BY<br>CALEB TSADO MAMMAN

(93/3545)

DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY MINNA NIGERIA

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF A BACHELOR DEGREE IN CHEMICAL ENGINEERING.

MARCH 2000

## CERTIFICATION

I hereby certify that I supervised, read and approved this project work presented by CALEB, TSADO MAMMAN (93/3545) in partial fulfillment of the award of Bachelor degree in Chemical Engineering
Project Supervisor $\quad$ Date

## DEDICATION

This project research is dedicated to my beloved parent Rev. Stephen .S. Mamman and Mrs Eunice A. Mamman for their parental care and for ensuring that I attain this level of education

## ACKNOWLEDGEMENT

It is my pleasure to express my profound gratitude to my project supervisor Mr. MOSES OLUTOYE for suggesting this interesting topic. I am highly indebted to his relentless effort in taking his time and energy to review this work, and offer constructive criticisms, which have contributed immensely in making this, work a success.

I appreciate the help given to me by Mr. Bala, and also by Mr. Emmanuel both, technologist in chemistry department who helped me when I was carrying out my experiment in their laboratory.

My profound gratitude goes to Mrs. D. Abu and also to Mrs. G. Jiya for their moral and financial assistance to me. I owe a lot to Mr. Ayeni a technologist with global soap and detergent industry Ilorin, Kwara State, Nigeria.

My special thanks also goes to Mr. Joshua K.I. Mamman who introduce chemical engineering to me as an engineering course, and also to my brothers and sisters for their support.

I thank my fellowship members for their prayer, my prayer is that almighty God will reward You.

Finally, to almighty God the king of kings and the Lord of lords to him be glory and honour that helped me to start and complete this research work.


#### Abstract

The project research was carried out to test and compare the effects of saponin from "Habiscus Sabdarifa" and saponin from "Enterolobium Cyclocapum" on soap production using palm kernel oil. Experiments were conducted to test the effects of saponin on hardness, foamability, hydrogen ion concentration and rate of wear of soap.

The research will involve pre-treatment of palm kernel oil. It will also involve an improvement on the hardness, odour, disinfectant, colour and formability, test will also be carried out to ascertain the effectiveness of the product on properties tested and finally, a sample of improved quality soap will be produced.

Saponin from "Saddarifa" was added to soap sample $\mathrm{A}_{1}, \mathrm{~A}_{2}, \mathrm{~A}_{3}$ and $\mathrm{A}_{4}$ at various (ml) 0 ml , $5 \mathrm{ml}, 10 \mathrm{ml}$, and 15 ml respectively. The same thing was done for saponin from "Enterolobium". It was added to soap sample $B_{1}, B_{2}, B_{3}$ and $B_{4}$ at various (ml), $0 \mathrm{ml}, 5 \mathrm{ml}, 10 \mathrm{ml}$ and 15 ml respectively.

For hardness test, it was observed that saponin does not increase or lower the hardness of soap samples are properly dried, they have equal level of hardness regardless of the quantity of saponin added. Also the results obtained from the rate at which soap diminishes when being used.

Foamability test shows that saponin increases the foamability of soap and it was also discover that saponin from "Enterolobium" produced more foam than saponin from "Habiscus Sabdarifa" and corrosive tests conducted shows that saponin increase also the level of alkaline in the soap; for this reason if saponin is to be used in large quantity in soap formulation, other additives will be needed to bring down the level of alkaline.


