All other values are calculated as above and the following table is generated

| n | $x$ | $\hat{Y}$ |
| :--- | :--- | :--- |
| 1 | 250 | 1.641 |
| 2 | 500 | 2.104 |
| 3 | 750 | 2.568 |
|  | $\Sigma X=1500$ | $\Sigma \hat{Y}=6.313$ |

The variation is calculated as

$$
V^{2}=\Sigma(\hat{Y}-\bar{Y})^{2} \text { and } V^{2}=\Sigma(Y-\bar{Y})^{2}
$$

Where

$$
\begin{aligned}
& \hat{Y}=\text { Estimated point of dependent variable } \\
& \overline{\bar{Y}}=\text { Average of dependent variable } \\
& Y=\text { Dependent variable }
\end{aligned}
$$

For $n=1$

$$
\begin{aligned}
& (\hat{Y}-\overline{\bar{Y}}\rangle=1.641-2.104=-0.463 \\
& (\hat{Y}-\bar{Y})^{2}=(-0.463)^{2}=0.214 \\
& (\bar{Y}-\bar{Y})=1.669-2.104=-0.435 \\
& (\bar{Y}-\bar{Y})^{2}=(-0.435)^{2}=0.189 .
\end{aligned}
$$

Following the above calculation for $n=2$ and $n=3$ the following table is generated:

| n | Y | Y | ( $\hat{\mathrm{Y}}-\mathrm{Y})$ | $(\hat{Y}-\bar{Y})^{2}$ | (Y-Y) | $(\mathrm{Y}-\overline{\mathrm{Y}})^{2}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 1.669 | 1.641 | -0.463 | 0.214 | -0.4355 | 0.189 |
| 2 | 2.050 | 2.104 | 0 | 0 | -0.054 | 0.003 |
| 3 | 2.594 | 2.568 | 0.464 | 0.215 | 0.490 | 0.240 |
|  | $\begin{aligned} & \Sigma \mathrm{Y}= \\ & 6.313 \end{aligned}$ | $\begin{aligned} & \sum \hat{Y}= \\ & 6.313 \end{aligned}$ | $\begin{aligned} & \Sigma(\hat{Y}-\vec{Y})= \\ & 0.001 \end{aligned}$ | $\begin{aligned} & \Sigma(\hat{Y}-\bar{Y})^{2}= \\ & 0.249 \end{aligned}$ | $\begin{aligned} & \Sigma(Y-\bar{Y})= \\ & 0.001 \end{aligned}$ | $\begin{aligned} & \Sigma(Y-\bar{Y})^{2}= \\ & 0.432 \end{aligned}$ |

The correlation coefficient is given by

$$
\begin{aligned}
& r^{2}=\frac{\sum(\hat{Y}-\bar{Y})^{2}}{\sum(Y-\bar{Y})^{2}}=\frac{0.429}{0.432}=0.9931 \\
& r=+\sqrt{ } 0.9931 \text { and this implies the graph is Linear. }
\end{aligned}
$$

THE STUDY OF THE EFFECTS OF SAPONIN ON THE ACTIVE INGREDIENT OF SOAP BY

CHIKWENDU, UGOCHUKWU GADUMBA (92/2493)
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 NIGERTA.
in partial fulfilment of the requirements FOR THE AWARD OF A bachelor degree in chemical engineering

I, hereby certify that I supervised, read and approved this project work presented by Chikwendu, Ugochukwu Gadumba (92/2493) in partial fulfilment of the award of Bachelor degree in chemical Engineering.

Project Supervisor


Head of Department.

External Examiner

Date


## DEDICATION

This project research is dedicated to my beloved parents
Mr Mathias Eze Chikwendu and Mrs Victoria Nwaneleuri Chikwendu for their parental care and for ensuring that $I$ attain this level of education.

