

**EFFECT OF SOME FACTORS ON SOLVENT EXTRACTION OF OIL
FROM ROSELLE SEED**

BY

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2003/14843EA**

**A PROJECT SUBMITTED TO THE DEPARTMENT OF
AGRICULTURAL AND BIORESOURCE ENGINEERING,
SCHOOL OF ENGINEERING AND ENGINEERING
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FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,
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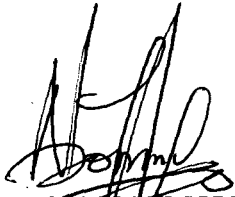
NOVEMBER, 2008

DEDICATION

This project is dedicated to God almighty, the most gracious, merciful lord whose guidance and protection made my academic pursuit a reality and also to my beloved parents Mr Mrs. Dominic.o.Nwokeke for there prayer's and supports throughout the program.

DECLARATION

I, Nwokek.c.Dominic with matriculation number 2003/14843EA, declares that this project, "effect of some factors on solvent extraction of oil from Roselle seed", is my original work and has not been presented else where to the best of my knowledge.



Nwokek.c.Dominic

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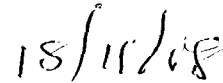
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CERTIFICATION

This is to certify that this project *effect of some factors on solvent extraction of Reselle seed.* was carried out by Nwokek.c.Dominic under the supervision of Engr Peter idah and submitted to the Department of Agricultural and Bio-resource Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna, Niger State, in partial fulfilment of the requirement for the award of Bachelor of Engineering (B. Eng.) degree in Agric and Bio resource Engineering.



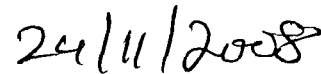
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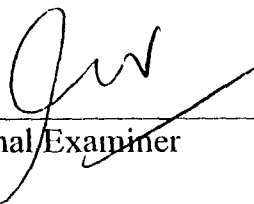
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External Examiner



Date

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My profound gratitude first goes to God Almighty for his infinite mercies and grace throughout this programme. May his Holy name be exalted Amen.

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I want to put on record my sincere appreciation to my brothers and sisters, Ugo, Tochi, Chidumga, Bro Chiji, Chioma, Amara, Bros onyi and Emeka and to my and to my wives, Blessing and Jojo, And to my boy success .D.

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ABSTRACT

The research work effect of some factor on solvent extraction of oil from seed.

Was carried out using petroleum ether and ethanol as solvent. The extraction was carried out at a temperature of 80°C, 90°C and 100°C at constant time and constant amount of mass solvent. The method used is aimed at determining the effect of temperature, heating time and particles size and material weight respectively.

At a range of 5 – 6hrs extraction time, the percentage yields of oil obtained at a temperature of 80°C, 90°C, 100°C the percentage yield of oil obtained at different runs for fine and coarse sample. and the result shows that oil yield is high at 4.4% of the initial moisture content when water has not been added to the sample for both fine and coarse sample

The characterization/evaluation test conducted on the extracted oil to determine the physical and chemical properties of the extracted oil, shows that the oil pale yellow in color, with a diesel-like smell, density of 0.815kg/m³, melting point of 34 – 36°C, aponification value of 178.12ml/g, acid value of 4.719ml/g and also the iodine value.

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CHAPTER ONE

1.0 INTRODUCTION

1.10 Background to study

Roselle (*Hibiscus Sabdariffa* Linn) is an erect sparsely branched annual tree of 4.8m high which is cultivated for its economical importance. The stem of this variety is green or red while the leaves are green the crop also have other names other than the English name these are Roselle, sorrele, Sour-Soure, guinea land Jelly plant, Jelly Okra, Lemon bush and Florida Strawberry (Morton 1987) In French, it is called Oseille rouge, Orseille de guinea, in Portuguese it is called Vinagreira, in Dutch Zuring, in West Africa and near the East it is called Karkade and in Nigeria it is popularly known as Sobo seed. It contains about 17% edible oil.

Fats and oil are water insoluble substances of plants and animal origin. Fats and oil are readily soluble in either petroleum, chloroform and comparatively few of them are soluble in alcohol such as castor oil.

Fats and oil have been one of the main food sources for man that forms the integral part of the living matter (Okaka 1992). Fats and oil could be of animal, vegetable (plant) or marine in origin. Fats are saturated triglycerides which could be solid or semi-solid at room temperature oil are unsaturated triglycerides which are liquid at room temperature.

Oils are mainly of vegetable source (plants) and account for about 3/5 this of the world's consumption. The rest comes animal source. In the last twenty years there has been a steady decrease in the consumption of butter and lard which are animal source. The consumption of vegetable oil (vegetable source) has been on the increase. Vegetable oils are derived from the seeds of plants which grow in many parts of the world. but among the large variety available only few are commercially significant e.g. coconut oil, palm oil, palm kernel oil, soybean oil etc while Roselle seed oil which is the scope of this research is found among the neglected oilseed, others include, African Mango, African pear and cashew to mention just but a few. These edible oils are consumed in their natural liquid state.

1.2 Significant of the research work

In Nigeria there are many industries that produce vegetable oils by combination of extraction methods. The focus of such industries has direct on few vegetable seeds such as groundnut, palm fruit, palm kernel, cotton, sesame, melon and soya bean seeds.

One of the significant of this research work dwells on the extraction and characterization of vegetable oil from much neglected seeds, in particular on Roselle seed (*Hibiscus Sabdariffa* Linn). The neglected vegetable seed. (oil seeds) have been referred to as lesser know" oil seeds by Oke (1974). This includes African oil been , African mango, African bread fruit and Roselle seed.

And this will equally lead people knowing more of Roselle seeds and its oil content.

1.3 Objectives of the work

The objectives of this project therefore is to

Extract oil from Roselle seed using ethanol and di-ethyle ether as the solvent.

And to establish the effect of temperature, moisture content and extraction time on the oil yield

1.4 Justification

This project will look at the need to extract oil from the seed, which are always thrown away after production and the farmer, has taken the little he is going to use for the next planting season. This will contributes as a new source of oil production for usage at both in domestic and commercial level.

CHAPTER TWO

LITERATURE REVIEW

Oil and fats

Fats and oil are natural occurring esters of glycerol and fatty acids. They are soluble in non polar solvents such as carbon tetrachloride (CCL_4): Benzene (C_6H_6): Hexane ($\text{CH}_3(\text{CH}_2)_4\text{CH}_2$) and ether (CH_3CH_2)₂O. In other words, if a sample of plant or animal tissue is placed into a container along with the non polar solvent and then crushed the compound that dissolves in non polar (1964)

Fat and oil are sources of energy; they are most concentrated form of food energy contributing about 38kg/g over twice that of carbohydrates which contains 17kg of energy per grain.

Characteristics of fats and oil

Some very important properties of fats and oils form the basis for commercial standards and trading rule. These properties includes:- melting point, solidification characteristics. While others are iodine value, acid value, saponification value e.t.c.

Extraction of oils

raw material preparation

To ensure maximum oil yield using any method of extraction, the oil seed must be subjected to different pretreatment conditions.