## TABLE OF CONTENT

PAGE
TITLE PAGE
CERTIFICATION ..... i
DEDICATION ..... ii
ACKNOWLEDGEMENT ..... iii
ABSTRACT ..... iv
TABLE OF CONTENT ..... v
CHAPTER ONE
1.0 INTRODUCTION ..... 1
1.1 AIM AND OBJECTIVE OF THE PROJECT WORK ..... 2
CHAPTER TWO
2.0 REVIEW OF LITERATURE ..... 3
2.1 ORIGIN OF SOAP ..... 3
2.2.0 METHOD OF SOAP MANUFACTURE ..... 3
2.2.1 SOAP PRODUCTION PROCESS ..... 4
2.2.2 COLD PROCESS ..... 4
2.2.3 SEMI-BOILED PROCESS ..... 5
2.2.4 FULLY-BOILED PROCESS ..... 5
2.2.5 MODERN CONTINOUS PROCESS ..... 6
2.3.0 PROPERTIES OF SOAP ..... 6
2.4.0 CONSTITUENTS OF SOAP ..... 8
2.4.1 FATS ..... 8
2.4.2 CAUSTIC SODA ..... 8
2.4.3 BRINE ..... 9
2.5.0 SOAP ADDITIVES ..... 9
2.5.2 OPTICAL BRIGHTENERS ..... 9
2.5.3 FILLERS ..... 10
2.5.4 ANTI-REDEPOSIT AGENTS ..... 10
2.5.5 PERFUMES AND COLOURANT ..... 10
2.6.0 TYPES OF SOAP ..... 10
2.6.1 TOILET SOAP ..... 10
2.6.2 HOUSE HOLD SOAP ..... 11
2.6.3 SOAP POWDER ..... 11
2.6.4 SCOURING POWDER ..... 11
2.6.5 FLAKES ..... 12
2.6.6 GERMICIDAL SOAP ..... 12
CHAPTER THREE
3.0 EXPERIMENTS ..... 13
3.1 EXTRACTION OF SAPONIN FROM GLYCOCIDIC PLANT ..... 13
3.2 EXTRACTION OF PERFUME FROM LEMON GRASS ..... 13
3.3 PRODUCTION OF SAMPLES OF SOAP ..... 16
3.4.0 TESTS ..... 17
3.5.1 FOAMABILITY TEST ..... 17
3.6.0 CORROSIVE TEST ..... 20
3.7.0 HARDNESS TEST ..... 20
3.8.0 TEST FOR RATE OF WEAR ..... 21
CHAPTER FOUR
4.0 RESULTS ..... 22
4.1 COMPOSITION OF SOAP SAMPLES ..... 22
4.2.0 FOAMABILITY ..... 22
4.3.0 pH VALUES ..... 24
4.4.0 HARDNESS ..... 24
4.5.0 RATE OF WEAR ..... 25
CHAPTER FIVE
5.0 DISCUSSION OF RESULTS ..... 27
5.1.0 EXTRACTION OF SAPONIN ..... 27
5.2.0 EXTRACTION OF PERFUME FROM LEMON GRASS ..... 27
5.3.0 HARDNESS ..... 27
5.4.0 FOAMABILITY ..... 28
5.5.0 CORROSIVE ..... 28
5.6.0 RATE OF WEAR ..... 28
CHAPTER SIX
6.0 CONCLUSION ..... 29
6.1 RECOMMENDATIONS ..... 29
REFERENCES ..... 30

## CHAPTER ONE

### 1.0 INTRODUCTION:

Soap means a cleaner made with naturally occurring materials, soap also means cleaning agent, made by the chemical action on fats or fatty acids and yielding the sodium or potassium salts of these acids. Little is known about the discovery of soap, one legend says it was accidentally discovered on Rome's sapo hill about 2,500years ago.

However soap is the common term for a number of related compound used as cleaning agents and for a wide range of industrial purposes. Ordinary household soaps consist primarily of the salts of fatty acids that occur in natural occurring carboxylic acids are also used to make a variety of soaps. A soap is a detergent in that "detergent" means "cleaning agent" but usually only the synthetic materials introduced in the 1930's are called detergents

Soap is produced from a type of chemical reaction known as saponification. This basic chemical reaction was obtained in the early days by combining oils or fat remnants with alkali made from wood ashes. These early soap were of many different kinds. They might be of different colour including black. In the modern method of soap production, the wood ashes are replaced by inorganic chemicals known as caustic alkalis. The most commonly used is sodium hydroxide, NaOH . Unlike in the past when soap that was mild enough for washing faces and hands was considered a luxury there are a good number of quality soaps now in use.

The soap produced by saponification reaction only cannot make a good quality detergent. This is because it may not be good for personal and many other uses due to its corrosiveness and difficult in cleaning. Research works have made possible the discovery of additives which improved the quality of soaps.

Additives are added during soap production to enhance the detergent of cleaning action of the soaps are produced, creating better market for the manufacturers.

It is on the basis of bringing the additives to the knowledge of the local producers that this research work is embarked upon.

### 1.1 AIMS AND OBJECTIVES OF THE PROJECT WORK.

The aim of this work is to test the effect of saponin from two sources on soap production using palm kernel oil to know the most effective.

The research work will involve pretreatment of palm kernel oil. It will also involve an improvement on the ; hardness, odour, disinfectant, colour and foamability more so test will be carried out to ascertain the effectiveness of the product on properties tested and finally a sample of an improved quality soap will be produced

## CHAPTER TWO

### 2.0 REVIEW OF LITERATURE

### 2.1 ORIGIN OF SOAP

Soap has been known for at least 2,300years. According to Pliny the Elder, the Phoenicians prepared it from goats tallow and wood ashes in 600 BC and sometimes used it as an article of barter with the Gauls.

Soap was widely known in the Roman Empire, whether the Romans learned its use and manufacture from ancient Mediterranean people or from the celt, inhabitants of Britannia. is not known. On this note, we can say no one knows who discovered soap. It is likely that someone many, many years ago made the first soap from animal fats and alkali made from wood ashes. The celts who produced their soap from animal fats and plant ashes, named the product "Saipo" from which the word soap is derived. The importance of soap for washing and cleaning was apparently not recognised until the 2nd Century after Christ; the Greek physician Galen mention it as a medicament and as a means of cleansing the body. Previously soap had been used as medicine. While we can only guess about soap origin, we do know that a crude kind of soap was used in France around A.D 100.

In Europe, soap production in the middle Ages centered first at Marseilles, later at Genoa then at Venice. Though some soap manufacture developed in Germany, the substance was so little used in central Europe that a box of soap presented to the Duchess of Juelich in 1549 caused a sensation. As late as 1672 , when the German, A leo, sent lady Von Schleinitz a
parcel containing soap from Italy he accompanied it with a detailed description of how to use the mysterious product.