## ACKNOWLEDGEMENT

It is my great pleasure to express my appreciation to my project supervisor Dr. M. O. Edoga 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 own a lot to the head of Department, Dr. J. O. Odigure, Dr. K. Aberuagba and Mr O. A. W. Akinbode, who really built up my courage to complete this research work. You have been very helpful to me, May the Almighty God reward you.

My appreciation is also due to other staff of chemical engineering Department, more especially the project coordinator Mr. M. Olutoye, and Mr . Abdul-Rahim.

I appreciate the help given to me by Mr. G. F. Ibikunle, a technologist in the Department of fisheries, who carried out extraction of saponin in his laboratory. I wish you more successes in further research engagements.

My profound gratitude goes to Mrs P. A. Nwafor and her husband, Mr 0. C. Nwafor for their moral and financial assistance to me. I owe a lot to the family of Engineer 0 . N. Wachuku, the chief engineer with the National electric power authority.

My special thanks also go to Mrs N. Onuora, Messrs Emmanuel, Humphery Ihuoma, Godson Amugwo and my darling sister Chioma.

I thank all my classmates for/support and encouragement. My prayers is that Almighty God will reward you.

Finally, to Almighty God be the glory and honour that the aim of this research work is achieved.

The project research was carried out to study the effects of saponin on soap. Experiments were conducted to test the effects of saponin on hardness, foamability, hydrogen ion concentration and rate of wear of soap.

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

Foamability and corrosive tests conducted show that saponin increases the foaming of soap and also the level of alkaline in teh soa; As a result, if saponin is to be sued in large quantity in soap formulation, other additives will be needed to bring down the level of alkaline.

A reactor was designed whose capacity is to produce 20000 Kg of soap per batch. In the design, equipment sizing and costing, power requirement and safety of operation were considered.
PAGE
TITLE PAGE ..... i
CERTIFICATION ..... ii.
DEDICATION ..... iii
ACKNOWLEDGEMENT ..... iv
ABSTRACT ..... v
TABLE OF CONTENT ..... vi
CHAPTER ONE
1.0 INTRODUCTION ..... 1
1.1 AIMS AND OBJECTIVES OF THE WORK ..... 2
CHAPTER TWO
2.0 REVIEW OF LITERATURE ..... 3
2.1 ORIGIN OF SOAP ..... 3
2.2.0 METHODS OF SOAP MANUFACTURE ..... 3
2.2.1 SOAP PRODUCTION PROCESSES ..... 5
2.2.2 CLEANSING PROPERTY OF SOAP ..... 5
2.2.3 PROPERTIES OF SOAP ..... 6
2.2.4 TYPES OF SOAPS ..... 7
2.3.0 SOAP CONSTITUENTS ..... 8
2.3.1 OIL AND FAT ..... 8
2.3.2 CAUSTIC SODA ..... 8
2.3.3 BRINE ..... 9
2.4.0 SOAP ADDITIVES ..... 9
2.4.2 OPTICAL BRIGHTENERS ..... 9
2.4.3 FILLERS ..... 10
2.4.4 ANTI-REDEPOSITE AGENTS ..... 10
2.4.5 PERFUMES AND COLOURANTS ..... 10
2.4.6 SAPONIN ..... 10
2.4.7 REACTION MECHANISM OF SAPONIN
3.0 EXPERIMENTS ..... 11
3.1 EXTRACTION OF SAPONIN FROM GLYCOSIDIC PLANT ..... 11
3.2 EXTRACTION OF PERFUME FROM LEMONGRASS ..... 12
3.3 PRODUCTION OF SOAP SAMPLES ..... 12
3.4.0 TESTS ..... 14
3.5.1 FOAMABILITY TEST. ..... 14
3.6.0 PH. MEASUREMENT ..... 1.4
3.7.0 HARDNESS TEST ..... 15
3.8.0 TEST FOR RATE OF WEAR ..... 16
CHAPTER FOUR
4.0 RESULTS ..... 17
4.1 COMPOSITION OF SOAP SAMPLES ..... 17
4.2 HARDNESS ..... 17
4.4 PH. VALUES ..... 18
4.5 RATE OF WEAR ..... 19
CHAPTER FIVE
5.0 PLANT DESIGN. ..... 20
5.1 INTRODUCTION ..... 20
5.2 CHOICE OF DESIGN. ..... 20
5.3 PROBLEM STATEMENT ..... 21
5.4. DESIGN DATA ..... 21
5.5 UTILITIES ..... 22
5.5. MATERIAL BALANCE ..... 25
5.7.0 EQUIPMENT SIZING ..... 28
5.7.1 REACTOR DIMENSION ..... 29
5.7.2 HEATING COIL DIMENSION. ..... 30
5.7.3 POWER REQUIREMENT. ..... 30
5.8 MATERIAL FOR CONSTRUCTION ..... 31
5.9 EQUIPMENT COSTING ..... 32
5.9.1 PROFITABILITY OF DESIGN ..... 32
5.9.2 SAFETY CONSIDERATION ..... 33
CHAPTER SIX
6.0 DISCUSSION OF RESULTS ..... 34
6.1 EXTRACTION OF SAPONIN ..... 34
6.2 EXTRACTION OF PERFUME FROM LEMON GRASS ..... 34
6.3 HARDNESS ..... 34
6.4 FOAMABILITY ..... 35
6.5 PH. VALUES ..... 35
6.6 THE RATE OF WEAR ..... 35
6.7 DESIGN WORK ..... 35
CHAPTER SEVEN
7.0 CONCLUSION ..... 36
7.1 RECOMMENDATIONS ..... 36
REFERENCES ..... 38