Notable among the methods of preparation raw sample for oil extraction are:

Cleaning

Milling

Heat treatment

Flaking. (McGraw hill Encyclopedic Dictionary of method of extraction.vol.14P.484.)

a. Seed cleaning

The first step is removal of foreign materials such as dirt, stones, e.t.c using mechanical winnowing machine.

b. Milling of seed

This is the breaking down of the oil bearing seeds to the required particle size this is done mostly using laboratory burr mill.

c. Heating treatment

This involves subjecting the seeds to heating which then ruptures the oil bearing cells and makes flake's possible. When the seeds are subjected to heating it is referred to as cooking. . (McGraw hill Encyclopedia of method of extraction.vol.14P.484.)

Cooking consideration

Cooking can be regarded as one of the first steps in extraction process. It may be restricted to mechanical means of extraction. In carrying out cooking process, the cleaned, dehulled often to as prepared meal are transferred to sack cookers where they are cooked.

It is universally known that oil seeds yield more of oils readily to mechanical expression after cooking. But the complete explanation of why it is so is still lacking.

The primary objectives of the cooking may therefore be summarized thus; Luncin M.(1953)

- a. To coagulate the protein in the seed causing coalescence of oil droplets and making the seeds permeable to the flow of oil.
- b. To decrease the affinity of the oil for the solid surface of the seed so that the best possible yield of oil may be obtained when the seeds are subsequently pressed

The secondary objective includes:-

- a. Completion of breakdown of oil cells
- b. lowering the viscosity of oil to be expelled
- c. Moisture content adjustment of the meal to the optimum level for pressing.
- d. Sterilization of the seeds thus destroying enzymatic action.

Method of oil extraction

There are few methods readily available for oil extraction from oil bearing seeds. These methods include mechanical pressing thermal method and solvent extraction among others. Mechanical pressing or expression cannot be used where the oil content of the oil seeds is low as seen in corn.

Thermal method which involves heat treatment could cause some decomposition which would lower the oil quality. The last method solvent extraction does not have the disadvantages being possessed by the other two methods. Solvent extraction has its own disadvantages, notable are problem of solvent loss, additional cost incurred for desolventization and purification of

both the solvent and the raffinate. However, these

Thieme (1968) carried out test with carbon disulphide to extract oil from olive cakes report it as being undesirable because of its low boiling point (46.5⁰ C), low flash point, its toxicity, smell and its acidic taste. Christic (1982) in his oven analysis using chloroform, discovered that it was good solvent for lipids when used alone, but poor in extracting complex lipids from tissues. It may also promote the action of phsopolipase. D. when used to extract plant tissue (Kates, 1956).

The most widely used solvents for extraction are the light paraffinnic fractions especially the hexane, ethanol and di-ethyle ether type with boiling point range of 64-80⁰C; cyclohexame with boiling point range of 70 – 80⁰C is also used. Among the non-flammable solvents, trichloroethyne is popular with boiling point of 87⁰C. Its only disadvantage is that it is easier than water and thus cause in the cost of equipment used for ectarction.

In 1973, analyst Lewkowisth stated that petroleum ether is superior to and should be proffered to diethyl ether because it does not dissolve the oxidized acids of sulfurs. Di-ethyl ether extracts non-fatty materials and is susceptible to traces of moisture (Josylin, 1970). However, two scientist William (1976) and Wagen-Knochot (1957) stated that mixture of Chloroform and methanol in the ratio of 2:1 by volume will extract lipids more exhaustively from animal, plant or bacterial tissures than will most other solvent system. Petroleum ether, n-hexane, ethanol and di-ethyl can be considered to be the most popular solvent system. Other substitutes include benzene, heptanes and chloroform/methanol mixture 2:1. ethanol and di-ethyl ether are popular because.

They do not extract no-fat constituents such as starch and proteins.

They are cheap and readily available

They are not affected by traces of moisture.

Advantages of solvent extraction

The extraction rates using this process are usually higher than from mechanical methods and the oil in the residue is normally less than 2%. For oil seed (such as roselle seed) containing about 7-17%, it is not possible by expelling to reduce the oil content of the cake below 2% without thermal degradation occurring. This process has being able to be achieved by direct solvent extraction. Karl Mattil (1993)

Disadvantages of solvent extraction

The main problem involved in this method of extraction is the extra cost for separation of the solvent and the extract. Most of the solvents used are highly volatile and flammable. Therefore, there is risk of explosion (Duke, 1983).

Solvents vary considerably in chemical and physical properties which affects their performance.

Stability

Extraction solvent should be able to withstand heat, light and water without degradation. Stability is required to prevent contamination of meal and oil with potentially hazardous decomposition products.

Recoverability

The extraction solvent normally must be recovered from the extract steam and also from raffinate steam in an extraction process, since distillation is often used, the relative volatility of the extraction solvent to non-solvent components should be significantly greater or less than unit.

Toxicity

Low toxicity from solvent vapour inhalation or skin contact is preferred because of potential exposure during repair of equipment or while connection are being broken after a solvent transfer.

Solubility

A low solubility of extraction in raffinate generally leads to high relative volatility in raffinate stripper on a low solvent loss if the raffinate is not desolventized. If the Oils is not soluble in the solvent, there would be no extraction. A low solubility of feed solvent in the extract leads to high relative separation and generally to low solute recovery costs.

Selectivity

It is desirable to use solvents which extracts only the triglycerides and leaves phosphatides, free fatty acids, waxes and pigment in the meal.

Density

The difference in density between the two liquid phases in equilibrium affect contaminant flow as well as the coalescence rates (Adeyemo, 2003).

Non-reactivity with equipment

The solvent should not react with equipment. Some solvents are corrosive to piping and metal components and soluble metallic ions, which can cause discoloration of the extract.

Effect of moisture content on oil extraction

While continuous extraction by percolation of flaked roselle seed of different moisture content by ethanol showed variation at the end of 60 minutes as great as 5.47% residual oil, the variation over the range of practical plant operation conditions are too small to be a significant factor. Moisture affects the rate of extraction of roselle seed and cotton seed meal flakes by trichloroethylene less than the rate of hexane. Some presence of moisture increases the weight of raw sample thereby creating further processing difficulties. Therefore moisture is needed to remove the moisture content since the dryness of the seed makes it easy for dehauling of the seed coat and the essence of reducing the moisture content is to increase the oil yield per weight. Eldridge, (1969) of bibliography on the solvent extraction.

Effect of temperature and pressure on oil extraction

Sunflower seed was extracted using supercritical carbon dioxide in a semi-continuous flow extraction with temperature range of 313-343K and pressure range 20-40Mpa. The extraction rate increased with temperature up to about 28Mpa. Thus higher temperature favored the extraction rate because of the competitive effect of solvent density change and solute vapor presence with the variation of temperature and pressure resulting in the cross-over effect of solubility of oil in supercritical carbon dioxide. by Roy bhepesh (1964)

CHAPTER THREE

3.0 MATERIALS AND METHOD

3.1 Source of seed

Roselle seeds for this project work was obtain from local farmers within Bida. The varieties of the Roselle seeds that were used are hibiscus sabdariffa variety which is characterized by a relatively debit at the seed edges

3.1.1 Seed cleaning

Cleaning of the seed was carried out using mechanical winnowing machine. To achieve proper cleaning, further cleaning was done by hand picking to remove stones, immature seeds from the bulk and other foreign bodies, which were still remaining after mechanical cleaning.