The first English soap makers appeared at the end of the 12th Century in Bristol. In the 13th and 14th centuries, a small community of them grew up in the neighbourhood of cheapside in London.

Even commercial soap making remained on a rather crude level until 1791 when Nicolas Leblance, a French man discovered a process for making soap ash or sodium from common salt, thus doing away with the old expensive method of obtaining alkalies from wood or ensive method of obtaining alkalies from wood or seaweed ashes. In 1823 Michel Eugene Chevreul, another French chemist, concluded 10year of research to reveal that all fats consists of glycerin combined with organic acids, called "fatty acids". All acids from salt when reacted with alkali and the alkali salt of fatty acid are called soap.

### 2.2 METHOD OF SOAP MANUFACTURES

Vegetable oils such as palmoil, groundnut olive, kernel oil and animal fats are naturally occurring asters formed from a long-chain organic acid, octadecanoic (stearic) acid, C 17 H 35 COOH (containing 18 carbon atom per molecule) and popan-1,2,3-triol(glycerol and alkanol containing three - OH group in a molecule

When palm oil( or groundnut oil) is boiled with an alkali such as sodium hydroxide solution, it breaks down releasing the organic acid and the alkanol. This process is known as saponification. The organic acid is immediately neutralized by the NaOH solution to form the sodium salt of the organic acid, which is the soap. The process can be explained in this way:
i Let the alkyl group in the octadecanoic acid i.e C 17 H 35 , be represented simply by R ; the simplified form of the organic acid then become RCOOH
ii Because propane $1,2,3,-$ triol, contain three hydroxyl(-OH) groups, one molecule requires three molecules of RCOOH to form one molecule of ester oil, i.e.

iii The reaction between the ester (oil) and NaOH solution(Saponification) gives soap and propane-1,2,3,-triol


The general equation for soap preparations is
$\mathrm{RCOOR}+\mathrm{NaOH}->\mathrm{RCOONa}+\mathrm{ROH}$
Soap alkanol
soap is therefore the sodium salt of an alkanoic acid, i.e sodium alkanoate.
Industrially, the palm kernel oil is first pretreated. At the end of the chemical reaction sodium hydroxide is always added to precipitate or "salt out" the soap, i.e free from any excess alkali. The soap is separated and purified. Dyes and perfumes are added to provide soaps.

### 2.2.1 SOAP PRODUCTION PROCESS

There are four soap production processes the use of which depends on the quality of soap to be produced. They include:-

### 2.2.2 THE COLD PROCESS

In this method only very pure raw materials may be used because, once made, there is no means of washing away impurities. All the glycerine is separated from the fatty glycerides but remains in final soap. The calculated amounts of fat charge consisting mainly of coconut, palm kernel, tallow fats or oil and others, are intimately mixed at a temperature just above their malting point $\left(32-35^{\circ} \mathrm{C}\right)$ together with concentrated caustic soda lye. It is preferable to undersaponify rather than use the correct equivalent of caustic soda required by the saponification value. This is to avoid the possibility of an excess of free caustic soda in the soap which cannot be dealt with once the soap has formed.

Once mixed and intimately stirred the fats and lye form a homogeneous mass which is quickly poured into large cooling frames(i.e open rectangular metal boxes the sides of which
can be removed ) Where saponification takes place with the evolution of considerable heat. The frames are kept at a temperature of $30^{\circ} \mathrm{C}$ and although the reaction only takes a few hours, the soap is allowed to stand in frames for several days, during which time it cools and solidifies

This sides of the frame are then removed and the soap slab is cut into bars on a wire cutting table, stamped and packed. The advantage of this method lies in the low initial cost of plant, speed of manufacture, and low heating and labour costs. Since glycerine is a valuable material which is not separated from the soap, only a relatively small amount of soap is made by this method

### 2.2.3 THE SEMI-BOILED PROCESS

In this process virtually the same procedure is adopted as for the cold process except that higher melting- point fats are used which necessitate higher reaction temperatures i.e around 700 C to 800 C . Once the caustic lye is added, the mass is stirred continuously at a temperature of notless than 700 C , where the soap is formed.

Here the fat is placed in the kettle and alkali solution is added while the mixture is stirred and heated but not boiled.

### 2.2.4 THE FULLY- BOILED PROCESS

This process differs from the cold process and the semi-boiled process in that glycerine is seperated as a by-product and recovered. Today, a considerable proportion of the total soap production is made by this method, which calls for the highest skill on the part of the operator.

In the hot process, the oil or fat is rigorously heated until it is fried. The required quantity of sodium hydroxide; NaOH solution is added while stirring. The soap thus obtained is seperated from the mixture by a process known as salting out, by adding sodium chloride, NaCl solution which causes the soap to collect in a solid layer at the surface making it possible to run off the mixture of glycerol, water and brine from the bottom of the container.

After seperation, the soap is washed, dried, coloured and perfumed, and run into tins or suitable moulds where it hardens.

### 2.2.5 THE MODERN CONTINUOUS PROCESS

The disadvantage of fully-boiled process lies in the time-consuming factor where by large pans of soap lie idle during settling, brining and washing periods, thus increasing the cost of the final soap.