Soap is the common term for a number of related compounds 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 fats and oils, but other bases and naturally occuring carboxylic acids are also used to make a veriety of soaps. A soap is a detergent in that "detergent" means "cleaning agent", but usually only the eynthetic 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 soaps were of many different kinds. They might be hard or soft, depending on the kinds of fats and alkalis used in making them and on the skill of the soap maker. They might be of different colour including black. They most of these soaps were too harsh for house-hold use. In the modern method of soap production, they wood ashes are replaced by inorganic chemicals known as caustic alkalies. 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 difficulty 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 detergency of cleaning action of the soap. By their addition better and more attractive 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 study the effects of saponin (a local additve) on the saponification value of soap.

Saponin is one of the numerous plant glycosides characterized by foaming in water. The researc work will also invlove extraction of saponin from the plant material as well as the analysis of the saponin. Moreso, samples of soap will be produced which will be tested for hardness, fomability, corrosiveness, and rate of wear...

### 2.0 REVIEW OF LITERATURE

### 2.1 ORIGIN OF SOAP

No one knows who discovered soap. It is likely that someone many, many years ago made the first soap by boiling together ainmal fats and alkali made from wood ashes. While we can only guess about soaps origin, we do know that a crude kind of soap was used in France around A. D. 100. From France, the knowlede spread to other countries around teh mediterranean sea. By A.D. 800 Italy and spain were leading soapmaking countries. By the 12 th century at least a dozen European coutnries were making and exporting soap.

A great break - through in soapmaking occured in 1787. In that year a french scientist named Nicholas Leblanc discovered how to make an alkali called soda ash from ordinary salt. This discovery, which Leblane patented in 1791, meant that soap could be made at much lower cost. Then everybody in Europe could afford good soap. ${ }^{3}$

### 2.2.0 METHOD OF SOAP MANUFACTURE

VEgetable oils such as palm oil, groundnut Olive, kernel oil and animal fats are naturally occuring esters formed from a long chain organic acid, octadecanoic (stearic) acid, C17H35 COOH (containing 18 carbon atoms per molecule) and propan - 1,2,3, - triol (glycerol and alkanol containing three - OH groups 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:

### 2.4.3 FILLERS

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

### 2.4.4 ANTI - REDEPOSITE AGENTS

These include sodium carboxyl - methyl celluslose which suspends dirt in solution so that it cannot redeposit on the washed material.

