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Soap Production From Cottonseed Oil by- product (Soapstock)

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APPROVAL

This project research work soap production from cottonseed oil by – product (soapstock)has been read and approved as meeting condition for partial fulfillment of the requirement for an award of Bachelor Of Engineering (B.Eng) degree in Chemical Engineering of the Federal University of Technology Minna,Niger State, Nigeria.

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CERTIFICATION

I certify that this research project work soap production from cottonseed oil by-product(soapstock) has been actually carried out by Abdulraheem Abdulhakeem under my supervision.

_____   _____   _____
Project supervisor   Signature   Date

DECLARATION

I, Abdulraheem Abdulhakeem, 97/5884EH. Do declare that this project work soap production from cottonseed oil by-product (soapstock) is original work of mine and is never copied.

However, I do receive useful informations from published and unpublished sources on the matter and due acknowledgement or reference is given to them.

_____	_____	_____
Student	Signature	Date

DEDICATION

This project is dedicated to my lovely Mother, Mrs. Madinatu Abdulraheem and him himself my father Mr. Abdulraheem Bamidele Ahmadu. "May Almighty ALLAH bestow on them his mercy in this world and the hereafter. I also dedicated this project to Prince Oye Badmus, Prince Tajudeen Badmus and their family. May Almighty ALLAH show them his mercy both in the world and hear after. Amin.

ABSTRACT

The project is aimed at producing soap with cotton-seed oil by-product (soapstock) in order to reduce the cost of raw materials by blending with palm kernel oil.

Properties of soapstock from cotton-seed oil refining such as, saponification value, percentage moisture content, percentage Free Fatty Acid and Acid value were found to be190.4, 35.8%, 36.2%and 71.34 respectively.

Soaps were produced from soapstock and palm kernel oil by varying the compositions of soapstock and palm kernel oil in the ratios 1:3, 1:1and 1:0 percentage by weight in each soap.

Properties of the soap produced such as percentage Free Alkali, PH, Percentage moisture content, volume of lather and lather retention time were determined.

The soap produce, containing 25% by weight soapstock has the best quality considering among others the volume of soap lather 7.5ml and lather retention time of 10 seconds.

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CHAPTER ONE

1.0. INTRODUCTION

The definition of soap is generally restricted to the combination of fatty acids and alkalis obtained by reacting various animal and vegetable fats and oil with caustic soda or potash, the former giving hard soap and the letter soft soap. The process on which these take place is known as saponification.

The raw material for the making of soap cover a wide range of substance which may be classified under fat and oils, alkalis, water, salt etc. however fats and oils constitutes approximately 90% of the soap maker's raw materials (Peter, 1986).

Fats and oils are esters of fatty acid and glycerol. The distinction between fats and oils is purely an arbitrary one, based on the physical state at ordinary temperature. Fats are solid or semisolid at room temperature while, Oils are liquids under same conditions.

Fats and Oils are divided into three classes, namely fixed oils, minerals oils and essential oils. However, fixed oils from the main raw materials for soapmaking as they decompose into fatty acids and glycerol when strongly heated, and are easily saponified by alkali. These fixed oils includes coconut oil, palm kernel oil, palm oil, animal tallow, cottonseed oil, groundnut oil, rice husk oil, e.t.c (Peter,1986).

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Soapstock results as a by-product from the alkaline refining of cottonseed oil to remove free fatty acids, phosphotides and other mucilaginous materials. As a result soapstock contains high degree of free fatty acid which happens to the important substance or compound in soap production. It requires less alkali for complete saponification, and is a cheaper raw material compared with other available ones. These facts about soapstock makes it a suitable material for soapmaking.

The diverse areas of use and quantity of soap requirement, continue to increase and this calls for corresponding increase in it's production. In another direction, the diversification of raw material will supplement the existing ones and bring down cost.

This project is aimed at using cottonseed oil soapstock to produce a cheaper soap and it will also serve as a way or means of reducing environmental pollution.

CHAPTER TWO

2.0   LITERATURE REVIEW.

2.1   SOURCE OF SOAPSTOCK

Crude fats or fatty oils are refined (purified to remove free fatty oils and phospotides and other gummy materials and to improve the colour.

Alkaline refining is generally carried out by treating the fat with strong (10-025°Be) solution of caustic soda.

The by - product of this reaction is the precipitated material known as " soapstock" or "foots". Soapstock (foots) is the mixture of soap, oil and impurities that precipitates when   natural fatty oils are refined by treatments with caustic soda or soda ash. It contains 30 to 50 % free and combined fatty acids, and is used in the manufacture of relatively low-grade soaps and also as a source of free fatty acid (Arthur, 7th edition).

2.2   COMPOSITION OF SOAPSTOCK

Soapstock as earlier mentioned is a mixture of soap, oil and impurities. Most of all of the impurities are non-glycerides component of the fats and oils. In the case of most fats and oils, the greater part of the gylcerides portion can be removed or is removed with alkali in the course of ordinary refining. The amount of removable non-glyceride substances other then fatty acids varies considerably. It is highest in certain seed oils, particularly in cottonseed, corn and soybean oils, where it may constitute

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as much as 2 to 3% of the crude oil. Other seed oils for example peanut and coconut oil, contain comparatively little of such materials , as do the common fruit pulp oils (olive and palm oil). Animal fats such as lard and edible tallow, which are derive from more or less pure fatty tissues, one very low in impurities of this class. This may not be the case with fish oils low grade tallow or other animal, or other animal fats rendered from entire carcass.