The continuous process of which a good example is the mazzoni process, is designed for the automatic and complete saponification of vegetable oil and animals fats or their fatty acids carrying right through the various stages of drying, plodding, milling and blending to the final bar soap.

### 2.3.0 PROPERTIES OF SOAP

Soap are surface-active agent that is, they reduce surface tension when dissolved in water or organic solvents, and in particular they tend to form emulsion or collodal suspensions with other materials present in the solutions.

Functionally, soap has a slippery feel, form gets emulsifies oils, lower the surface tension of water, has detergent and wetting properties, forms curds in hard water, and foams when shaken up in soft water. For example, when soapy water is used to wash hands, dirt particles on the hands are wetted and loosened by the soap molecules, then emulsified or held in suspension as the water is rinsed away. According to Rideal these properties can be brought about by the presence of a hydrophilic polar group at the end of a wide variety of long chain hydrophobic molecules. The difficulty that soap present as a cleaner is that in hard water, water rich in calcium and magnesium salt- it forms insoluble salts that are left behind as residues and it does not produce a good lather, for proper cleaning action.

A well formulated soap has the following characteristics:-
i Wetting of the surface and incase of textiles, penetration of the fibre structure by wash liquor containing soap. Soap increases the spreading and wetting ability of water by reducing its surface tension, that is, the affinity its molecules have for each other in the presence of the molecules to be washed.
ii Absorption of a layer of soap at the interface between the water and the surface to be washed and between the water and soil
iii Dispersion of soil from the fibre or other material into the water facilitated by mechanical agitation and high temperature. IN the case of toilet soap, soil is dispersed in the foam formed by mechanical action of the hands.
iv Preventing the soil from being deposited again onto the surface cleansed. The soap accomplishes this by suspending the dirt in a protective colloid, sometimes, with the aid of special additives in a great many soiled surface the dirt is bound to the surface by a thick film of oil or grease. The cleansing of such surface involves the displacement of this film by the detergent solution, which is in turn washed away by rinse waters.

The oil film breaks up and separates into individual droplets under the influence of the soap solution.

Furthermore, the substance known as 'soap' may be defined as a chemical compound or mixture of chemical compounds resulting from the interaction of fatty acids or fatty glycerides with a metal radical(or organic base). The metal generally concerned in soap making are sodium, potassium, which produce water-soluble soaps, differing from those made from divalent metals such as calcium, magnesium, iron or aluminium which are water solution. Fatty acid salts of alkaline earth metals or heavy metals, as a rule, are termed "metallic soap" and posses grease- like instead of detergent properties.

By further definition, soap may be understood to mean any water- soluble salt of those fatty acids, which contain EIGHT OR MORE CARBON ATOMS. Chemically soap may be represented by the formular: $\mathrm{R}_{1}-\mathrm{COOR}_{2}$ where $\mathrm{RI}=$ a straight chain hydrocarbon radical saturated or unsaturated with between eight and twenty-one carbon atoms, and $\mathrm{R}_{2}=$ a base forming radicals either inorganic or organic, of such a nature that $\mathrm{R}_{1}-\mathrm{COOR}_{2}$ is soluble in water.

### 2.4.0 CONSTITUENTS OF SOAP.

When discussing the chemical properties of soap, it is important to study the chemical properties of the major constituents of soap which make the soap itself.

### 2.4.1 FATS:-

A knowledge of functional groups and their chemical behaviour is essential to the understanding of the properties of more complex organic molecules. Chemically, fats are esters of fatty acids and glycerine. These esters are either triglycerides or mixture of triglcerides with small quantities of mono-and diglycerides aND SOME FREE FATTY ACIDS. For example fats from animal tissues and oils from plants are esters of glycerol and long chains carboxyic acids. The glycerides of stearic acid may be described as follows:-


### 2.4.2 SODIUM HYDROXIDE

Sodium hydroxide is a white deliquescent solid, which melts to a clear liquid at 591 K without decomposition, it absorbs moisture and CO 2 from air which forms crust of Na 2 CO 3 .

The alkali metal hydroxide are freely soluble in water, it is soapy to touch due to the hydrolysis of the peptide linkage of the amino acids of skin it is a strong base and forms salts with acidic and atmospheric oxides. Being a strong alkali, it displaces some weak bases e.g. NH3, molten NaOH is very corrosive and can attack many minerals like Silicates, oxides and phosphates e.t.c.

### 2.4.3 BRINE

Brine is sodium Chloride solution. The percentage concentration in solution depends on the manufacturers specification but ideally $40-50 \% \mathrm{NaCl}$ in solution is used. Brine is used for salting out during hot process of soap production

### 2.5.0 SOAP ADDITIVES

Soap additives are materials added during production of soap to improve the quality of soap. They include builders like Sodium Carbonate, Sodium Silicate and Sodium tri-polyphosphate. Also grouped as soap additives are optical brighteners like trinopal; and substance, which only increase the quantity of soap known as fillers.