### 2.4.5 PERFUMES AND COLOURANTS

Perfumes and colourants are added in soap to incorporate pleasant odour and appearance respectively. Perfumes include citronella oil, lemon grass oil, Bergamot andoil of bay. While colourants can be red, gree, blue, yellow, e.t.c.

### 2.4.6 $\frac{\text { SAPONIN }}{\text { is }}$

Saponin/a certain group of chemicals occuring in soapberry, soapwort, sorrel (hibiscus sabdarifa), and a number of other plants. These plants have long been used as soap substitutes in various countries, because saponins produce quantities of lather when the plants are, shaken with water. A commercial product also called saponin is derived from such $p$ lants and used as foaming and emulsifying agen for detergent. Saponin ae glycosides, or chemicals that yield sugar when hydrolyzed. However, one kind also yields steriods, while another kind are poisonous in the bloodstream because they dissolve red blood cells.


The Saponin is a surface active agent (surfactant) which dissociates in water to produce the polar and non-polar ends. The polar end is sugar like, while the non-polar end is the important aspect of the Saponin (the foaming end).

The extraction with $n$-hexane is of two significance. First, n-hexane extracts the secondly, frees the non-polar end from the polar end.

## CHAPTER THREE

### 3.0 EXPERIMENTS

This aspect of the project covers all the experimental procedure. - These include extraction of saponin from hibiscuss sabdarifa, extraction of perfume from lemon grass, production of soap samples.

### 3.1.0 EXTRACTION OF SAPONIN

Materials: (i) Calcyces of calyxes (sepals of hibiscuss sabdarifa) --a ring of leaves which protects the flower before it opens and later supports the opened flower, (ii) Norma1 hexane.

### 3.1.2 Apparatus: Glass soap reactor, mechanical stirrer, weighing

 balance, Glass funnel, Filter paper and soap mold, wash bottle.
### 3.1.3 Method

Step 1: SOLID LIQUID EXTRACTION (LEACHING)
100 Kg of hibiscus sabdarifa was ground to powerdery form and soaked in $1000 \mathrm{~cm}^{3}\left(1 \mathrm{dm}^{3}\right)$ boiled water. The misture 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.

Step 2:
50 g of the solution was poured into a separating funnel and 10 g of $n$-hexaire added and shaken a number of times. The mixture turned foamy 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 funne1. The foamy extraction on top which contain some $n$-haxane 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.

Figure 1 shows the sketch of the saparating funnel.

### 3.2.0 EXTRACTION OF LEMON GRASS PERFUME

Materials- Lemon grass, and n-hexane.
3.2.1 Apparatus:- Mortar 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 was placed in the motar and pounded with a pestle. After pounding it to very ting pieces, it was transfered 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 2vaporate. 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 Method

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

Step 1: Preparation of Oil and NaOH
To obtain a very good soap, the percentage of caustic soda in solution has to be $40 \%$. To obtain this formation, the ratio of water to caustic Soda (300g: 200g) was used. 200 g of caustic soda was dissolved in 300 g of water ald used against the oil. Palm oil was bleached by either activated clay or by heating.


## Step 2: Saponification

$50 \%$ of oil was pured into a sample container and 16.85 of sodium hydroxide, NaOH , solution was mixed with it. The mixture was stirred vigorously for 1 minute, after which calculated quantity of saponin which had already been boiled and cooled. The boiling of the saponin was to free it of n-haxane. Stirring was continued until the paste becomes thicker. It was allowed to stand for several hours to cool and solidify.

Five more samples of teh same formation were produced with different amounts of saponin added.

On extraction, the hexane is entangled with the saponin. A little heating evaporates the hexane, leaving the saponin free.

### 3.4.0 TESTS EOAMABILITY TESTE-

### 3.5.1 MATERIALS AND APPARATUS

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

### 3.5.2 PROCEDURE

20 g of each sample of soap was accurately weighed and ground before and made into 1000 ml uniform solution. Then 500 ml of the soap solution was poured into another 1000 ml measuring cylinder and a mechanical stirrer inserted well into the solution. The stirrer is one 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 4.4 for the results, and figure $\mathbf{3}$ for sketches.