3.1.2 Grinding/size reduction of the seed

The sample portion were grinded using electrically operated grinder or blender. to achieve the require sizes, grinding was done for three good times.,

3.2 Screening/sieving

Tyler's sieve and ro-tab machine

A set of woven wire cloth sieves of frame diameter 20.3mm was used for particle size analysis.

The ro-tab machine is electrically operated type.

Sieves size of 2.376mm, 1.77mm and 1.128mm were used to obtain coarse samples while sieve sizes of 600 μ m, 300 μ m, 150 μ m and 75 μ m were used to obtain fine samples.

3.2.1 Weighing balance

Digital weighing balance was used for all weighing determination of the samples and mass of oil obtained after oil extraction.

3.2.2 Soxhlet apparatus unit

Solvent extraction using the soshlet apparatus unit and di-ethylether and ethanol was used to carryout the test, the soshlet apparatus consist of condenser, conversion flask, sample chamber, heating mantle to heat the mixture, thimble Colton wool, retort stand round bottom flask were the mixture are held during heating

3.2.3 Apparatus used

- Oven
- Stop watch
- Refrigerator

A standard oven was used for moisture content determination and for heating the samples. Stopwatch was used to monitor the extraction time and heating time. A refrigerator was used to equilibrate the samples after the samples moisture content has been adjusted to the desired level for 24 hr minimum.

3.2.4 Test procedure

Clean Roselle seeds were grinded, the samples were graded using particle size analysis in which standard Tyler's seed size of 2.36mm, 1.70mm and 1.18mm were used to obtain coarse samples while sieve sizes of 600 μ m, 300 μ m, 150 μ m and 75 μ m;with pan inclusive were used to obtain fine samples. After obtaining the fine and coarse samples, 40g of fine samples were weighed into 36 places while 40g of coarse were weighed into 36 places.

Moisture content of both i.e. the fine and the coarse were determined using oven dry method on dry basis. Moisture content of fine sample of each in 36 places were increased with some quantity of water while moisture content of coarse sample were also increased by same way. After Spraying evenly on the samples, the calculated quantity of water did the conditioning in a refrigerator for 24hr. Each conditioned fine and coarse samples were placed in a container and placed in the oven for stated period of 10,20 and 30 minutes heating time, and 80,90 and 100 heating temperature. Immediately the heating times stated were reached, the oven was put off and the sample in the container was removed from the oven and then put into a soshlet apparatus for oil extraction.

3.2.5 Solvent extraction

After the conditioning time, heating time and heating temperature, the samples were put into the soshlets apparatus for oil extraction using di-ethyl ether for fine sample and ethanol for coarse sample. The unit was set up and extraction produre followed 40g of finely grinded and coarse sample were wrapped with filter paper and inserted into the thimble holder. The thimble was connected to a 250cm quick fit weghed round botton flask, containing 200ml solvent. The system was completed by connecting the thimble condenser with water as a coolant. The top of

the condenser condenses the vaporized solvent into the thimble holder. This give room for proper contact of the solvent with the sample which result in several refluxes of the solven.

The mantle heater was put on and the system allowed to run for 5hours with several refluxes. On the expiration of 5hour, the thimble holder was removed. The solvent was the distilled off recovered leaving the oil in the flask which was the weighed after cooling and necessary data was taken. Oil yield in grams were determined and recorded. The percentage oil yields were determined using;

$$Y = \frac{AOY}{W_s} \times 100 \text{ -----(2)}$$

Where Y = percentage oil yield

AOY =Average oil yield (mean)

Ws =weight of samples

Note Ws =40g.

Kirk Othimer(1983)

3.0 Characterization of extracted oil

Parameter considered in this chapter are grouped into chemical and physical test

3.1.1 Chemical test

Saponification value (SV)

Acid value (AV)

Iodine value (IV)

Peroxide value

3.1.2 Physical Tests

Specific gravity

Discosity

Boiling point

Melting point

PH

Acid value

Aim – To determine the acid value of Roselle seed

3.1.3 Apparatus

Conical flask

Retort & land with clamp

Burette, - pipette, - filler paper – funnel

Reagents

0.1N Na OH

Diethyl ether, ethanol

Phenolphthalein Indicator

Sample.

Roselle seed oil.

3.1.4 Procedure

The value is obtained by mixing equal value of diethyl ether and ethanol.

20ml of the mixture was pipeltd and mixed a lamon quantity of oil in a conical flask

the mixture was interacted against a 0 IN normal sodium hydroxide in the burette using phenolphthalein as indicator the mixture gave pink blouration at the end point.

The acid value is given by the relation below

$$A.V = \frac{56.1 N}{W}$$

Where A.V = Acid value and N and V are normality and volume of N aol+ used respectively W = weight of oil (g) the necessary calculations are shown in apperdia.

Note 56.1 = molar mass expressed in gram / mo / NaoH

3.1.5 Iodine value (I V)

Aim – to determine the iodine value of Roselle seed oil

Apparatu

Retart stand with clamp, burette, pipette, conical flask

Filler papers, finnel

Reagents

CCL+ - OIIL WIJIS solution

% potassium iodine solution

Starch indicator

Distilled water

Sodium thiosuphale

Sample

Roselle seed oil

3.1.0 Procedure

The weighted sample of oil was mixed in two conical flasks with its ml carbon tetrahlonto . 25ml or w y's solution from pipelte was added to each of the mixture. This was closed and allowed to

stand in the dark at 20°C for one hour. This was done to take care of any oxidized material present in the sample mixture at the end of one hour, to each of the flask was added 20ml potassium iodine solution and approximately 150ml distilled water. The solution was titrated with sodium thiosulphate solution (0.1N) using freshly prepared starch as indicator. The experiment was repeated under the same conditions without Roselle seed oil to serve as blank (control). The starch indicator should be added towards the end of the titration for easy determination of Iodine value was calculated using the relationship below.

$$V = \frac{12.69 N (V_2 - V_4)}{W}$$

W

Where V1 volume of sodium thio-sulphate used in the test

V2 - Volume of sodium thio-sulphate in blank

N - Normality of thio-sulphate

W2 - Average weight of oil used

Detailed calculation are found in appendix

3.2.1 Saponification value

Aim - to determine the saponification value of Roselle seed oil

3.2.2 Apparatus.

Heating mantle.

3.2.3 Reagents

Ethanol

potassium hydroxyl solution

phenolphthalein (indicator)

0.5 N IRI

3.2.4 Sample

Roselle seed

3.2.5 Procedure

50ml of 0.5 N ethanol to potassium hydroxide (mixture of ethanol and KOH in equal proportion) was measured but two conical flasks. A and the other B. flask A contained weighed Roselle seed oil and flask B was without oil. Thus serving as a control.