### 2.5.1 THE BUILDERS

These are certain inorganic salts and alkaline materials added to soap which functions to increase the detergency. Builders which enhances the cleansing action of soap include sodium silicate( Na 2 SiO 3 ), sodium tri-poly-phosphate, e.t.c They boost the cleansing and shining power of soap.

### 2.5.2 OPTICAL BRIGHTENERS

Optical brighteners are dyestuff absorbed by textile fibres from solution but not subsequently removed in rinsing. They convert invisible ultraviolet light into visible light on the blue side of the spectrum, causing the fibres to reflect a great proportion of visible light and making it appear-brighter and white. An example is trinopal.

### 2.5.3 FILLERS

These are added to increase the quantity of soap and reduce manufacturing cost of the product. Example Koalin, Starch, fine clay and Borites.

### 2.5.4 ANTI- REDEPOSIT AGENTS

These include sodium carboxylic-methyl cellulose which suspend dirt in solution so that it cannot redeposit on the washed material.

### 2.5.5 PERFUMES AND COLOURANTS

Perfumes and colourant are added in soap to incorporate pleasant odour and appearance respectively. Perfumes include Citonella oil, lemon grass oil, bergamot and oil of bay. While colourant can be red, blue, green, yellow e.t.c

### 2.6.0 TYPES OF SOAP

Perhaps the most popular soap and soap types in general use in the average home are as follows:-

### 2.6.1 TOILET SOAP

These are made in oval, rectangular, curved or round shapes, with price usually dependent on brand name, grade of perfume employed and quality of soap.

The composition of the fat charge prior to saponification is all important and predetermines the hardness, lathering power, stability on storage and soap fill to be expected in the skin, the free alkali in the soap tablet must not exceed 0.03 percent, the free fat should not be in excess of 0.1 percent, and the salt present in the soap should not exceed 0.1 percent, since higher amounts can render the tablet brittle and subject to cracking. The iodine value of the fat charge should not be greater than 45 .

### 2.6.2 HOUSEHOLD SOAP

Household soaps, usually in the form of rectangular bars are used for floor-scrubbing or hand-bowl washing, and are now gradually being suspended by heavy-duty powder or liquid detergents.

The fatty acid content is usually between 63 and 70 percent.
Some household soap brands have stammed falling sales by redefining their usage image, linked with 'mildness' to take part of the toilet soap market.

### 2.6.3 SOAP POWDERS

In their earliest forms, these were made by first producing flakes, ribbons or soap chips and grinding. Today soap is spray-dried. In this process the molten soap is crutched (kneaded) with builders(mild alkalis) and water, to solubilize the latter and lower the viscosity of the mass. The mixture is heated and pumped at high pressure through fine jets down a high tower. To assist the drying process, hot air is blown upwards into the tower, and by suitably arranging the variables of hot air temperature, jets size original crutcher mix, moisture, and pumping pressure, the particle size, bulk density, and moisture content of the final soap powder can be controlled within fine limits.

### 2.6.4 SCOURING POWDERS

Scouring powders although not soaps in the strict sense of the word, nevertheless certain up to 10 percent of soap powder combined with abrasives such as fine silica, feldspar, tripoli, diatomaceous earth and ground pumice.

In the modern scouring powders, soap powder has been replaced by alkyl benzene sulphonates and other synthetic detergent which do not form, a scum in hard water. These are combined with builders, abrasives, perfume, and sometimes up to two percent of a powdered bleaching agent.

### 2.6.5 FLAKES

The soap is first made from a good tallow coconut oil $85: 15$ ratio blend. The molten soap mill which spreads the soap in a thin, solid film which is cut by the last roller into diamond or square shapes. Flakes, because of high sud formation at warm temperature, solubility, and excellent cleansing power, are most suitable for the washing of woolens, silks, knitted goods and fabrics made from artificial yarns. Compound with bar soap-flakes have of course, a tremendous surface area for the same weight of soap. Hence they dissolve more easily and yield latter more quickly.

### 2.6.6 GERMICIDAL SOAPS

This type of soap comprises of a group developed to combat specific skin infections or for general skin disinfection. They are generally based on a good quality toilet soap, and the earliest germicidal soap was "carbolic soap"

## CHAPTER THREE

### 3.1 EXTRACTION OF SAPONIN

### 3.0 EXPERIMENTS

This aspect of the project covers all the experimental procedure. These include extraction of saponin from hibiscus sabdarifa, extraction of saponin from enterolobium cyclocarpum, extraction of essential oil from lemon grass, production of soap samples.
3.1.1 MATERIAL:- (i) Calcyces of calyxes (sepals of hibiscus sabdarifa - a ring of leaves which protects the flower before it opens and later support the opened flower, (ii) Normal - hexane.
3.1.2 APPARATUS:- Glass soap reactor, mechanical stirrer, weighing balance, Glass funnel, filter paper and wash bottle.

METHOD:
STEP1: SOLID LIQUID EXTRACTION (LEACHING)
100 Kg of hibiscus sabdarifa was grounded to powdery form and soaked in 1000 cm 3 ( 1 dm 3 ) boiled water. The mixture was left for about 12 hours for leaching to be completed. By this time the mixture turned deep red in colour and the residue was then filtered off, leaving a clear deep red solution.