### 3.6.0 CORROSIVE TEST

This was based on calculating the percentage of uncoverted caustic soda, NaOH , in the soap sample as different saponification products have different degree of reactivities to the human skin fabrics, plastic and metal surfaces. The test deternined how mild or hazardous the soap samples was.

### 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 APPARATUS 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 the soap samples having equal surface area and volume were cut with a knife. These equal volume of samples were then put into different conical flasks. Equal volume of water ( 150 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 removed from. the flask using a fork and weighed.

See table 4 for the results obtained during the experiment.

## CHAPTER FOUR

4.0

RESULTS AND CALCULATIONS
4.1.0. COMPOSTTION OF SOAP SAMPLES

Tables 4.1 and 4.2 show the composition of the soap samples prepared using palm oil and groundnut oil, respectively.
4.1

| Sample | Composition in Grammes |  |  |  | ) | (ml) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| of <br> Soap | Palm oil | $\mathrm{NaOH}$ <br> Solution | $\mathrm{H}_{2} \mathrm{O}$ | Perfume | Dye | Saponin |
| A | 50 | 16.85 | 10.00 | 1.0 | 1.0 | - |
| B | 50 | 16.85 | 10.00 | 1.0 | 1.0 | 5 |
| C | 50 | 16.85 | 10.00 | 1.0 | 1.0 | 10 |
| D | 50 | 16.85 | 10.00 | 1.0 | 1.0 | 15 |

4.2

| Sample | Composition in Grammes |  | $(\mathrm{g})$ | $(\mathrm{ml})$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| of <br> Soap | Groundnut | NaOH | $\mathrm{H}_{2} 0$ | Perfume | Dye | Saponin |
| A | 50 | 16.85 | 10.00 | - | - | - |
| B | 50 | 16.85 | 10.00 | - | - | 5 |
| C | 50 | 16.85 | 10.00 | - | - | 10 |
| D | 50 | 16.85 | 10.00 | - | - | 15 |

## RESULTS ON FOAMABILITY TEST

The Table 4.3 gives the results obtained during foandability test. The net height of foam of samples are recorded for the set of samples prepared using palm oil and groundnut oil.

```
4.3
```



```
                    pH READINGS OF THE SAMPLES
    pH values of the various samples as measured from a pH meter
is shown on Table 4.5:
```



## RESULTS ON HARDNESS TEST

The Lable 46 gives the results obtained during the test for hardness of the soap samples produced.

| Samples of soap <br> (sets 1 and 2) | Quantity of saponin added | Hardness |  |
| :---: | :---: | :---: | :---: |
|  |  | After 2 days | After 7 days |
| A | - | Not so hard | Strong |
| B | 5 (m1) | Same as above | $\checkmark \checkmark$ |
| C | 10 | Same as above | $\checkmark \checkmark$ |
| D | 15 | Same as above | $V$ |

## RESULTS ON RATE OF WEAR TEST

| Samples <br> Soap | Intial mass of sample (g) | Final mass palm oil sample in (g) | Final mass of of groundnut oi'l samples (g) | Observation |
| :---: | :---: | :---: | :---: | :---: |
| A | 10.00 | 5.64 | 5.99 | there is no |
| B | 10.00 | 5.97 | 6.13 | marked difference |
| C | 10.00 | 6.20 | 5.78 | at the rate the |
| D | 10.00 | 5.78 | 5.86 | soap samples are used up. |

## CHAPTER FIVE

### 5.0 PLANT DESIGN

5.1 INTRODUCTION

The plant to be designed is that of a soap plant which on operation with produce 20000 Kg ( 20 tons) of soap flakes per batch. There will be determination of material and energy balances around each process equipment. The determination will be based on experimental results, assumed values and conditions.