The material largely removed (impurities) in refining are broadly described as consisting of phosphotides carbohydrates and carbohydrate derivatives, protein, fragments as various resinous and mucilaginous materials of certain identify, in addition alkali refining removes any fat free fatty acids resulting from partial hydrolysis of the oil and effects a partial removed of sterols, carotenoid pigments, tocopherols heavy metals etc. Neutral oils loss during refining is also an impurity in the soapstock (Bailey,1951).

2.3. PRODUCTION OR MANUFACTURE OF SOAPSTOCK

Since soapstock is a by - product of the refining process, it quality well depend on the type of refining operation in practice. There are four main types of refining operations these are:

(a)   Batch refining by dry method.

(b)   Batch refining by wet method.

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(c)   Continuous caustic refining.

(d) Clayton soda Ash refining process and these are all described in detail in the following paragraphs.

2.3.1 BATCH REFINING BY DRY METHODS

The stage of this refining process is carried out with the oil at atmospheric temperature, or in the case of a fat of a temperature only high enough to keep the material molten and liquid. Higher temperatures are avoided partly because they tends to increase the amount of neutral oil saponified, but more because lighter refined oil colour are obtained at relatively low temperatures. The equipment required for batch refining is simple, consisting of an open tank kettle, equipped with an agitator, steam coils for heating and a conical bottom flask.

If the oil contains occluded air after it is pumped to the refining kettle it must be settle long enough for the air to rise to the surface and escape as otherwise the foots will entrain sufficient air to float partially thus will not settle properly to the bottom of the kettle.

After the charge of the oil is at the proper temperature, and free from air, the agitator is started at high speed, and the proper amount of lye is rapidly run in. The lye, is usually distributed fairly evenly over the surface of the oil, although an elaborate distribution or spraying is not necessary. Agitation is then continued until the alkali, and oil are thoroughly emulsified.

With some oils the best result is obtained if the mixing period is relatively short for example 10 to 15 minutes. Other oil of the slow breaking type may be mixed as long as 30 to 45 minutes. At the end of the mixing period the agitators is reduced to a low speed, sufficient only to keep the content of the kettle stirred, and heat is applied to bring the temperature of the charge up to 134- 145°F as rapidly as s possible. Under the influence of the heat. The emulsions breaks and the temperatures breaks and the soapstock separate from the clear oil in the form of small flocculent particles which tends to coalesce as stirring is continued. After the desired degree of break is obtained, agitation is stopped, heat is turned off the kettle and the soapstock or foot now produced is allowed to settle to the bottom of the kettle by gravity. After the setting of the soapstock, the oil is then withdrawn and the soapstock is pushed to a soapstock receiver's tank (Bailey 1951).

2.3.2 BATCH REFINING BY WET METHOD

In European refineries where most of the oil refined are high in free fatty acid and produce soft soapstock, the wet refining method is commonly practiced. In general its involve heating the oil charge to a relatively high temperature, example 150F mixing in the lye and washing down the precipitated soapstock formed with a spray of hot water directed onto the surface of the oil. In some cases, salt sodium carbonate or other electrolyte

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is added to assisted in breaking the emulsion of soapstock and oil and to aid graining out and settling the soapstock formed (Bailey, 1951).

2.3.3 COTINUOUS CAUSTIC REFINING

In this process, the crude Oil from a feed tank is pumped continuously to a motor driver mixer, where it is treated with a metered amount of caustic soda solution: a ration flow controller is used to maintain a constant flow of caustics in relation from the mixer passes though a low pressure stream heater, where a beak is obtained by raising the temperature to 140-160° F: immediately thereafter the refined oil and soapstock are separated in a primary battery of high speed centrifuges. From the centrifuge the soapstock formed drops by gravity into an elongated receiving tank and the oil passes thought a small tank where any foot accidentally entering the oil dream can be skimmed off , and hence into a wash tank . The entire time lapsing between introduction of lye and separation of the soapstock is only about three minutes (Bailey,1951).

2.3.4 CLAYTON SODA ASH PROCESS

For this process, the mixing of the oil and the soda ash is carried out in a closed mechanically agitated vessel, with a steam jacket. During this operation the temperature is raised to 210-212°F. From the mixer the

mixture is discharged to a dehydrator vessel maintained under about 28 inches of vacuum by a steam ejector with a surface condenser, where carbon dioxide is removed and the moisture content is reduced 0.5% or less. To enable the dried soapstock to flow from the centrifuges in the subsequent stage of separation it is dehydrated with about 3 - 7% of 20° Be soda ash (by volume, on the basis of the oil). The amount of dehydrating solution required depend upon the characteristics of the oil: it is kept of a minimum consistent with a satisfactory separation, to produce a saopstock as high as possible in fatty acids. Mixing in a second stream jacketed mixer or series of mixer and during the operation the temperature, which falls to about $160^{O}F$ in the dehydrator is again raised to $205^{O}F$.

The mixture of oil and rehydrateted soapstock is separated in dics-bowl centrifuges, with the receiving tank, and the neutralized oil passing on the storage tank. To assist in the discharge of the soapstock from the centrifuges, about 1% by volume of 12° Be. (8.57%) soda ash is introduced continuously into the centrifuge bowls.