STEP2:
50 g of the solution was poured into a separating funnel and 10 g of n -hexane added and shaken a number of times. The mixture turned foaming showing that saponin had been extracted from the liquid. This was allowed to settle for about 2 to 3 minutes. During this time, the reddish solution settled below and was drained off the funnel. The foamy extraction on top which contain some $n$-hexane entangled with the saponin was washed a number of times.

Step 2 was repeated many times with the new solutions to obtain the required quantity of saponin. 13

The same procedure was repeated to obtain saponin from cyclocarpum enterolobium


FIGURE 1

### 3.2.0 EXTRACTION OF LEMON GRASS PERFUME

3.2.1 MATERIALS: Lemon grass, and n-hexane.
3.2.2 APPARATUS: Mortal and pestle, Round bottomed flask, cork, knife and container bottles.

### 3.2.3 METHOD

STEP 1 The grass was washed with water and dried under the sun. It was then cut into small pieces and weighed.

STEP 2: 50 g of the grass as placed in the mortar and pounded with a pestle. After pounding it to very tiny pieces, it was transferred into a round-bottomed flask in which 10 g of n -hexane was added and the cork was placed. The flask was shaken many times until the hexane had turned deep green. At this point, the extract was drained from the flask. The perfume was heated for the hexane to evaporate. After evaporation, the greenish liquid having the odour of lemon could be decolourised.

### 3.3.0 PRODUCTION OF SAMPLES OF SOAP

3.3.1 MATERIAL: Beakers, measuring cylinder, weighing machine, container bottles, sample containers and glass stirrer.

### 3.3.2 METHOD

A cold-batch process in which no heating was involved was used.

STEP 1: PREPARATION OF OIL AND NaOH

### 3.5.2 PROCEDURE

20 g of each sample of soap was accurately weighed and grounded and made into 1000 ml uniform solution. Then 500 ml of the soap solution was poured into another 1000 ml measuring cylinder and mechanical stirrer inserted well into the solution. The stirrer is switched on for 3 minutes. It was then removed and the foamy solution allowed to settle for 5 minutes After this time, the foam level which had reduced was measured. See table 4.3 for the results and figure 2 for sketches.

To obtain a very good soap, the percentage of caustic soda in solution has to be $40 \%$. To obtain this formation 40 g of caustic soda was dissolved in 100 ml of water and used against the oil. Palm kernel oil was used in which the specific gravity free fatty and moisture content was known

## STEP2: SAPONIFICATION

50 ml of oil was poured into a sample container and 20 ml of sodium hydroxide, $(\mathrm{NaOH})$ solution was mixed with it. The mixture was stirred vigorously for 1 minutes, after which calculated quantity of saponin which was to free it of n-hexane, stirring was continued until the pasta becomes thicker

Three more samples of the same formation were produced with different amounts of saponin (from hibiscus sabdarifa)

The same procedure was repeated for saponification, expect that in this case saponin from (enterolobium cyclocapum) was the additives used. Three more samples of the same formation were produced with different amount of saponin.

### 3.4.0 TESTS FORMABILITY TEST

### 3.5.1 MATERIALS AND APPARATUS

The reagent used is water. The apparatus are weighing machine stop-watch, 2 measuring cylinders, mortal and pestle.


## FIGURE 2

### 3.6.0 CORROSIVE TEST

This was based on calculating the percentage of unconverted caustic $\operatorname{soda}(\mathrm{NaOH})$, in the soap sample as different saponification products have different degree of reactiveness to the human skin fabrics, plastic and metal surfaces. The test determined how mild or hazardous the soap samples was.

### 3.6.1 PROCEDURE

This test was carried out by dissolving 2.0 g of a soap sample in 150 ml of hot distilled water. After dissolving properly, 3 or 4 drops of methyl orange was added to produce a yellowish colour. Then standard acid solution of 0.5 M H 2 SO 4 was titrated against the soap solution until the end-point was reached. The yellow colour at this point changed to orange, hence, the volume of acid used was read from the burette.

The formula below was used in calculating the active(free) alkalinity of the soap as

$$
\% \mathrm{NaOH}=\mathrm{T} 1 \mathrm{X} \mathrm{Mw} \mathrm{X} \mathrm{M}
$$

Wg X 20
Where $\mathrm{T} \mathbf{1}=$ Volume of acid used
$\mathrm{Mw}=$ Molecular weight of NaOH
$\mathrm{M}=$ Molarity of acid
$\mathrm{Wg}=$ Weight of soap sample dissolved
$20=$ a constant.
see table 4.4 for the results obtained using the formular above.

### 3.7.0 HARDNESS TEST

Due to different chemical compositions, different soap samples produced possess various degree of hardness. This test was to show how soap samples will be resistive to pressure

### 3.7.1 PROCEDURE

The process of determination was simple as compared with the other tests so far carried out. This was done by cutting same size of the samples and subjecting each of them to fingerpress for a definite time (5-10 seconds to be precise) The feelings to the finger was then noted.

The results obtained are in table 4.6

### 3.8.0 TEST FOR RATE OF WEAR

This is the terminology used in describing the reduction in size of solid soap after it has been used for a period of time. It is important because it determines the durability of the soap.