There will be detailed design of a soap reactor for the saponification process which will involve a vegetable oil or fat and sodium hydroxide solution. In the detailed design of the reactor, the design specification will be obtained through calculations. Also, the materials for construction will be considered to ontain the best type of material that can withstand the heat of reaction and the corrosive nature of concentrated sodium hydroxide. Finally, there will be equipment costing to determine the profitability of design, and safety consideration to minimise risks and loses.

### 5.2 CHOLCE OF DESIGN

The choice of design is a reactor. It is one of the most important equipment in a soap plant and many other chemical manufacturing process plants. In the reactor, the raw materials.

soap is feel to dryer at $25^{\circ} \mathrm{C}$
Dryer operates at $100^{\circ} \mathrm{C}$.
No accummulation of producet in finishing will.
There is accummulation of $0.1 \%$ of soap in plodder.
$20 \%$ of molten soap is evaporated in the dryer as water.
\% additives added/total soap produced
Factor of safety is $10 \%$ of reactor volume.
5.4.1 UTILITIES

1. Steam
2. Electricity.
3. Clean water.

### 5.5 MATERIAL BALANCE OF PLANT

Basis : 1 batch
OUtput: $20000 \mathrm{~kg} /$ batch
MATERIAL BALANCE AROUND FINISHING MILL
No accummulation of product in finishing mill.


MATERIAL BALANCE AROUND THE PLODDER
Assumption: The is accummulation of $0.1 \%$ of the soap from dryer.


$$
\begin{aligned}
D & =99.9 \% \text { of } C \\
& =\frac{99.9}{100} C=0.999 \mathrm{C}
\end{aligned}
$$

ie, $0.999 \mathrm{C}=20000$
i.e $C=\frac{20000}{0.999}=20020 \mathrm{~kg}$

Accummulation in plodder $=20020-20000$
$=20 \mathrm{~kg} / \mathrm{batch}$
Material balance around the dryer
Assumption: $20 \%$ of molten soap into the dryer is evaporated as water

$F=$ amount of water evaporated
$E=$ Amount of molten soap coming into the dryer
$G=$ Amount of dried soap of flakes.
MATERIAL BALANCE:

$$
\begin{aligned}
& E=F+G \\
& F=20 \% \text { of } E=\frac{20}{100} E=0.2 E
\end{aligned}
$$

$\rightarrow E=0.2 E+20020$
ie. $E-0.29=20020$
$\Rightarrow E=\frac{20020}{0.8}=25025 \mathrm{~kg}$ of molten soap
Amount of wate evaporated $=0.2 \mathrm{E}=0.2 \times 25025$

$$
=5005 \mathrm{~kg} .
$$

MATERIAL BALANCE AROUND THE MIXER


Assumption:- Addotoves added os $5 \%$ of molten soap coming out of mixer.

$$
I=5 \% \text { of } \mathrm{J}=0.5 \mathrm{~J}=1251 \mathrm{~kg}
$$

Saponin $=60 \%$ of the total additives added.

$$
=\frac{60}{100} \times 1251=751 \mathrm{~kg} \text { of saponin }
$$

Amount of other additives $=1251-751=500 \mathrm{Kg}$
Material Balance:

$$
\mathrm{H}+\mathrm{I}=\mathrm{J}
$$

ie. $\quad H=J-I$
$=25025-1251=23774 \mathrm{~kg}$ of soap from reactor .

## MATERIAL BALANCE AROUND THE REACTOR

From the saponification reaction below:

where $\mathrm{R}=\mathrm{C}_{17} \mathrm{H}_{35}$
For quantity of oil Used:
890 g of oil $\longrightarrow 3$ (306) $=918 \mathrm{~g}$ of soap
i.e 0.890 kg of oil $\longrightarrow 0.918 \mathrm{~kg}$ of soap.