As the refining processes changes from batch through continuous caustic to continuous soda ash, the percentage free fatty acid (%FFA) decreases which might lead to low quality soapstock.

2.4 UTILIZATION OF SOAPSTOCK

Apart from the utilization of soapstock in soap manufacture, it is also used for other different purposes.

Cotton seed cake enriched with granulated or ungranulated soapstock was fed to young pigs where 5% lower daily weight gain, 5% higher energy and 3% higher protein consumption per kg of weight gain was recorded for feed (Chemical Abstract, Vol 75).

After treating soapstock using sulphuric acid (Acidulation of Soapstock) and drying to about 4% moisture. It was added to a broiler ration. The feed efficiency and the gain of chicken equal that obtained with a common feed fat added at the same level. This is to say that soapstock can perform the same function in broiler feed ration to feed chickens (McGraw Hill, 5th Edition).

Soapstock also serves as a source of free fatty acid for distillers (Arthur , 7th Edition).

2.5.0 CHARACTERISTICS OF SOAPSTOCK WITH RESPECT TO SOAP MANUFACTURING.

The characteristics of soapstock which determines its use, in soap manufacture, are Acid Value, Saponification Value and Iodine Value of the soapstock. These are discussed below.

2.5.1 ACID VALUE

The acidity of fat is expressed in terms of its acid value which is define as the number of milligram of potassium hydroxide, required to neutralize the free fatty acid in one gram of fat. Gradual decomposition of the glycerol and free fatty acid takes place when a fat is exposed to the action of light and air, with the result that the acid value steadily rises. This value may therefore be a test for the freshness of the soapstock.

2.5.2 IODINE VALUE

This is the number of grams of Iodine or equivalent halogen absorbed under standard conditions by 100g of fat. It is a measure of the proportion of unsaturated acids present. The quantity absorbed is a measure of the number of double bonds.

2.5.3 SAPONIFICATION VALUE

This is he number of milligram of potassium hydroxide required to neutralize the fatty acid resulting from the complete hydrolysis of one gram of the sample, soap is formed during the process. This value gives an estimation of the non fatty impurities present in sample (Soapstock). This value also gives the actual amount of alkali required by a given weight of fat for its conversion to soap. The saponification values of some oils such as palm oil, palm kernel oil, groundnut oil, coconut oil, etc. are 200-205, 248, 186-194, 251-264 respectively (Peter, 1986).

2.5.4 PERCENTAGE FREE FATTY ACID.

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2.5.4 PERCENTAGE FREE FATTY ACID.

Free fatty acid is one of the objectionable impurities which has to be removed from the oil during refining, which together with other impurities can render the oil dark coloured, or cause it to foam or smoke. But this happen to be the important substance or compound in soap production. The higher the percentage free fatty acid in an oil or soapstock, the better the soap to be produced.

2.5.5 PERCENTAGE MOISTURE AND VOLATILE MATTER.

Moisture and volatile matter is the amount of water and other evaporable matters that might be present in the soapstock. This is also important in soap production because the higher this value, the lower the quality of soap produced which is brought about by fermentation or decay of the soapstock, this is particular evident if this value is well above 50%.

2.6 THE PROCESSES OF SOAPMAKING

Various attempts have been made to produce soap by first decomposing the fat or oil into fatty acids and glycerin and then converting the acids into soap by treatment with sodium or potassium carbonate. However, three convectional methods of soapmaking are generally used in both large- and small scale soap production units. These methods include

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semi boiling, full-boiling and cold processes and will be discussed in the following passages.

2.6.1 SEMI-BOILING PROCESS.

The process although not suitable for the production of toilet soaps, can be used to produce laundry, all types of soft and liquid soaps. The process does not permit the removal of waste bye which contains the glycerin produced in the soapmaking process and hence the glycerin which tends to decrease the hardening properties of the soap but improves the cosmetic property. However, the method has some advantages over the other two since large quantities of good soap can be produced within a short time. The use of the method also allows a high percentage of fillers to be incorporated in soaps, thus increasing the soap bulk.

2.6.1.1 PROCESSING METHOD

The soft and hard oils or their blends are very suitable for this process which involves melting the oil or fat and running a weak 9-10% caustic soda solution into the oil, and boiling the mixture. In all 14-15% of the weight of the oil is the quantity of caustic soda required for the saponification of the oil. This weight of caustic soda is dissolved in ten times its weight of water to obtain a 9% solution. When the caustic solution

is run into the oil, soponification a starts when an emulsion is formed as the soap is stirred. More caustic solution is then run in to prevent the mass thickening. After sufficient solution is added but bit to complete the saponification, boiling of the mass continuous until the soap is clear.

After the completion of the boiling process, the time is withdrawn, and the soap allowed to cool in the boiling pan with occasional stirring. At this point, perfume and colour can be stirred into the soap moulding boxes. It is then allowed to stay for 24-36 hours to harden, after which the moulds are emptied and the solid soap block cut into the required sizes and stamped.

2.6.2 FULL-BOILING PROCESS

The process popularly used by large and some small soap Industries is the most important commercial method of soapmaking. It involves the treatment of fats or oil with an appropriate amount of alkali and removal of waste lye from the soap at the end of the process. The process produces hard and firm soaps, thought it takes longer time than the semi-boiling process and is mostly used for the production of laundry soap and soap basis for toilet soaps.

