## 3.0 MATERIALS AND METHODS

The apparatus, raw, materials and chemical used for the research work are listed below. The major raw material was sourced from kwoi southern part of Kaduna state.

## 3.1 MATERIALS

1. Soxhlet apparatus.
2. Morter.
3. Oven.
4. Weighing balance.
5. Heating mantle.
6. Thermometer
7. Stop watch.
8. PH.
9. Refractometer.
10. Density bottle.
11. Visco tester.
12. Beaker, cornical, round bottom, and flat bottom flask.
13. Burret, pipette and measuring cylinder.
14. Filter paper.
15. Dried Ginger.
16. Distilled water.
17. Diethyl ether.
18. Ethanol.

## 3.2 PREPARATION OF GINGER RHIZOME FOR EXTRACTION AND DETERMINATION OF MOISTUER CONTENT.

The ginger rhizome (dried) collected was weighed and dried in an oven, which was maintained at 100oc for one hour. After the weight was noted.

The weights of dried ginger before and after drying were denoted by w1 and w2 respectively .The moisture and other volatile content were calculated in percentage as;

$$\frac{W_1 - W_2}{W_1} \times 100\%$$

## 3.3 EXTRACTION OF OLEORESIN FROM GINGER BY SOLID LIQUID EXTRACTION WITH A SOXHLET EXTRACTION APPARATUS (USING ETHANOL AS SOLVENT).

Dried ginger sample were crushed in a grinder, an empty flask and timble were weighed and their weight were denoted, $M_1$and$T_1$, some amount of sample was transferred into the thimble and the weight was noted. The weight of the sample W1was then noted from difference.

The thimble containing the sample was then transferred into the extractor. The known volume of solvent, 250ml was poured into the extractor and the flask .solvent used was 3/5of the volume of the flask.

The extractor was then connected to a condenser above it to the flask at it bottom the heater was then set at 70%. The heating make the solvent in the flask to evapourate and condense by a condenser into the extractor, of which the dark brown coloured of solution was observed which rose up in the level of the extractor capillary tube and then fell down through the capillary tube into the flask. The coloured solution remain in the flask, the process continued for 5 hours after which it was observed that the solvent condensing in the extractor formed no coloured solution.

### 3.1.1CHARACTERISATION OF OLEORESIN GINGER

This is divided into physical and chemical analysis of the ginger oleoresin.

### 3.4.1 PHYSISCAL ANALYSIS.

#### 3.4.1.1 SPECIFIC GRAVITY.

**PROCEDURE**:

An empty container was weighed on weighing balance and the weight was noted .Then it was filled up with water and the weight was also noted. The water was poured away an ginger oleoresin was filled into the same container. The weight was also noted

The specific gravity was then calculated using

- Specific gravity = $\frac{\text{weight of oleoresin ginger}}{\text{Weight of equal volume of water}} \times 100$

### 3.4.1.2 BOILING POINT OF GINGER OLEORESIN

The boiling point of ginger oleoresin was determine by putting the extract in a beaker an a thermometer immerse in it. It was heated on a heating mantle. After some minutes of heating first bubble was observed. The temperature was immediately noted.

### 3.4.1.3 PH OF GINGER OLEORESIN

**PROCEDURE;**

The PH electrode was lowered into buffer solution .The temperature control was adjusted to temperature of buffer solution .The digital control was adjusted and the meter indicate exert PH which was raised and raised with buffer .the electrod was then immersed in the ginger oleoresin .The PH and the temperature of the ginger oleoresin was then noted.

### 3.4.1.3 REFRACTIVE INDEX

**PROCEDURE;**

Few drop of oleoresin were place in the face of the prism of refractometer and gently spreaded, closed and tightened. An ample time was allowed the oleoresin and prism to attain a steady temperature .The refractive index was then read from the demarcation line after adjusting to where it coincides with the diagonal crossing.