### 3.8.1 APARATUS AND MATERIALS

Materials needed are distilled water, sample of solid soap while the equipment required are stop-watch, knife, weighing machine, conical flask and fork.

### 3.8.2 PROCEDURE

To carry out this experiment 10 g of soap samples having equal surface area and volume was cut with a knife. These equal volume of samples were then put into different conical flasks. Equal volume of water $(150 \mathrm{ml})$ was added to each of the conical flasks containing the samples. Then the flask was shaken for 10 seconds and allowed to settle for a minute interval. This process was repeated for 10 minutes. After this time, the remaining soap samples were remowed from the flask using a fork and weighed see table 4.8 for the results obtained during the experiment.

## CHAPTER FOUR

### 4.0 RESULTS AND CALCULATIONS

### 4.1.0 COMPOSITION OF SOAP SAMPLES

Table 4.1 and 4.2 shows the composition of the soap samples prepared using saponin (hibiscus sabdarifa) and saponin (form enterolobium cyclocarpum), essential oil (from lemon grass) as additives.
4.1

| Samples of | Composition in (ML) |  |  |  |  | (ML) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| soap | Palm <br> kernel oil | $\mathrm{NaOH}$ <br> solution | $\mathrm{H}_{2} \mathrm{O}$ | (Lemon <br> Grass)essential <br> oil | Dye | (HIBISCUS <br> SADARIFA) <br> SAPONIN |
| A | 50 | 20.00 | 10.00 | 1.00 | 1.00 | - |
| B | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 5 |
| C | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 10 |
| D | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 15 |

4.2

| Samples of | Composition in (ML) |  |  |  |  | (ML) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| soap | Palm <br> kernel oil | $\begin{aligned} & \mathrm{NaOH} \\ & \text { solution } \end{aligned}$ | $\mathrm{H}_{2} \mathrm{O}$ | (Lemon <br> Grass)essential <br> oil | Dye | (ENTEROLOBIUM <br> CYCLOCARPUM) <br> SAPONIN |
| A | 50 | 20.00 | 10.00 | 1.00 | 1.00 | - |
| B | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 5 |
| C | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 10 |
| D | 50 | 20.00 | 10.00 | 1.00 | 1.00 | 15 |

### 4.2.0 RESULTS ON FOAMABILITY TEST

The table 4.3 gives the results obtained during foamability test. The net height of foam of samples are recorded for the set of samples prepared using palm kernel oil, saponin (from hibiscus) and saponin ( fom enterolobium)
4.3

| Soap samples | Net height of foam for samples <br> prepared using saponin ( from <br> hibiscus sabdarifa) | Net height of foam for samples <br> prepared using saponin ( from <br> enterrolobium cyclocarpum) |
| :---: | :---: | :---: |
| A (without <br> Saponin) | $8.08(\mathrm{~cm})$ | $8.10(\mathrm{~cm})$ |
| B (with 5 ml of <br> saponin) | $10.20(\mathrm{~cm})$ | $11.8(\mathrm{~cm})$ |
| C (With 10 ml of <br> saponin) | $12.5(\mathrm{~cm})$ | $14.6(\mathrm{~cm})$ |
| D (With 15 ml of <br> saponin) | $14.8(\mathrm{~cm})$ | $16.4(\mathrm{~cm})$ |

### 4.3.0 RESULTS ON CORROSIVE TEST

The table 4.4 gives the results obtained during corrosive test. They are given as percentage unconverted sodium hydroxide $(\% \mathrm{NaOH})$ in the samples.

| Soap <br> samples | Samples of saponin from (hibiscus) |  |  | Samples of saponin from (enterolobiun) |  |  |
| :---: | :---: | :--- | :---: | :---: | :---: | :---: |
|  | Acid volume used | Free-Alkaline (\% <br> NaOH) | Acid volume used | Free-Alkaline <br> $(\% \mathrm{NaOH})$ |  |  |
| A | 58.34 | 2.92 | 56.14 | 2.81 |  |  |
| B | 61.56 | 3.08 | 59.98 | 3.00 |  |  |
| C | 67.76 | 3.62 | 65.51 | 3.28 |  |  |
| D | 72.42 | 3.62 | 71.73 | 3.59 |  |  |

### 4.3.0 pH READING OF THE SAMPLES

pH values of the various samples as measured from a pH meter is shown on table 4.5 .

| Soap samples | Sample preapared using saponin <br> (from Hibiscus) | Sample prepared using <br> saponin (from Enterolobium) |
| :--- | :--- | :--- |
| A (without saponin) | 11.79 | 12.00 |
| B (With 5 ml of saponin) | 12.33 | 11.97 |
| C (With 10 ml of saponin) | 12.21 | 12.00 |
| D (With 15 ml of saponin) | 12.42 | 12.00 |

### 4.4.0 RESULTS ON HARDNESS TEST

The table 4.6 gives the result obtained during the test for hardness of the soap samples produced

| Samples of Soap | Quantity of saponin <br> added | HARDNESS |  |
| :--- | :---: | :--- | :--- |
| A | - | After 2 days | After 7 days |
| B | 5 ml | Not so hard | Strong |
| C | 10 ml | Not so hard | Strong |
| D | 15 ml | Not so hard | Strong |