The process consist of four stages, namely saponification of the oil with alkali, graining out of the soap, boiling on strength (or clear boiling) and fitting (Peter ,1986).

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2.6.3 COLD PROCESS

This process involves the treatment of fat or oil with a definite amount of alkali and no separation of waste lye.

Although it is possible with great case to produce neutral soap by this process the soap is liable to contain free alkali and unsaponified fat. The process is based on the fact that the glycerols of certain fatly acids oils (e.g nut oils like coconut and palm kernel oils) readily combine with strong caustic soda solutions at low temperatures, and generate sufficient heat to complete the saponifiction reaction. (Peter, 1986).

2.6.3.1 PROCESSING METHOD

The process involves stirring into the milled fat or oil in a tank, half its weight of caustic soda solution of 40° Be of (relative density 1.37) at a temperature of 24° C for coconut and 38o c to 49° c for the blends. The running of the caustic solution into the oil must be done slowly and continuously. When the solution is being run into the oil the mixture must be stirred in one direction. When all the caustic soda has been run into the oil and the mixture stirred for 30 - 45 minutes, chemical reaction takes place with the generation of heat, finally resulting in the saponification of

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the oil. The contents of the tank looks thin at first, but after some few hours its becomes a solid mass. The edges of the soap becomes more transparent as the process advances, and when the transparently has extended to the whole mass, the soap is ready after perfuming to be poured into moulding boxes for hardening, cutting and stamping.

A little caustic potash solution used to blend the caustic soda solution greatly improves the appearance of the soap, making it smoother and milder. (Peter, 1986).

2.7.0 BLEACHING OF OILS AND FATS

Three bleaching methods are generally employed in the bleaching of oils and fats, namely earth bleaching, air bleaching and chemical bleaching.

2.7.1 EARTH BLEACHING

In this process, the clarified oil to be bleached is heated to 90 - 100°C and 4% of the fuller's Earth and a small quantity of activated charcoal are added to the oil and slowly agitated for 15 - 20 minutes to keep the bleach in suspension. The oil and bleach are then filtered in a

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filter press, or the oils is allowed to cool for a night and decanted, when the bleaching agents settles down to the bottom of the tank.

2.7.2 AIR BLEACHING

This process is widely used in the rural soap making for the bleaching of palm oil. In this process, clarified palm oil is heated to 200-250°C in an open tank for 4-6 hours depending on the quantity of the oil treated. In the course of the heating, the oil is oxidized and the red colour of the oil soon vanishes. In some cases where the bleaching is done in steam-jacketed tank with an open (perforated) coil, air is blown continuously through the oil by means of the coil when the oil is heated to 100°C.

2.7.3. CHEMICAL BLEACHING:

This process is generally used for the bleaching of palm oil and other deep colour oils like cottonseed oil and mustard oil. The method, which is referred to as the bichromate methods, involves dissolving potassium bichromate in water and adding the solution obtained to the clarified clean oil heated to 52°C. Dilate sulphuric acid is then run into the oil and stirred well. The bright red colour of the oil gradually changes into a green colour and after some minutes of agitation, it is allowed to cool and the green chrome liquor at the bottom of the tank on which the bleached oil floats is

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drained off. The oil is then washed (without further heating) with hot water containing some common salt to remove any traces of chrome liquor left. For every tonne of oil 10 - 12kg of potassium bichromate and 20 - 27kg of diluted sulphuric acid is used. Care must be taken so that the bleaching temperature of 50oC of the oil on saponification will yield a soap of reddish brown colour.

2.8.0 SAPONIFICATION (CHEMISTRY OF SOAP)

Saponification is the process of converting chemicals into soap. It involves the alkaline hydrolysis of fat or oil. In other words, it can be said to be the neutralization of fatty acid.

The reaction of alkali metal hydroxide with glycerides (esters of fatty acid) yields common soap with glycerol as the by-product

The basic chemical reaction is as shown below:

$$CH_2\ COOR\ +\ 3NaOH \longrightarrow 3(RCOONa)\ +\ CH_2OH \text{----------} (1)$$
$$|$$
$$CH\ COOR \qquad\qquad\qquad\qquad CHOH$$
$$|$$
$$CH_2\ COOR \qquad\qquad\qquad\qquad CH_2OH$$
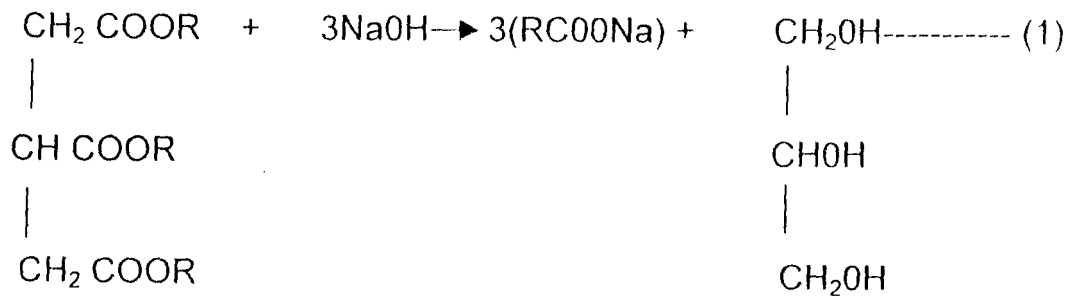
(Triglyceride)   Sodium

  oil/fat   hydroxide   Soap   Glycerol

The Intermediate process is to split, or hydrolyse, the fat and then, after separation from the valuable glycerol, to neutralize the fatty acid with a caustic soda solution.