### 3.4.1.4 DENSITY

**PROCEDURE;**

250ml of oleoresin was weighed by firstly weighing the empty container on a weighing balance .This was followed by the addition of the oleoresin into the empty weighed container, the weight of empty container was subtracted from the total weight of container and ginger oleoresin, this gives this give the mass of 250ml ginger oleoresin .The density is calculated at a temperature of 29°C.

$$\text{Density} = \frac{\text{mass}}{\text{Volume}}$$

### 3.4.1.5 VISCOSITY

**PROCEDURE;**

Two viscosity cups was in stalled on triple stands each. Equal volume of ginger oleoresin was poured into one cup while the draining hole was blocked. Immediately it

was released, digital timer was press off. The time was noted .The same procedure was repeated two more time using equal volume. All the time was noted and average time was taken.

The same method was used for water .The average time taken to drain out of viscosity cup was also noted.

Then, viscosity of ginger oleoresin was determined using below expression.

$$\frac{\text{Viscosity of oleoresin}}{\text{Average time taken to drain}} = \frac{\text{Viscosity of water}}{\text{Average time taken to drain}}$$

### 3.4.1.6 ASH CONTENT DETERMINATION

A clean flask bottomed silica dish was held on a bursen burner flame for about one minute. It was the transferred to a desiccator to cool and weighed.

50ml of the oleoresin sample was taken into the dish and the total weight was obtained the silica dish and the sample was heated gently on a bursen burner in fume cupboard until the smoke ceased. It was then transferred to muffle furnace heated to about $500^{O}$c. the temperature was maintain for about 4 hours, to ensure that the whole carbon has been burnt away. The furnace was switched immediately and placed inside a desiccator to cool and weighed.

### 3.4.2 CHEMICAL ANALYSIS.

### 3.4.2.1 FREE FATTY ACID (F. F. A)

**PROCEDURE;**

25ml of diethyl ether was mixed with 25ml of ethanol in a beaker .The solution was poured on 10g of oleoresin oil in the flask and 1ml of phenolphthalein was added to the mixture . The mixture was then titrated against 0.1M NaOH with constant shaking for which dark pink colour was observed and volume of 0.1M NaOH ($V_b$) was noted .

$$\text{FFA} = \frac{\text{Volume of base } (V_b) \times 2.82 \times 100}{\text{Sample weight } (W_o)}$$

Where 100ml of 0.1M NaOH = 2.82g of oleic acid

### 3.4.2.2 ACID VALUE

**PROCEDURE**

### 3.4.2.2 SAPONIFICATION VALUE

**PROCEDURE**

2g of oleoresin was weighed in a conical flask .25ml of alcohol KOH was pipetted and added into the conical flask .The conical flask was attach to a reflux condenser and was boiled over heating water bath for 30 minutes with occasional shaking immediately after 30 minutes of boiling, 1ml of phenolphthalein was added to the mixture and was titrated while hot against 0.5M HCL acid solution.

The mixture was observed to be light yellow .The volume of 0.5M HCL acid solution (a ml) used was noted.

Blank determination was carried out; the same method was out as stated above. But the oil was substituted for some volume of distilled water. Volume of 0.5ml HCL acid used (b ml) was also noted.

1st titration =a ml

2nd titration =b ml

$$\text{Saponification value} = \frac{(b-a) \times 2}{\text{Sample weight } (W_o)}$$

### 3.4.2.3 IODINE VALUE

**PROCEDURE**

2g of oleoresin oil was added to 5ml of carbon-tetrachloride (c cl4) and was then dissolve in 10ml of mij's solution. The mixture was previously moistened with potassium iodine solution.This was then allow to in the dark for 30minute.After which 7.5ml added and mixed the solution was titrated with 0.1m sodium thiosulphate solution using starch as the indicator just before the end point. After the addition of other drops the liquid looked colourless (5ml), it then turn purple the same process was repeated for blank (Bml) titration. The titre value for both (5 ml) and (Bml) were noted. The lodine value was then calculated thus:

$$\text{Iodine value} = \frac{(B-S) \times M}{\text{weight of sample}}$$

# CHAPTER FOUR

## 4.1 RESULT AND DISCUSSION OF RESULTS

### TABLE 4.0 PHYSYCAL PROPERTIES OF GINGER OLEORESIN

| *PROPERTIES* | *DESCREPTION* |
|---|---|
| 1. Colour | Dark brown |
| 2. Odour | Spicy |
| 3. Specific gravity | 0.99 |
| 4. Boiling point | 142°C |
| 5. $P^H$ | 6.9 |
| 6. Refractive index at 29°C | 1.492 |
| 7. Viscosity at 29°C | 1.4470 |
| 8. Yield of oil | 10.17% |
| 9. Density at 29°C | 0.98g/ml |
| 10.Ash content | 2.7% |