The two flasks were heated under the ethanol oil mixes completely. About time drops of

phenolphthalein indicator was added to the solution. The process was repeated to determine the average weight of oil used. Blank titration was also carried out under the same condition saponification value can be determined using the relationship below

$$S.V = \frac{56.N (V_2 - V_1)}{W}$$

W = Average weight of oil (g)

N = Normality of hydrochloric acid

V₁ = Volume of HCL in test

V₂ = Volume of HCL in blank

3.3.0 Peroxide value (PV)

Aim - to determine the peroxide value of Roselle oil

3.3.1 Apparatus

Conical flasks

retort stand with clamp

filter paper

burette

3.3.2 Reagents

0.002N Sodium thiosulphate

Glacial acetic acid

Chloroform

Potassium iodide.

Starch indicator.

3.3.3 Sample

Roselle seed oil

3.3.4 Procedure

Pour in to the burette 0.002 N Sodium thiosulphate solution A mixture of glacial acetic acid and chloroform in the ratio of 2:1 was added to the flask containing weighed sample of oil, few drops of starch indicator was added to the mixture and titration carried out the solution gives a yellow colouration at the beginning and turns colourless at the end point.

The experiment was repeated for blank titration under the same conditions, but in the absence of oil. Peroxide value can be calculated using the relationship below.

$$P v = \frac{1000 (v_1 - v_2) N}{W}$$

W = weight (g) oil

V1 = volume (ml) sodium thiosulphate used in test

V2 = volume (ml) sodium thiosulphate in blank

N = normality of sodium thiosulphate.

CHAPTER FOUR

4.0 RESULT AND DISCUSSION

4.1.1 Result

TABLE 4.1: Average mean and average percentage mean yield of fine sample = 40g

Quantity of water added	Mc%	Heating time (Mins.)	Heating temp. (°c)	Average mean oil yield(cm ³)	Average mean % yield
M ₁ = 0	4.4	10	80	3.5	8.74
		20		5	12.5
		30		12.1	30.25
M ₁ = 0		10	90	1.25	3.12
		20		2.8	5.7
		30		0.25	0.625
M ₁ = 0		10	100	2.8	7
		20		4.7	11.75
		30		5.25	13.12
M ₂ = 0.38010 liters	5.3	10	80	4.8	12
		20		2	5
		30		4.7	11.75
M ₂ = 0.38010 liters		10	90	7.9	19.75
		20		6.9	17.2
		30		2.8	5.7
M ₂ = 0.38010 liters		10	100	3.1	7.75
		20		3	7.5
		30		5.2	13
M ₃ = 0.68084	6.0	10	80	3	7.5
		20		3	7.5

liters		30		3.1	7.75
M ₃ = 0.68084 liters		10 20 30	90	3.8 2.5 4.9	9.8 6.25 12.25
M ₃ = 0.68084 liters		10 20 30	100	3.9 2.3 6.8	9.75 5.75 17
M ₄ = 1.11828 liters	7.0	10 20 30	80	1.8 4.1 1.5	4.5 10.25 3.75
M ₄ = 1.11828 liters		10 20 30	90	4.5 1.5 5	11.25 3.75 12.5
M ₄ = 1.11828 liters	7.0	10 20 30	100	4.4 5.7 3.7	11.00 14.25 9.25
Initial moisture content, no Heat temp. And addition of H ₂ O				3.1	7.75

TABLE 4.2: : Average mean and average percentage mean yield of coarse sample = 40g each

Quantity of water added	Mc%	Heating time (Mins .)	Heating temp. (°c)	Average mean oil yield (cm ³)	Average mean % yield
M ₁ = 0	5.14	10	80	1.0	2.5
		20		1.5	3.75
		30		4.5	11.25
M ₁ = 0	5.14	10	90	5.2	13
		20		0.4	1.0
		30		2.3	5.75
M ₁ = 0	5.14	10	100	2.2	5.5
		20		0.1	0.25
		30		11	27.5
M ₂ = 0.452ml	6.2	10	80	1.0	2.5
		20		4.8	12
		30		6.8	17
M ₂ = 0.452ml	6.2	10	90	4.1	10.25
		20		0.9	2.25
		30		4.4	11.0
M ₂ = 0.452ml	6.2	10	100	6.6	16.5
		20		3.1	7.75
		30		3.0	7.5
M ₃ = 0.887921	7.5	10	80	5.7	14.25
		20		2.1	5.25

iters		30		1.2	3.00
M ₃ = 0.887921 iters	7.5	10 20 30	90	3.0 6.2 2.0	7.5 15.5 3.5
M ₃ = 0.887921 iters	7.5	10 20 30	100	1.4 3.0 3.3	3.5 7.5 8.25
M ₄ =1.3784 Liters	8.3	10 20 30	80	3.0 3.0 2.0	7.5 7.5 5 7.5
M ₄ =1.3784 Liters	8.3	10 20 30	90	3.4 5.1 1.1	8.5 12.75 2.75
M ₄ =1.3784 liters	8.3	10 20 30	100	4.3 3.3 1.9	10.75 8.25 4.75
Initial moisture content, no Heat temp. And addition of H ₂ O				6.2	15.5

TABLE 4.3: Analysis Of Variance For The Split-Split Plot Design

Source of variation	Degree of freedom	Sum of squares	Mean square	Computed F- value	Table value of F at 5%
Main plot analysis	2	2.80	1.40	1.16 ^{NS}	5.14
Replication					
Moisture (A)	3	10.02	3.34	2.76 ^{NS}	4.76
Error (a)	6	7.27	1.21	-	
Sub-Sub plot Heating time (B)	2	23.04	11.52	37.16 ^{**}	3.63
A x B	6	66.91	11.51	35.97 ^{**}	2.74
Error (b)	16	4.96	0.31	-	
Sub-Sub plot Heating Temperature (c)	2	6.75	3.38	9.14 ^{**}	3.20
A x C	6	184.13	30.7	9.03 ^{**}	2.31
B x C	4	38.66	9.67	26.14 ^{**}	2.58
A x B x C	12	170.93	14.24	38.49 ^{**}	1.97
Error (c)	48	17.69	0.37	-	
Total	107	533.16	-	-	

Cv (a) =27.5%, Cv (b) =13.9%, Cv (c) = 15.2%

Note Cv means coefficient of variation

Ns means not significant

TABLE 4.4: Analysis Of Variance For The Split-Split Plot Design

Source of variation	Degree of freedom	Sum of squares	Mean square	Computed F- value	Table value of F at 5%
Main plot analysis Replication	2	0.49	0.25	1.92 ^{NS}	5.14
Moisture (A)	3	14.12	4.71	36.23 ^{**}	4.76
Error (a)	6	0.78	0.31	-	
Sub-Sub plot Heating time (B)	2	19.03	9.52	47.60 ^{**}	3.63
A x B	6	156.40	26.07	130.35 ^{**}	2.74
Error (b)	16	3.22	0.20	-	
Sub-Sub plot Heating Temperature (c)	2	8.38	4.19	24.65 ^{**}	3.20
Ax C	6	31.37	5.23	30.76 ^{**}	2.31
B x C	4	41.11	10.28	60.47 ^{**}	2.58
A x B x C	12	265	22.15	130.29 ^{**}	1.97
Error (c)	48	8.24	0.17	-	
Total	107	548.91	-	-	