$$(RCOO)C_3H_5\ +\ 3H_2O \longrightarrow 3RCOOH\ +\ C_3H_5(OH)_3 \qquad \{2\}$$
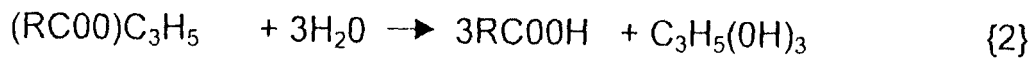
Triglyceride   Fatty
               Acid   Glycerol

$$RCOOH\ +\ NaOH \longrightarrow RCOONa\ +\ H_2O \qquad \{3\}$$

Fatty   Caustic   Soap
Acid   Soda

CHAPTER THREE

3.0   EXPERIEMENT PROCUDURE

3.1   MATERIALS

Glassware and laboratory equipments

i.   Beaker

ii.   Funnel

iii.   Conical flasks

iv.   Electric oven

v.   Filter paper

vi.   Desiccators

vii.   Separating funnel

viii.   Burette

ix.   Pipette

x.   Glass rod

xi.   Spatula

xii.   Measuring cylinder

xiii.   Volumetric flask

xiv.   Weighing balance

xv.   Electric heating plate

xvi.   Stop watch

xvii.   Resort stand

xviii. Reflux condenser

xix.   Electric heating bath.

CHEMICAL

i.   Soapstock (sample)

ii.   Palm kernel oil

iii.   Sodium hydroxide

iv.   Potassium hydroxide

v.   Ethanol

vi.   Wy's solution

vii.   Carbon tetrachloride

viii.   Potassium iodide

ix.   Sodium thiosulphate

x.   Tap and distilled water

xi.   Phonolphalene indicator

xii.   Hydrochloric acid.

xiii.   Sulphuric acid

xiv.   Colourrant

xv.   Perfume.

3.2   METHODOLOGY

3.2.1   CHARATERISITION OF SOAPSTOCK

Characterization of soapstock with respect to soap manufacture involve the determination of the soapstock properties such as Acid value, Saponification value, Iodine value and Percentage Moisture and Volatile matter. Below are the detailed procedures used in determining these properties.

3.2.2   SAPONIFICATION VALUE

A weighed sample of 2 gram soapstock was taken into a 25ml comical flask that a reflux condenser can be connected to it and 25ml alcoholic potassium hydroxide prepared as follows was pipette into the flask.

40 grams of potassium hydroxide pellets was dissolved in 20ml of water, mixing the solution with 1 litter of alcohol (95 percent) allowing the mixture to stand overnight and pouring or filtering off the clear liquid.

Another 25ml of the solution was into a second flask of similar size in the same manner. Reflux condenser were connected to the flask and were then heated in a boiling bath for 30 minutes with frequent shaking. The flask were there after removed from the bath and 1ml of phenolphalene solution was added into each titration while the content were still hot were carried out with 0.5N Hcl until a faint pink colour appears.

Saponification value was calculated as follows (Samson, 1995)

$$Srs = \frac{(A - B) \times 28.05}{W}$$

Where

Srs – saponification value of raw soapstock

W – weight of saopstock sample taken (grams)

A – amount of acid use in the blank test (ml).

B – amount of acid used in the test (ml).

3.2.3 ACID VALUE

A weighed sample of 2 gram saopstock was taken into a 250ml conical flask. 50ml of 96% ethanol was brought to boil, 1ml of phenolphalene solution was added and titrated to a paint pink colour while still hot with 0.1N sodium hydroxide solution. The neutralized alcohol was then added into the 25ml flask containing the soapstock stirred and then brought to boil. The mixture was titrated while hot with 0.1N sodium

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hydroxide solution until a paint pink colour persist for at least ten second.

The acid value was calculated as follows:

$$Ars = \frac{N \times 5.61}{W}$$

Where

Ars – acid value for raw soapstock

N – number of ml of 0.1 N NaoH used

W – weight of soapstock taken in grams

The percentage free fatty acid (%FFA)

$$\%FFA = frs = \frac{N \times M}{(W \times 100)}$$

Where

Frs. – percentage free fatty acid for raw soap stock

N – is as above

W – is as above

M – molecular weight of predominating fatty acid which id major fatty acid of the oil of which is major fatty acid of the oil of which the soapstock is made from.

3.2.4 PERCENTAGE MOISTURE AND VALATILE MATTER

A sample of 5.57 grams soapstock was taken into a 50ml beaker and heated in an electric oven for three hour of $100^{\circ}C$ followed by cooling in a

desiccators and weighing. This sample was further heated of same temperature for 1 hour followed by cooling and weighing. This procedure was repeated until constant weight was obtained.