### 4.5.0 RESULTS ON RATE OF WEAR TEST

4.8

| Samples of <br> soap | Initial mass of <br> samples (g) | Final mass <br> using <br> (sabdarifa) <br> samples in (g) | Final mass using <br> (Enterolobium) <br> Samples in (g) | Observation |
| :--- | :--- | :--- | :--- | :--- |
| A | 10.00 | 5.78 | 5.88 | There is no marked <br> difference at the rate the |
| B | 10.00 | 6.26 | 5.93 | soap samples are used |
| C | 10.00 | 5.92 | 6.78 | up. |
| D | 10.00 | 5.89 | 5.85 |  |

A GRAPH SHOWING THE COMPARISON OF THE PH VALUE OF SAPONIN FROM TWO SOURCES USED


GRAPH SHOWING THE COMPARISON OF FOAMABILITY TEST CARRIED OUT ON THE SAMPLES


## CHAPTER FIVE

### 5.0 DISCUSSION OF RESULTS

### 5.1.0 EXTRACTION OF SAPONIN

The results of the experiment carried out on the extraction of saponin, showed that saponin could be extracted from the calyces of hibiscus sabdarifa (sorrel plant) material and also from the leaves of Enterolobium Cyclocarpum(An exotic plant) material.

The leaching of the material could be done by soaking it in cold water, but the best result was obtained by the first crushing or grinding the material and soaking it in hot boiled water.

Results also showed that about 10 ml of saponin can be extracted from the clear deep red solution treated with 10 ml of n -hexane and also about 15 ml of saponin can be extracted from the clear deep green solution of enterolobium Cyclocarpum treated with 10 ml of n -hexane.

### 5.2.0 EXTRACTION OF LEMON GRASS PERFUME

The process of extraction carried out during this experiment was the crude type, because this project was not so much concerned with extraction of perfume, no proper research was carried out on how to extract high quality perfume from lemon grass.

### 5.3.0 HARDNESS TEST ON SOAP SAMPLES

The result obtained from hardness test shows that in the first-two days of production, the samples are soft with no remarkable difference. This might be because dryer was not use. However in about seven days and above, the samples becomes very strong, i.e saponin does not increase on reduce the hardness of soap.

### 5.4.0 RESULTS OF FOAMABILITY TEST

The results of foamability test obtained is obvious. They showed that the more quantity of saponin added, the more the foamability of soap increases. The test also shows that foamability of saponin from enterolobium is more compare to that of saponin from sorrel plant (sabdarifa). The test shows that saponin increases the foaming of soap

### 5.5.0 RESULTS OF CORROSIVE TEST

This is another clear result the more saponin was added to soap samples, the more the percentage unconverted NaOH increased. This of a fact contradicts the interesting results obtained on foamability test

It showed that when large quantity of saponin is to be used, the soap will be highly corrosive and will require other additives to reduce the level of corrosiveness.

### 5.6.0 THE RATE OF WEAR TEST

There was no marked difference in the rate at which the soap samples would diminish when in use. This may be due to lack of difference in hardness when samples are properly dried

### 5.7.0 RESULTS ON pH MEASUREMENT

The PH values shows the saponin increases the alkalinity of soap. A sharp increase in alkalinity was noticed in soap samples prepared using saponin from enterlobium Cyclocapum. An increase was noticed but not as when saponin from hibiscus sabdarifa is used as soap additives.

## CHAPTER SIX

### 6.0 CONCLUSION

The research work has shown that saponin could be extracted from entedobium cyclocarpum and also from Hibiscus sabdarifa.

Analysis on soap sample, of calculated quantities of the substances showed that saponin (a foam producer) on its own) increases the foamability of soap. From the pH Values as recorded from a pH meter and result obtained on the corrosive test, it was observed that saponin increases the alkalinity of soap.

However, the test on rate of wear showed that saponin diminishes when in use. Also, hardness test showed that saponin does not reduce or increase the hardness test showed that saponin does not reduce or increase the hardness of soap

### 6.1 RECOMMENDATIONS

In view of the discussion which have been made above, the following recommendations are for future work

1. Other Solvent apart from $n$-hexane should be tried for the extraction of saponin. Their effectiveness should be tested and compared with that of normal hexane used in this work.
2. Other local plant materials should be used for the extraction of saponin, and the characteristics of saponin from them should be compared with that from sorrel plant and with that from enterolobium

## REFERENCES

1. William Benton; Toronto: Encyclopedia Britannica, Volume 16 Pp. 914.
2. Lexicon Publications; China: New Age Encyclopedia Volume 16 Pp. 564
3. Grolier Incorporated; Danbury: The Encyclopedia Americana volume 25.
4. Educational Corporation; Chicago: New Standard Encyclopedia.
5. Funk and Wagnalls, New Encyclopedia volume 21 Pp. 439.
6. Perry Robert .H (Ed) and Don W. Green; New York: Perry's Chemical Engineering Handbook 1984.