### TABLE 4.1 CHEMICAL PROPERTIES OF GINGER OLEORESIN

| *PROPERTIES.* | *DESREPTION.* |
|---|---|
| 1. Free fatty acid | 564% |
| 2.Acid value | 1.128mg KOH/g |
| 3. Saponification value | 17.95mg KOH/g |
| 4. iodine value | 71.63 |

## 4.2 DISCUSSION OF RESULTS

The ginger oleoresin extracted by soxhlet apparatus using ethanol as solvent yielded 10.17%. This value is lower than literature values because the extraction was not first just carried out in a single stage. The samples were placed four times after each extraction has been assumed to complete. However, it is known that the concentration or viscosity of the solvent increase with extraction and this reduce the extraction driving force. Hence, the subsequent extraction may not be as effective as when pure solvent is employed.

The oleoresin ginger was characterized under the physical and chemical analysis. The physical tests are: colour which was achieved by more observation to be dark brown. Specific gravity of 0.99 which is close to the standard value of 0.851. The oil had a boiling point 142°C. The refractive index of was found out to be 1.4470 at 29°C which was lower than that of literature ranging 1.460 to 1.470. The refractive index is useful

detecting adulteration, which is done by comparing the value obtained originally for the oil with that of adulterated oleoresin. The pH value of 6.9.

The physiochemical properties showed an agreement on the with the physiochemical properties of ginger oleoresin, most of the slight difference arose from the fact that yield of oil and some other properties depend on cultiva variety,place it was obtained and method of extraction of oleoresin.Other factor that may lead to the deviation of analysed values is the operating temperature of the process which is 60°C.Since temperature has an effect on gengerol which is one of the most important constituent of ginger .

The chemical analysis carried out include; percentage free fatty acid . which was found to be 56.4%.This is express as the commonest acid present in oleoresin. The acid value was found to be 1.28mg/KOH/g. this is the measure of amount of free fatty acid present in oleoresin. The specification value was found to be 17.95mgKOH/g.this is a measure of of the mea molecular weight of the free fatty acid present in oleoresin oil. The process of saponification is the hydrolysis of triglyceride into glycerol and the potassium hydroxide in alcohol. This process measure the amount of alkali, which is required to combine with the free fatty acids.Iodin value was found to be 71.63 i.e 71.63 milligram of iodine was absorb by 100g of fat. This measure proportion of unsaturated acid present. Particle size of 2mm was discovered to have the best yield.

Finally the capacity of the condensing unit was not efficient enough and it attribute to the loss of some amount of solvent. Within experimental limitation the Nigeria ginger obtained from southern part of Kaduna State is viable for any industrial raw material for ginger oleoresin processing.

# CHAPTER FIVE

## 5.0 CONCLUSION AND RECOMMEMDATION

## 5.1 CONCLUSION

Within the limit of experiment errors, the ginger oleoresin extracted by soxhlet apparatus using ethanol as solvents has a yield 10.17% dark brown in colour, odour type is spicy, appearance dark brown viscous liquid, specific gravity 1.008, boiling point of 142°C pH value of 6.9, refractive index of 1.4470, viscisity $3.65x10^{-3}$ kg/ms, density of 0.99g/ml, free fatty acid 56.4% acid value of 1.128mg/koh/g, saponification value of 17.95mg/koh/g and iodine value of 71.63mg of iodine. It can also conclude that oleoresin contain phenolic compound such as gingerols and shogaols. The main pungent flavour chemicals are gingerols which are not volatile.

## 5.2 RECOMMENDATION

The following are recommended for further studies:

1) Design of pilot plant for ginger oleoresin extraction.
2) Each component of the ginger oleoresin should be extracted individually and tested on rats in order to ascertain the one responsible for the cure of common cold.