Cv (a) =11.1%, Cv (b) =138%, Cv(c) = 12.7%

** Means significant at 5%

Ns=not significant

TABLE 4.3 Physical Properties Determination

S/N	ANALYSIS	STANDARD	ANALYSED
1	Specific gravity	0.817	0.762
2	Viscosity	60kg/m	54.6kg/m
3	Boiling point	100°C	110.5°C-115°C
4	Melting Point	35-36°C	35-40°C
5	PH		8
6	Colour	Whitish	Pale yellow
7	Density	0.817kg/m ³	0.762kg/m ³

TABLE 4.4 Chemical Properties Determination

S/N	ANALYSIS	STANDARD	ANALYSED
1	Saponification value	179.40mol/g	178.12mol/g
2	Acid value	6.01mol/g	4.719mol/g
3	Iodine value	79.00mol/g	75.9mol/g
4	Peroxide value	8.83	6.67

4.1.2 DISCUSSIONS

From table 4.3, analysis of variation for split-split plot design

When f -tabulated is greater than f -calculated then we said it is not significant, so therefore, from the result in table 4.3, in the main plot analysis replication, f -tabulated is greater than f -calculated at 5% and so therefore it is not significant at the main plot analysis replication.

It was also observed that f -tabulated is less than f -calculated in moisture content, heating time and heating temperature. This makes moisture content, heating time and heating temperature significant at 5% in both fine and coarse sample.

4.1.3 Effect of moisture content on the % oil yield

It was observed that for both the fine and coarse samples the percentage oil yield was highest at the initial moisture content of 4.4 % and 5.14% dry basis respectively from those samples heated at 80 °C for fine sample and 100°C for coarse sample for a period of 30 mins. This could be attributed to the fact that no moisture has been added and the solvent could be able to penetrate the oil pore space to drain out the oil.

4.1.4 Effect of heating temperature on the % oil yield

It was observed that as the heating temperature increases on the addition of moisture the samples, the samples became dryer and coagulation occurs. This may be the reason why the oil content obtained for the fixed extraction time, for the different temperature level were low as a result of the solvent not been able to adequately penetrate the coagulated portion of the sample.

The extraction time will also be extended in order to improve on the oil recovery from the sample used.

4.1.5 Color variation and percentage oil yield

There was a distinct color variation of the oil samples which was due to the heating temperature and the heating time, the higher the heating temperature and heating time, the darker the color of the oil yield, because, during heating of the samples, the samples have eventually turned dark brown as a result of moisture lost, this also reflects in the oil yield.

Some of the oil yields are very light in color due to the temperature and moisture introduced. Color variation is associated with heating temperature and heating time of the sample.

4.2.0 Effect of the heating time on the percentage oil yield.

The heating time also has effect on the yield, the higher the heating time with some moisture content, the higher the oil yield. While the lower the heating time with some moisture content, the lower the oil yield. The heating time has effect on the oil yield by allowing the sample to be heated for a short or a long time.

4.2.1: Effect of particle size

The higher the particle size, the higher the penetration rate and visa viz.

The fine particles are not easily penetrated by the solvent, their extraction is slower since the particle size are fine, they clumps together and slows the penetration rate, with the help of heating temperature and heating time, these dries up the moisture in the samples, thereby increasing the rate of solvent penetration into the samples. The coarse sample has larger particles when compared with the fine, and this makes their penetration rate faster. The finer the particle size, the higher the rate of the surface area exposure.

4.2.2 Characterization of the oil

The characterization of the oil was carried out to determine the following parameters; the acidic value, the free fatty acid value, the specific gravity, the peroxide value, the iodine value, the viscosity, the saponification value, the Ph, the boiling and melting point.

From the results (table 4c), the acid value was got as 5.01ml/g while the free fatty acid was got to be 1.29%. The acid value which is low for the raw sample gives the oil the advantage of being edible. The result obtained from the FFA indicates a low fatty acid content of the oil due to the fresh state of the oil. This supports the literature that freshly prepared animal oils and vegetable oils are relatively devoid of free fatty acids.

However, during storage, fats may become varied due to peroxide formation at the double bonds by atmospheric oxygen and hydrolysis by micro organisms with the liberation of free fatty acids (FFA). The saponification value of oil gives an indication of the nature of the fatty acids in

the fat. From table 4.C, the saponification value was obtained as 178.12, this shows that the oil is of short carbon chain since high acid is liberated per gram of fat hydrolysed. This will now require a large amount of KOH to neutralize the liberated acid; this is because the oil has a high degree of hydrolysis.

The physical state of the oil is liquid, this further conforms that the oil is of short chain carbon. Short chain length and unsaturated nature enhances the fluidity of the fatty acids (Okaka et al, 1992). The oil therefore contains a high percentage of unsaturated acid and soaps made from such oil is soluble in tepid water and therefore can be advantageously used cleansing at moderate temperature, a requisite for the washing of delicate surface and fibres. The iodine value is the quantity of iodine absorbed per gram of oil. It is an index of the degree of unsaturation (double bonds) of the oil. Generally, the greater the degree of unsaturation, the higher the iodine value, the tendency for the fat/oil to rancid.

From table 4.C the iodine value was got as 76.55ml/g which fall within the range of less than 90 (< 90). Therefore the oil is classified as non-drying oil and can be used in food, soap and pharmaceuticals. It can equally be used as a lubricant. The mean ph of the oil was got as 8 which indicate that the oil varies between alkaline and neutral points. Thus the oil can be said to be either alkaline or neutral by + 1.

The boiling point (BP) and the melting point (MP) was found to be between the range of 110°C -120°C and 60°C-80°C respectively. The colour of the oil obtained is golden yellow, pure fatty acids and their glycosides are colourless and therefore do not have spectral properties in the visible range. The colour of the neutral fats is due to the presence of small amounts of fat-soluble pigments such as carotenoids and chlorophyll or sometimes due to oxidation and polymerization products of the fatty acids. Therefore, in the case of the coloured oil got in this work. The oil pigments were extracted along with the fat. The odour of the oil resembles that of the seed. This is because other components of the seeds still exist the oil, but in minute quantity. Another important characteristics of oil is the viscosity is defined as the resistance to flow. Temperature is one important factor that affects viscosity.

The viscosity variation with temperature can be due to the fact that in liquids, momentum

exchange due to the molecular activity is small compared to the cohesive force between the molecules. Thus, viscosity is primarily dependent on the magnitude of the cohesive force that tend to keep adjacent molecules in a fixed position relative with increase in temperature, the knowledge of viscosity can be useful when dilution prior to use is required as well as in the estimation of diffusivity and in determining flow pattern. The amount of viscosity decrease depends on both the chemical nature of the oil and the magnitude of temperature changes. The viscosity, of this oil can be used as a lubricant due to its low viscosity index. The viscosity of an oil can be increased by hydrogenation. The specific gravity of the oil was found to be 0.762g this shows that the oil is lighter than water and thus floats on it.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1.1 Conclusion

It was discovered that the higher the temperature for moisture content 1, for the coarse sample, the higher the oil yield. The heating of the coarse sample also reduce the rate of the solvent penetration, high heating temperature darkens the color of the oil.