The percentage moisture and volatile matter

= $\dfrac{\text{Difference in weight of sample before and after drying}}{\text{Weight of sample taken in grams.}}$

3.2.5 DETERMINATION OF MISTURE CONTENT

A 3g sample of the soapstock was weighed into a beaker. These was heated in an oven set of 100°C for three to four hours. This was followed by cooling in a desiccators and then weighing. The sample was further heated at same temperature for another hour, cooled and weighed. This procedure was repeated until a constant weight was obtained. The percentage moisture content was then calculated as :

% Moisture Content (MC) = $\dfrac{\text{difference in weight of sample before and after drying}}{\text{weight of sample taken}}$

3.2.6 SOAP PRODUCTION

The soapstock used in this work was derived from the continuous refining of cotton – seed oil. It was collected from Sunseed Nigeria Limited, Zaria Kaduna state. It was used in the raw form and in combined form with

palm kernel oil to prepare soap in different percentage as 100, 50, and 25% by weight of soapstock that is in ratios 1:0,1:1 and 1:3. The soap produced were subjected to laboratory test in order to determine their characteristic. The detailed procedure followed in making of the soaps and laboratory analysis of the soap produced, are discussed in this section.

3.2.7 SOAP PRODUCTION PROCEDURE

The procedure for the production of the soap having 25% and 75% content of soapstock and palm kernel oil respectively, is explained here. The same procedure was followed for the production of soap with other composition. The full- boiling process for soapmaking was used and is as summarized below.

25g of the raw soapstock was accurately weighed into a 500ml beaker, and 75 g palm kernel oil was accurately weighed and poured into the beaker containing the soapstock and stirred. A weak 9-10% caustic soda solution was prepared and poured into the beaker containing the blended soapstock and palm kernel oil. The mixture was then boiled to start the saponification. The beginning of the saponification was identified by the formation of an emulsion. As the saponification started caustic soda of higher strength (18°Be solution of relative density 1.4) was frequently added in small quantities with continued boiling. The end of the saponification was crudely determined by the ' ribbon and 'taste' tests that

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would be discuss later. When saponification was completed, the soap becomes firm and dry with a permanent faint caustic taste on the tongue when cooled. The soap, which now consists of imperfect soap together with water in which is dissolved glycerin and any slight excess of caustic soda, was then grained out as follows.

The objective of this was to separate the waste lye (which was a mixture of glycerin produced during the soap boiling process and excess caustic solution) from the soap. Brine (salt) of relative density 4.2 was added to the boiling soap and boiling was continued. The salt used was 8-10% by weight of the saopstock and palm kernel used. As the salt was added and the soap allowed to boil and stirred, the soap was gradually thrown out of the solution, and loses its transparent and homogenous appearance, becoming opaque and grainy. The graining exercise was completed when the soap is practically free from foam and floats as neat soap on the lye. The boiling was then stopped and the excess lye was allowed to settle for overnight.

The boiling pan was covered and allowed slow setting of the lye over a longer period of time to give the soap mass enough time to separate into four layers namely a small bottom layer of brine with impurities, and excess lye, a second layer of clean transparent soap (neat soap), and a top layer of a thin crust of soap. The bottom layer of brine was drained out

while the second and top layer was removed and converted into soap by boiling with water.

The third layer of next grainy soap was boiled with a small quantity of water to bring it to a smooth homogeneous form and cooled after draining into soap moulds.

3.3 ANALYSIS OF SOAP PRODUCED

The physical and chemical properties of the soaps produced were analysed. The properties analysed for and the procedure employed are given below.

3.3.1 MEASUREMENT OF SOAP LATHER.

0.13g of the soap was taken and dissolve in 100ml of distilled water. 10ml of this solution was placed in a 50ml graduated stoppered measuring cylinder and 10 ml of 1N sodium hydroxide solution was added. The mixture was then tilted twenty times in 10 second and then allowed to stand. The volume of the lather and that of the solution was taken and recorded. The suspension period of the lather formed was also recorded.

3.3.2 DETERMINATION OF FREE ALKALI CONTENT

5g of the soap sample was taken into a 600ml beaker. 300ml of hot ethanol was added to the sample and heated on a steam bath for about one hour with frequent stirring. It was then removed from the steam bath and allowed to settle. Two drop of phenolphthalein indicator was added.

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The solution turns pink and was neutralized using 0.5N sulphuric acid

0.1N sodium hydroxide solution was added until pink colour appears. The

solution was filtered through a filter paper.

The solution was then heated to boiling and 0.5ml of phenolphthalein

indicator was added. It was then titrated with 0.5N H2So4 solution until the

pink colour disappear. The percentage free alkali. Content was calculated

as :

$$\%F.A = \frac{VNF}{W}$$

Where

V = volume of acid used (ml)

N = normality of the acid used

F = 3.1 for Na_2O (if % F.A is obtained as % Na_2O)

W = weight of sample taken (grams)

3.3.3 PH MEASUREMENT:

1g of sample was weighed and transferred into a 1litre volumetric

flask. The flask was partially filled with water and agitated until the sample

was completely dissolved. The solution was diluted to the volume of the

flask with water. The flask was then tapered and the solution mixed

thoroughly and allowed to stand at room temperature for 30 minute. The

PH of the solution was then measured using s PH meter.

CHAPTER FOUR

4.0   RESULT AND DISCUSSION

4.1   RESULTS

Table 4.0: Summary of the characterization of soapstock.

ANALYSIS	VALUE
Saponification Value	190.4
Moisture Content (%)	35.8%
Free Fatty Acid(%)	36.2%
Acid Value	71.34

Table 4.10: Summary of properties of soap produced for soapstock.