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## CALCULATION AND RESULTS

## APPENDIX ONE

## PERCENTAGE OF MOISTURE AND OTHER VOLATILE SUBSTANCE

Weight of ginger before drying $W_1 = 450g$

Weight of ginger after drying $W_2 = 425g$

% of moisture and other volatile substance.

$$= \frac{W_1 - W_2}{W_1} X \quad 100$$

$$= \frac{450 - 425}{450} X \quad 100$$

$$= 24.4\%$$

## PERCENTAGE OF OLEORESIN YIELD BY SOXHLET EXTRACTION EQUIPMENT USING ETHANOL SOLVENT

Weight of empty flask ($M_1$) = 4.01g

Weight of thimble ($T_1$) = 6.18g

Weight of thimble + sample ($T_2$) = 146.7g

1st Weight of sample ($W_s$) = 40.32g

Weight of flask + oil = 17.68g

Weight of oil ($W_o$) = M2-M1

= 17.68- 4.01

=13.67g

2nd Weight of sample = 37.38g

3rd Weight of sample = 28.20g

4th Weight of sample = 28.46g

Total weight of sample = 40.32 + 37.38 + 28.2 + 28.46=134.36g

$$\% \text{ Oil yield} = \frac{M2 - M_1}{Ws} X \quad 100$$

$$= \frac{17.68 - 4.01}{134.36} X \quad 100$$

$$= 10.17\ \%$$

**SOLVENT RECOVERD**

Ethanol boiling point = 78.5 °C

Volume of solvent used =1140 ml

Volume of solvent recovered = 825ml

Volume of solvent lost =315ml

## APPENDIX TWO

## CHARACTERIZATION OF OLEORESIN GINGER BY PHYSICAL ANALYSIS;

### SPECIFIC GRAVITY

Weight of empty container = 2.39g

Weight of container + oil = 3.37g

Weight of container + water = 3.47g

Weight of oil = 0.998g

Weight of water =0.99g

$$\text{Specific gravity} = \frac{\text{weight of oleoresin}}{\text{Weight of water}}$$

$$= \frac{0.998\text{g}}{0.99\text{g}}$$

Specific gravity = 1.008 at 27°C

### VISCOSITY

Volume of oil = 3.5ml

Time taken for 1st flow of oil =5.62s

Time taken for 2nd flow of oil =6.59s

Time taken for 3rd flow of oil = 6.71s

$$\text{Average time taken} = \frac{5.62 + 6.59 + 6.71}{3}$$

$$= 6.35\text{s}$$

Volume of water = 3.5ml

Time taken for 1st flow of water = 1.37s

Time taken for 2nd flow of water =1.28s

Time taken for 3rd flow of water = 1.32s

$$\text{Average time taken} = \frac{1.37 + 1.28 + 1.32}{3} = 1.32\text{s}$$

Viscosity of water = 7.65 x 10-4

Using the expression

$$\frac{\text{Viscosity of oleoresin}}{\text{Average time taken for oil}} = \frac{\text{Viscosity of water}}{\text{Average time taken for water}}$$

$$\text{Viscosity of oleoresin} = \frac{\text{Viscosity of water x Average time taken (oleoresin)}}{\text{Average taken for water}}$$

$$= \frac{7.65 \times 10\text{-}4}{1.32} = 3.651 \times 10\text{-}3$$

**DENSITY**

Volume of oleoresin =10ml

Weight of oleoresin + beaker =12.19g

Weight of beaker = 2.39g

Weight of oleoresin = 9.8g

$$\text{Density} = \frac{\text{mass}}{\text{Volume}}$$

$$\text{Density} = \frac{9.80\text{g}}{10\text{ml}}$$

Density = 0.98g/ml

## APPENDIX THREE

## CHARATERIZATION OF GINGER OLEORESIN BY CHEMICAL ANALYSIS

### FREE FATTY ACID

**TABLE 5.0 ; 0.1M NaOH Titrations values.**

| *NaOH* | *Initial(ml)* | *Final (ml)* | *Results (ml)* |
|---|---|---|---|
| **1st Titre** | **2.20** | **4.10** | **1.90** |
| **2nd titre** | **4.10** | **6.20** | **2.10** |
| **3rd titre** | **6.20** | **8.20** | **2.00** |