The fine sample has the highest oil yield at moisture content of 4.4 %, db and temperature of 80°C for 30mins heating time.

Heating is a very important factor in extraction. The higher the heating temperature the darker the extract. Introduction of moisture to increase the moisture content ha effect on the oil yield of both samples that is the fine and the coarse samples. The best result was obtained at the moisture content were moisture has not been introduced. The initial moisture content at different heating temperatures and heating time has the highest extract.

5.1.2 Recommendation

Based on the result, the initial moisture content of the sample is best for extraction.

Coarse sample has higher rate of extraction when subjected to high heating temperature and high heating time.

The fine samples have their higher rate of extraction when they are subjected to lower temperature and higher heating time.

To achieve a lighter color in the yield, lower heating temperature and heating time is required.

To optimize yield during extraction oil from local seeds with Roselle seed as a case study, the following recommendations, if strictly adhered to, will be of help.

Appropriate safety facilities should be adopted. These include use of apron (laboratory coats), handgloves, goggles etc.

Seeds should be oven-dried to remove as much moisture as possible.

Seeds should be properly grinded while raw to provide much surface area for solvent/solute contact

A sensitive weighing balance should be used to minimize errors due to weight.

Large enough value of solvent should be employed.

Solvent of extraction should be employed, since it extracts oil even below 0.5% when compared with 4-67% by mechanical expression.

Ethanol solvent is recommended, since it gives the highest yield among the others used in this work.

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APPENDIX

From table 3.3

$$\text{Correction factor (cf)} = G = \frac{(350.0)^2}{rabc} = 1138.15$$
$$\frac{(3)(4)(3)(3)}$$

Here $r=3, a=4, b=3, c=3$

$$\text{Sum of square total (SST)} = \sum X^2 - (C.F)$$
$$= (3.50)^2 + (3.00)^2 + \dots + (3.60)^2 - 1730.00$$
$$= 2263.16 - 1730.00 = 533.16$$

From Table 3.2

$$\text{Sum of squares Replication (SSR)} = \frac{\sum R^2}{abc} - C.F$$

$$= \frac{(145.33)^2 + (150.48)^2 + (136.44)^2}{(4)(3)(3)} - 1730.00$$
$$= 1732.80 - 1730.00 = 2.80$$

$$\text{Sum of squares (moisture contents) or SSA} = \frac{\sum A^2}{RBC} - C.F$$

$$= \frac{(112.95)^2 + (122.80)^2 + (99.90)^2 + (96.60)^2}{(3)(3)(3)} - 1730.00$$

$$= 1746.20 - 1730.00 = 16.20$$

$$\text{Sum of squares Errors (a)} = \frac{\sum (RA)^2}{BC} - C.F - SSR - SSA$$
$$\text{SSE(a)}$$

$$= 1750.09 - 1730.00 - 10.02 = 10.07$$

From Table 3.4

$$\text{Sum of square of Heating time (SSB)} = \frac{\sum B^2}{Rac} - C.F$$

$$= \frac{(134.25)^2 + (130.50)^2 + (167.5)^2}{(3)(4)(3)} - 1730.00$$

$$= 1753.04 - 1730 = 23.04$$

Sum of squares of moisture and Heating time (SSAB)

$$= \frac{\sum (AB)^2}{RC} - C.F - SSA - SSB$$

$$= \frac{(22.65)^2 + (37.50)^2 + (49.40)^2 + (35.70)^2 + (39.70)^2 + (32.10)^2 + (23.40)^2 + (44.40)^2 + (32.10)^2 + (33.90)^2 + \dots + (30.60)^2}{(3)(3)} - 1730.00 - 10.02 - 23.04$$

$$= 1829.97 - 1730.00 - 10.02 - 23.04 = 66.91$$

Table 3.4

$$\text{SS Error (b)} = \frac{\sum (RAB)^2}{C} - C.F - SSR - SSA - SSE(a) - SSB - SSAB$$

$$= \frac{(7.50)^2 + (7.15)^2 + \dots + (9.70)^2}{3} - 17,300 - 2.80 - 10.02 - 7.27 - 23.04 - 66.91$$

$$= 1845.00 - 1730.00 - 2.80 - 10.02 - 7.27 - 23.04 - 66.91 = 4.96$$

From Table 3.5

$$\text{Sum of squares of Heating Time (SSC)} = \frac{\sum C^2}{Rab} - C.F$$

$$= \frac{(145.80)^2 + (132.30)^2 + (154.15)^2}{(3)(4)(3)} - 1730.00$$

$$= 1736.75 - 1730.00 = 6.75$$

$$= \text{SSAC} = \frac{\sum (AC)^2}{Rb} - C.F - \text{SSA} - \text{SSC}$$

$$= \frac{(61.80)^2 + (12.90)^2 + \dots + (4.440)^2}{(3)(3)} - 1730 - 10.30 - 6.75$$

$$= 1930.90 - 1730.00 - 10.02 - 6.75 = 184.13$$

From Table 3.5

$$\text{SSBC} = \frac{\sum (BC)^2}{Ra} - C.F - \text{SSB} - \text{SSC}$$

$$= \frac{(39.30)^2 + (52.35)^2 + \dots + (64.45)^2}{(3)(4)} - 1730.00 - 23.04 - 6.75$$

$$= 1798.45 - 1730.00 - 23.04 - 6.75 = 38.66$$

From Table 3.6

$$\text{SSABC} = \frac{\sum (ABC)^2}{r} - C.F - \text{SSA} - \text{SSB} - \text{SSC} - \text{SSAB} - \text{SSAC} - \text{SSBC}$$

$$= \frac{(10.50)^2 + (3.75)^2 + \dots + (11.10)^2}{3} - 1730.00 - 10.02 - 23.04 - 6.75 - 66.91 - 184.13$$

$$- 38.66$$

$$= 2230.44 - 1730.00 - 10.02 - 23.04 - 6.75 - 66.91 - 184.13 - 38.66 = 170.93$$

$$\text{SS Error (C)} = \text{SSTotal} - (\text{sum of all other sum of squares})$$

$$= 533.16 - 2.80 - 10.02 - 7.27 - 23.04 - 66.91 - 4.96 - 6.75 - 184.13 -$$

$$38.66 - 170.93 = 17.69$$

COMPUTATION OF THE MEAN SUM OF SQUARES FOR EACH SOURCE OF VARIATION

Mean Square Replication = $\frac{SS_{\text{Replication}}}{r-1}$

$$\frac{2.80}{3-1} = \frac{2.80}{2} = 1.40$$

$$MSA(\text{moisture}) = \frac{SSA}{a-1} = \frac{10.02}{4-1} = \frac{10.02}{3} = 3.34$$