Amount soapstock In Soap (%) by weight	Amount palm kernel in soap (%) by weight	(%) Free Alkali as Na20	P.H	(%0 Moisture Content	Volume of lather (ml)	Lather retention Time (sec)
25	75	0.0301	10.72	5	7.5	10
50	50	0.0370	10.42	15.33	6.7	25
100	----	0.0341	10.34	19.67	3.5	40

Table 4.20: saponification value (S.V

st number	Wt of sample taken (g)	Vol. of used for blank test A (ml)	Vol. of acid used for the test B (ml)	(A – B) (ml)	S.V	Average S.V
1	2	85.98	72.80	13.58	190.45	
2	2	85.60	72.01	13.89	190.60	190.4
3	2	85.90	72.34	13.56	190.18	

Table 4.30: percentage moisture content (% M.C)

Test number	Wt of sample taken (g)	Wt of beaker plus sample before drying (g)	Wt of beaker plus sample after drying (g)	Wt of moisture content	Percentage moisture content	Average % MC
1	3	52.080	51.020	1.060	35.3	
2	3	52.080	51.015	1.065	35.4	35.8
3	3	52.080	50.980	1.100	36.7	

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Table 4.40: Percentage Free fatty Acid (%FFA)

Test number	Wt of sample taken (g)	Vol. of 0.1n NaOH used (ml)	%FFA	Average % FFA
1	2	25.2	35.84	
2	2	25.4	36.12	36.17
3	2	25.7	36.55	

Table 4.50: Acid Value (A.V)

Test number	Wt of sample taken (g)	Vol. of 0.1N	Acid Value	Average Acid Value
1	2	25.2	70.69	
2	2	25.4	71.25	71.34
3	2	25.7	72.09	

Table 4.60: percentage free alkali as Na20(%FA)

Soap (%) by of palm kernel (approx)	Soap (%) by weight of 8.5 (approx)	Wt of soap taken (g)	Vol. of 0.5N acid used	%F.A as Na20
75	25	5	0.10	0.0301
50	50	5	0.12	0.0370
____	100	5	0.11	0.0341

Table 4.70: P.H Value

Soap % by weight (appox)	P.H
25	10.72
50	10.42
100	10.34

Table 4.80: Soap Lather And Lather Retention Time

Soap (%) by weight (approx)	Vol. of liquid (ml)	Vol. of soap lather plus liquid (ml)	Vol. of soap lather (ml)	Lather retention time(s)
25	18	25.5	7.5	10
50	18	24.7	6.7	25
100	18	21.5	3.5	40

Table 4.90 Percentage Moisture Content (%MC)

Soap (%) by weight (approx.)	Weight of soap before drying(g)	Weight of plus beaker before drying(g)	Weight of soap +beaker after drying(g)	Weight of soap after drying(g)	%MC
25	3	48.59	48.74	2.85	5
50	3	49.20	49.66	2.54	15.33
100	3	48.99	48.40	2.41	19.67

4.2 DISCUSSION OF RESULTS:

From table 4.0, the saponification value was found to be 190.4 which falls slightly below the literature range of 193-195 for cottonseed oil. This may be due to presence of saponified fraction in the soapstock and or presence of other impurities in the soapstock.

The percentage moisture content of soapstock also varies depending on the soapstock source and degree of exposure to atmosphere. The literature value for the moisture content of cottonseed oil raw soapstock is 45.6% and degummed soyabean oil raw soapstock is 50% (Bailey, 1951). The value of 35.8% moisture content obtained for this particular soapstock as seen in table 4.0 is lower. This may be due to exposure to atmosphere resulting in loss of moisture, but this shows that this soapstock might be more conveniently stored without the fear of decay in a short time. And this also shows that it is a good material for soap production.

The percentage free fatty acid (%FFA) varies depending on the refining method involved in it's production. There are various or different refining methods and their corresponding value of %FFA of soapstock produced, from these value one can know the refining method used in producing any particular soapstock. From table 4.0 36.2% of %FFA value was obtained for this particular soapstock and which falls between or within the literature range of 35 – 40% for the continuous caustic soda refining process.

The Acid value and %FFA are all dependent on the number of (ml) of the alkalis used during the experimental determinations. This follows that if the %FFA of any particular soapstock fall within the literature range, the Acid value will also fall within the range. The higher these value, the better the quality of the soap produced.

The percentage free alkali (%FA) of soap should not be greater than 0.3% as Na_2O limit. (Peter, 1986). Looking at the table 4.10 %FA of the soaps produced satisfies this specification. The soap with 50% by weight of soapstock have the highest while those with 25% and 100% by weight of soapstock have he lowest. The values of %FA tells or gives one whether a soap is corrosive or not when used.

The P^H of soap for most effective action is about 10.0 −11.0 since soap for operation and ordinary soap solution decomposes at a P^H less than 10 (Samson, 1995). From table 4.10 one might note that P^H of the soap produced fall within or between this range. Therefore this soaps may be used for those operation without the fear of decomposition of he soap solution.

The volume of lather or lather formation of any soap depends on the soap quality or rather the raw material type used in the soap production. From table 4.10 the lather volume increases with decrease in percentage weight of soapstock. The soap with 100% by weight soapstock has the lowest lather formation of 3.5ml and 40 seconds lather retention time.

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These may be due to non-mixing of the soapstock with other oils in the soap production.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Based on the project work carried out, the following points could be considered as the concluding remarks

(1) It was observed that the presence of saponified fraction and or other impurities in the soapstock lead to a decrease in the saponification value which was found to be 190.4 as compared to the range of 193-195 for pure cottonseed oil.