Average volume of NaOH used $V_b = \frac{1.90 + 2.10 + 2.00}{3}$

$= 6/3 = 2.00ml$

Free fatty acid volume of base used $= \frac{\text{Volume of base used } V_b \times 2.82 \times 100}{\text{Sample weight}}$

$= \frac{2.00 \times 2.82 \times 100}{10}$

$= 56.4\%$

### ACID VALUE

Acid value $= \frac{\text{Volume of base used Vb } 2.82 \times 2}{\text{Sample weight}}$

$= \frac{2.00 \times 2.82 \times 2}{10}$

$= 1.128 mgKOH/g$

**SAPONIFICATION VALUE**

**TABLE 6.0: 0.5M HCL**

**BURRETTE READINGS**

| | *1st Titre (ml)* | *2nd Titre (ml)* |
|---|---|---|
| **Final** | **16.60** | **44.20** |
| **Initial** | **5.00** | **16.40** |
| **Result** | **11.60 (a)** | **27.60 (b)** |

Saponification value $= \frac{b - a \times 28.05}{\text{Wt of sample}}$

$= 27.6 - 11.6 \times 28.05$

$= \frac{16 \times 28.05}{25}$

$= 17.95$mg KOH/g

**IODINE VALUE**

**Table 7**

**BURRETTE READINGS**

| | *SAMPLE TITRATION (ml)* | *BLANK TITRATION (ml)* |
|---|---|---|
| **1st Titre** | **81.70** | **71.50** |
| **2nd Titre** | **84.20** | **69.30** |
| **3rd Titre** | **82.95 (B)** | **70.40 (S)** |

B = Blank titre value.

S = Sample titre value.

M = molarity =0.1

126.9 = Atomic weight of Iodine.

Iodine value $= \frac{(B-S) \times 126.9 \times M}{\text{Wt of sample}}$

$= \frac{(82.95 - 70.40) \times 126.9 \times 0.1}{2} = \frac{159.26}{2} = 71.63$

**PROCESS FLOW CHART**

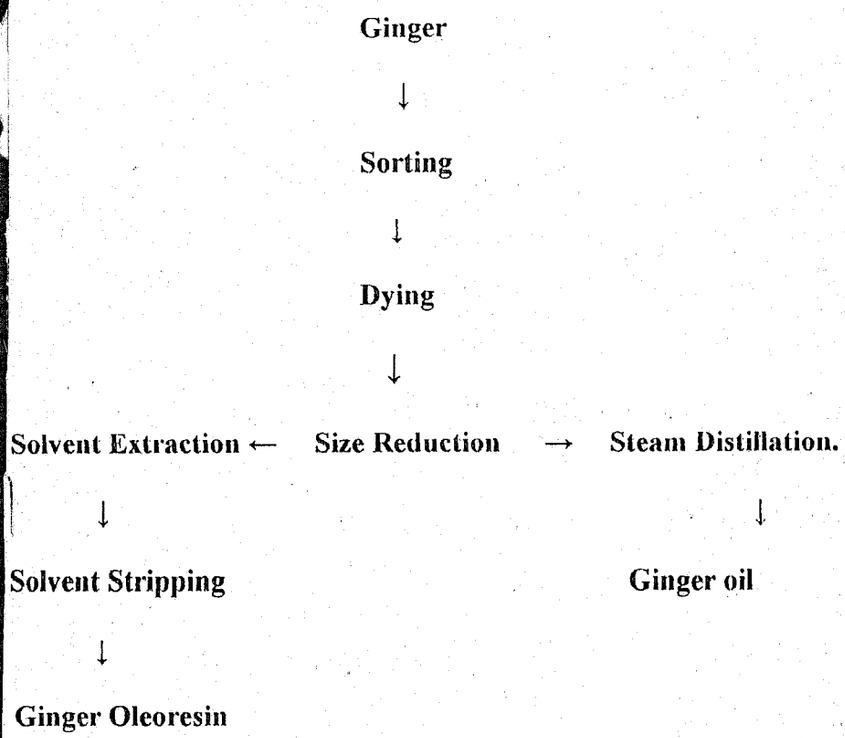


**FIGURE 2: PROCESS FLOW DIAGRAM FOR OLEORESIN PRODUCTION**