$$MSE_{\text{Error (a)}} = \frac{SSE_{\text{Error(a)}}}{(r-1)(a-r)} = \frac{7.27}{(3-1)(4-1)} = \frac{7.27}{(2)(3)} = 1.21$$

$$MSB(\text{Heating time}) = \frac{SSB}{b-1} = \frac{23.04}{2} = 11.52$$

$$MSAB = \frac{SSAB}{(a-1)(b-1)} = \frac{66.91}{(4-1)(3-1)} = \frac{66.91}{(3)(2)} = 11.15$$

$$MSE_{\text{Error (b)}} = \frac{SSE_{\text{Error(b)}}}{a(r-1)(b-1)} = \frac{4.96}{(4)(3-1)(3-1)} = \frac{4.96}{4 \times 2 \times 2} = \frac{4.96}{16} = 0.31$$

$$MSC = \frac{SSC}{C-1} = \frac{6.75}{3-1} = \frac{6.75}{2} = 3.38$$

$$MSAC = \frac{SSAC}{(a-1)(C-1)} = \frac{184.13}{(4-1)(3-1)} = \frac{184.13}{(3)(2)} = 30.7$$

$$MSBC = \frac{SSBC}{(b-1)(c-1)} = \frac{38.66}{(3-1)(3-1)} = \frac{38.66}{(2)(2)} = 9.67$$

$$MSABC = \frac{SSABC}{(a-1)(b-1)(c-1)} = \frac{170.93}{(4-1)(3-1)(3-1)} = \frac{170.93}{(3)(2)(2)} = 14.24$$

$$MSE_{\text{Error}(C)} = \frac{SSE_{\text{Error}(c)}}{ab(r-1)(c-1)} = \frac{17.69}{(4)(3)(3-1)(3-1)} = \frac{17.69}{(4)(3)(2)(2)} = 0.37$$

COMPUTATION OF THE F-VALUE FOR EACH EFFECT

$$F(A) = \frac{MSA}{MSE_{\text{Error}(a)}} = \frac{3.34}{1.21} = 2.76$$

$$F(B) = \frac{MSB}{MSE_{\text{Error}(b)}} = \frac{11.52}{0.31} = 37.16$$

$$F(A \times B) = \frac{MSAB}{MSE_{\text{Error}(b)}} = \frac{11.15}{0.31} = 35.97$$

$$F(C) = \frac{MSC}{MSE_{\text{Error}(C)}} = \frac{3.38}{0.37} = 9.14$$

$$F(AC) = \frac{MSAC}{MSE_{\text{Error}(C)}} = \frac{3.34}{0.37} = 9.03$$

$$F(BC) = \frac{MSBC}{MSE_{\text{Error}(C)}} = \frac{9.67}{0.37} = 26.14$$

$$F(ABC) = \frac{MSABC}{MSE_{\text{Error}(C)}} = \frac{14.24}{0.37} = 38.49$$

From Table 3.7

$$\text{Correction factor (cf)} = \frac{G^2}{rabc} = \frac{(350.0)^2}{(3)(4)(3)(3)} = 1138.15$$

Where $r=3, a=4, b=3, c=3$

$$\begin{aligned} \text{Sum of square total (SST)} &= \sum X^2 - (C.F) \\ &= (0.8)^2 + (1.3)^2 + \dots + (1.4)^2 - 1138.15 \\ &= 1687.06 - 1138.15 = 548.91 \end{aligned}$$

From Table 3.29

$$\text{Sum of squares Replication (SSR)} = \frac{\sum R^2}{abc} - C.F$$

$$\begin{aligned} &= \frac{(116.70)^2 + (119.90)^2 + (114.00)^2}{(4)(3)(3)} - 1138.15 \\ &= 1138.64 - 1138.15 = 0.49 \end{aligned}$$

$$\text{Sum of squares (moisture A) or SSA} = \frac{\sum A^2}{rbc} - C.F$$

$$= \frac{(85.7)^2 + (104.1)^2 + (79.5)^2 + (81.3)^2}{(3)(3)(3)} - 1138.15$$

$$= 1152.27 - 1138.15 = 14.12$$

$$\text{Sum of squares Errors (a)} = \frac{\sum (RA)^2}{bc} - C.F - SSR - SSA$$

$$= 1153.54 - 1138.15 - 0.49 - 14.12 = 0.78$$

From Table 3.10

$$\begin{aligned}\text{Sum of square of Heating time (SSB)} &= \frac{\sum B^2}{rac} - C.F \\ &= \frac{(124.3)^2 + (95.8)^2 + (130.5)^2}{(3)(4)(3)} - 1138.15 \\ &= 1157.18 - 1138.15 = 19.03\end{aligned}$$

Sum of squares of moisture and Heating time (SSAB)

$$\begin{aligned}&= \frac{\sum (AB)^2}{rc} - C.F - SSA - SSB \\ &= \frac{(26.8)^2 + (5.50)^2 + \dots + (15.0)^2}{(3)(3)} - 1138.15 - 14.12 - 19.03 \\ &= 1327.70 - 1138.15 - 14.12 - 19.03 = 156.40\end{aligned}$$

Table 3.11

$$\begin{aligned}\text{SS Error (b)} &= \frac{\sum (RAB)^2}{C} - C.F - SSR - SSA - SSE(a) - SSB - SSAB \\ &= 1332.19 - 1138.15 - 0.49 - 14.12 - 0.78 - 19.03 - 156.40 = 3.22\end{aligned}$$

From Table 3.12

$$\begin{aligned}\text{Sum of squares of Heating Time (SSC)} &= \frac{\sum C^2}{rab} - C.F \\ &= \frac{(105.1)^2 + (115.9)^2 + (129.6)^2}{(3)(4)(3)} - 1138.15 \\ &= 1146.53 - 1138.15 = 8.38\end{aligned}$$

$$= \text{SSAC} = \frac{\sum(\bar{AC})^2}{rb} - \text{C.F.} - \text{SSA} - \text{SSC}$$

$$= \frac{(20.5)^2 + (25.3)^2 + \dots + (28.5)^2}{(3)(3)} - 1138.15 - 14.12 - 8.38$$

$$= 1192.02 - 1138.15 - 14.12 - 8.38 = 31.37$$

From Table 3.13

$$\text{SSBC} = \frac{\sum(\bar{BC})^2}{ra} - \text{C.F.} - \text{SSB} - \text{SSC}$$

$$= \frac{(32.1)^2 + (48.7)^2 + \dots + (57.6)^2}{(3)(4)} - 1138.15 - 19.03 - 8.38$$

$$= 1206.67 - 1138.15 - 19.03 - 8.38 = 41.11$$

From Table 3.14

$$\text{SSABC} = \frac{\sum(\bar{ABC})^2}{r} - \text{C.F.} - \text{SSA} - \text{SSB} - \text{SSC} - \text{SSAB} - \text{SSAC} - \text{SSBC}$$

$$= \frac{(3.0)^2 + (17.2)^2 + \dots + (5.7)^2}{3} - 1138.15 - 14.12 - 19.03 - 8.38 - 156.40 - 31.37 - 41.11$$

$$= 1674.33 - 1138.15 - 14.12 - 19.03 - 8.38 - 156.40 - 31.37 - 41.11 = 265.77$$

SS Error (C) = SSTotal - (sum of all other sum of squares)

$$= 548.91 - 0.49 - 14.12 - 0.78 - 19.03 - 156.40 - 3.22 - 8.38 - 31.37 - 41.11 - 265.77 = 8.24$$

COMPUTATION OF THE MEAN SUM OF SQUARES FOR EACH SOURCE OF VARIATION.