(2) From the result a value of 35.8% moisture content was obtained for the soapstock, which means the soapstock can conviently be Stored without the fear of decay in short time.

(3) The value of 36.2% free fatty acid obtained for this soapstock falls between the ranges of 35-40% for continuous caustic soda refining process and these shows that the soapstock was obtained/collected from continuous caustic soda refining process.

(4) Since the higher the Acid Value and percentage free fatty Acid the better the soap to be produced. The acid value and percentage free fatty Acid of the soapstock being 71.34 and 36.2% falls within the literature range and shows that the soapstock can be used to produce good quality soap.

5.2 RECOMMENDATION

The department should do something concerning it's lack of laboratory chemical and equipments, so as to ease the laboratory work of the student. That is, all necessary equipments and chemicals should be produced so that the finances involved during a student research project will be minimized.

These particular soapstock should be Acidulated that is treated with an acid, used to produce soap and compare it properties with the one of the raw soapstock. This could be given to a student in the coming set as a project.

CHAPTER SIX

6.0 APPENDICES:

6.1 CHARACTERIZATION OF SOAPSTOCK.

6.1.1 CALCULATIONS OF SAPONIFICATION VALUE (S.V)

$$S.V = \frac{(A - B) \times 28.05}{W}$$

Unknown defined as in 3.2.2

Test No 1

$$S.V = \frac{(85.88 - 72.3) \times 28.05}{2} = 190.45$$

Test No 2

$$S.V = \frac{(85.60 - 72.01) \times 28.05}{2} = 190.60$$

Test No 3

$$S.V = \frac{(85.90 - 72.34) \times 28.05}{2} = 190.18$$

$$\therefore \text{Average S.V} = \frac{190.45 + 190.60 + 190.18}{3} = 190.40$$

6.1.2 CALCULATION OF PERCENTAGE MOISTURE CONTENT (%MC)

$$\% MC = \frac{\text{weight before drying} - \text{weight after drying}}{\text{weight of sample taken in (g)}} \times 100$$

Test No 1

$$\% MC = \frac{52.08 - 51.020}{3} \times 100 = 35.30\%$$

Test No 2

$$\% \text{ MC} = \frac{52.08 - 51.015}{3} \times 100 = 35.40\%$$

Test No 3

$$\% \text{ MC} = \frac{52.08 - 50.980}{3} \times 100 = 36.70\%$$

$$\therefore \text{ Average } \% \text{ MC} = \frac{35.30 + 35.40 + 36.70}{3} = 35.80\%$$

6.1.3 CALCULATION OF PERCENTAGE FREE FATTY ACID (% FFA)

$$\% \text{FFA} = (N \times M) / (W \times 100)$$

Where unknowns are as defined in 3.2.3

Test No 1

$$\% \text{FFA} = \frac{25.2 \times 284.47}{2 \times 100} = 35.84$$

Test No 2

$$\% \text{ FFA} = \frac{25.4 \times 284.47}{2 \times 100} = 36.12$$

Test No 3

$$\% \text{ FFA} = \frac{25.7 \times 284.47}{2 \times 100} = 36.55$$

$$\therefore \text{ Average Percentage free fatty Acid} =$$

$$\frac{35.84 + 36.12 + 36.55}{3} = 36.17\%$$

6.1.4 CALCULATE OF ACID VALUE (AV)

$$AV = \frac{N \times 5.61}{W}$$

Where unknown is a s defined in 3.2.3

Test No 1

$$A.V = \frac{25.2 \times 5.61}{2} = 70.69$$

Test No 2

$$A.V = \frac{25.4 \times 5.61}{2} = 71.25$$

Test No 3

$$A.V = \frac{25.7 \times 5.61}{2} = 72.09$$

$\therefore$ Average Acid Value $= \frac{70.69 + 71.25 + 72.09}{3} = 71.34$

6.2 CHARACTERISATION OF THE SOAP PRODUCED USING SOAPSTOCK.

6.2.1 CALCULATION PERCENTAGE FREE ALKALI (%FA)

$$\%FA = \frac{VNF}{W}$$

Where

V – Volume of acid used (ml)

N – Normality of acid

F – 3.1 for Na_2O

W – Weight of soap sample in grams.

For 25% by weight soapstock in soap.

$$\% \text{ FA} = \frac{0.10 \times 0.5 \times 3.1}{5} = 0.0301\%$$

For 50% by weight soapstock in soap.

$$\%\text{FA} = \frac{0.12 \times 0.5 \times 3.1}{5} = 0.037\%$$

For 100% by weight soapstock in soap.

$$\%\text{FA} = \frac{0.11 \times 0.5 \times 3.1}{5} = 0.0341\%$$

6.2.2 CALCULATION OF PERCENTAGE MOISTURE CONTENT (M.C).

$$\%\text{MC} = \frac{\text{Weight of soap before drying - Weight Of soap after dry}}{\text{weight of soap before drying}} \times 100$$

For 25% by weight soapstock in soap:

$$\%\text{MC} = \frac{48.59 - 48.74}{3} \times 100 = 5.00\%$$

For 50% by weight soapstock in soap;

$$\%\text{MC} = \frac{49.20 - 49.66}{3} \times 100 = 15.33\%$$

For 100% by weight soapstock in soap;

$$\%\text{MC} = \frac{48.99 - 48.40}{3} \times 100 = 19.67\%$$

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