Mean Square Replication = $\frac{\text{SSReplication}}{r-1}$

r-1

$$= \frac{0.49}{3-1} = 0.25$$

$$MSA(\text{moisture}) = \frac{SSA}{a-1} = \frac{14.12}{4-1} = \frac{14.12}{3} = 4.17$$

$$MSE_{\text{Error (a)}} = \frac{SSE_{\text{Error(a)}}}{(r-1)(a-r)} = \frac{0.78}{(3-1)(4-1)} = 1.21$$

$$MSB(\text{Heating time}) = \frac{SSB}{b-1} = \frac{19.03}{2} = 9.52$$

$$MSAB = \frac{SSAB}{(a-1)(b-1)} = \frac{156.40}{(4-1)(3-1)} = \frac{156.40}{(3)(2)} = 26.07$$

$$MSE_{\text{Error (b)}} = \frac{SSE_{\text{Error(b)}}}{a(r-1)(b-1)} = \frac{3.22}{(4)(3-1)(3-1)} = 0.20$$

$$MSC = \frac{SSC}{C-1} = \frac{8.38}{3-1} = \frac{8.38}{2} = 4.19$$

$$MSAC = \frac{SSAC}{(a-1)(c-1)} = \frac{31.37}{(4-1)(3-1)} = \frac{31.37}{(3)(2)} = 5.23$$

$$MSBC = \frac{SSBC}{(b-1)(c-1)} = \frac{41.11}{(3-1)(3-1)} = \frac{41.11}{(2)(2)} = 10.28$$

$$MSABC = \frac{SSABC}{(a-1)(b-1)(c-1)} = \frac{265.77}{(4-1)(3-1)(3-1)} = \frac{265.77}{(3)(2)(2)} = 22.15$$

$$\text{MSError}(C) = \frac{\text{SSError}(c)}{ab(r-1)(c-1)} = \frac{8.24}{(4)(3)(3-1)(3-1)} = \frac{8.24}{(4)(3)(2)(2)} = 0.17$$

COMPUTATION OF THE F-VALUE FOR EACH EFFECT

$$F(A) = \frac{\text{MSA}}{\text{MSError}(a)} = \frac{4.17}{0.13} = 36.23$$

$$F(B) = \frac{\text{MSB}}{\text{MSError}(b)} = \frac{9.52}{0.21} = 47.60$$

$$F(A \times B) = \frac{\text{MSAB}}{\text{MSError}(b)} = \frac{26.07}{0.20} = 130.35$$

$$F(C) = \frac{\text{MSC}}{\text{MSError}(C)} = \frac{4.19}{0.17} = 24.65$$

$$F(AC) = \frac{\text{MSAC}}{\text{MSError}(C)} = \frac{5.23}{0.17} = 30.76$$

$$F(BC) = \frac{\text{MSBC}}{\text{MSError}(C)} = \frac{10.28}{0.17} = 60.47$$

$$F(ABC) = \frac{\text{MSABC}}{\text{MSError}(C)} = \frac{22.15}{0.17} = 130.29$$

Calculation of moisture

Weight of Petri-dish (W_1)	Weight of sample	Weight of Petri-dish + sample before (W_2)	Weight of Petri-dish + sample after (W_3)	Moisture content	Percentage moisture content
44.86g	40g	88.86g	85.84g	0.075	7.5

$$\frac{W_2 - W_3}{\text{Weight of sample}} \times 100$$

Weight of sample

$$\frac{88.86 - 85.84}{40} \times 100$$

40

$$= 0.075 \times 100$$

$$= 7.5$$

Calculation of specific gravity

Weight of empty bottle (w_1) = 7.004g

Weight of bottle + oil (w_2) = 15.49g

Weight of bottle + equal volume of H₂O (w_3) = 18.131g

Weight of oil = $w_2 - w_1 = (15.49 - 7.004)g = 8.486g$

Weight of water = $w_3 - w_1 = (18.131 - 7.004)g = 11.127g$

$$\text{Specific gravity} = \frac{W_2 - W_1}{W_3 - W_1}$$

$$\frac{8.486}{11.127} = 0.762$$

11.127

Calculation of density

Density of the oil = specific $\times 1g/cm^3$

$$0.762 \times 1 = 0.762g/cm^3$$

Calculation of iodine value.

Sample	Mass of sample used (g)	1 st titer used (cm ³)	2 nd titer used (cm ³)	Gram of iodine per 100g of sample
Roselle seed oil	2g	5.80	5.80	75.9g
Blank	--	125.40	125.50	

Average volume of acid used for blank.

$$\frac{125.40 + 125.50}{2} = 125.45$$

Average volume of acid used for sample

$$\frac{5.80 + 5.80}{2} = 5.80$$

$$\text{Iodine value} = \frac{12.69 \times N \times (v_1 - v_2)}{W}$$

$$= \frac{12.69 \times 0.1 (125.45 - 5.80)}{2} = 151.798 / 2$$

$$= 75.90 \text{g of iodine / 100g of sample}$$

Calculation of Saponification value.

Samples	Mass of sample used (g)	1 st Titre used (cm ³)	2 nd Titre used (cm ³)	Saponification value MgKOH/g
Roselle oil	2	2.60	2.60	178.12
Blank	----	15.30	15.10	

$$\text{Saponification value} = \frac{(V_2 - V_1) \times N \times 56.1}{W}$$

$$\text{S.V} = \frac{56.1 \times N \times (15.2 - 2.60)}{2} = 178.12.$$

Average volume of acid use for blank

$$= \frac{15.30 + 15.10}{2}$$

$$= 15.2\text{g}$$

Average volume of acid used for sample of oil

$$= \frac{2.60 + 2.60}{2}$$

$$= 2.60\text{g}$$

Where V_2 = Average volume of acid use for blank

V_1 = Average volume of acid used for sample of oil

Calculation of acid value

Quality of oil (g)	Volume of 0.1m KOH solution used	Acid value	Average acid value (g/ml)
0.85	0.7	4.41	4.614
0.85	0.73	4.818	

$$\text{Acid value} = \frac{V \times N \times 56.1}{W}$$

For $V=0.70$, $N=0.1$, $w=0.85$

$$\text{Acid value} = \frac{0.7 \times 0.1 \times 56.1}{0.85}$$

$$= \frac{3.927}{0.85} = 4.41$$

For $V_2 0.73$, $N=0.1$ $W=0.9$

$$\text{Acid value} = \frac{0.73 \times 0.1 \times 56.1}{0.85} = 4.818$$

$$\text{Acid value} = 4.818$$

$$\text{Average acid value} = \frac{4.41 + 4.818}{2} = 4.614$$