Product from reactor $=$ Neat Soap + Glycerol .
Quantity of neat soap $=$ quantity of neat soap going into the mixer. $=23774 \mathrm{~kg}$

From saponification equation;
$\begin{aligned} 0.890 \mathrm{~kg} \text { of oil } & \longrightarrow 0.918 \mathrm{~kg} \text { of soap } \\ \mathrm{xkg} \text { of o11 } & \longrightarrow 23774 \mathrm{~kg} \text { of soap } \\ \mathrm{xkg} \text { of o11 } & =\frac{23774 \times 0.890}{0.918}=23049 \mathrm{~kg} \text { of o11 used. }\end{aligned}$

For quantity of NaOH used;-
From the saponification equation above, $3(40) \mathrm{g}$ of NaOH $\qquad$ 3(306)g of soap
1.e $\quad 120 \mathrm{~g}$ of NaOH $\qquad$ 918 g of soap
i.e $\quad 0.120 \mathrm{~kg}$ of NaOH $\qquad$ 0.918 kg of soap

$x \mathrm{~kg}$ of $\mathrm{NaOH}=23774 \times 0,120$
0.918
$=3108 \mathrm{~kg}$ of NaOH .
For a good soap, mass of NaOH in solution is $40 \%$,
Let mass of NaOH solution $=\mathrm{Y}$
ie. $0.4 y=3108$
$\Rightarrow y=\frac{3108}{0.4}=7770 \mathrm{~kg}$ of NaOH solution.
Operating capacity of reactor $=23049+7770$
$=30819 \mathrm{~kg}$
Quantity of soap $=23774 \mathrm{~kg}$
Quantity of glycerol = Operating capacity of reactor -
Quantity of soap
$=30819-23774=7045 \mathrm{~kg}$

### 5.6 ENERGY BALANCE CALCULATION

Energy balance around dryer;

25.

```
    Hsl = Energy supplied by soap feed at 25*'C
    Hs2 = Energy absorbed by dried soap at 100 C
    Hw = Total energy absorbed by evaporating H2O.
    = Sensible heat + Enthalpy of H2}\mp@subsup{\textrm{H}}{2}{}\mathrm{ .
    Q = Total energy supplied to dryer.
Cp of soap = 1.6472 KJ/kgk
Cp of water = 4.216 KJ/kgk
Enthalpy of lkg of water = 2506 KJ/hr.
Hs1 = M1CP (soap t=25025 < 1,6472 x 25 = 1030530 KJ
Hs2 = M2Cp soap t=20020 x 1.6472 x (100-25) = 2473271KJ
Hw = sensible Heat + Enthalpy of water.
ie. Hw = M M H2O CP P
    =5005 x 4.216 x (100-25) + 5005 (2506)
    = 14125111 KJ
Q = Total energy supplied
    = Hs2 + Hw - Hsl = 2473271 + 14125111 - 1030530
    = 15567852KJ
```



Hoil $=$ energy supplied by oil at $35^{\circ} \mathrm{C}$
$\mathrm{HNaOH}=$ energy supplied by NaOH solution at $25^{\circ} \mathrm{C}$
HsG $=$ energy absorbed by glycerine and soap at $120^{\circ} \mathrm{C}$
Q $\quad=$ Total energy supplied.
\% of Glycerine in reactor $=\frac{7045}{30819} \times 100=23 \%$
$\%$ of soap in reactor $=\frac{23774}{30819} \times 100=77 \%$
Mean Cp of (glycerine + water) mixture is given as;
\% of glycerine in reactor x Cp of glycerine $+\%$ of soap in
reactor $\mathrm{x} C \mathrm{p}$ of soap.
$=0.23 \times 2.428+0.77 \times 1.6472$
$=1.826784$
H(oil) $=$ Moil Cpoil $t=23049 \times 1.675 \times 35=1351248 \mathrm{Kj}$
$\mathrm{H}(\mathrm{NaOH})=\mathrm{M}_{\mathrm{NaOH}} \mathrm{C}_{\mathrm{PNaOH}}=7770 \times 3.4526 \times 25=670668 \mathrm{KJ}$
HsG $=$ (mass of Glycerine + mass of soap) $x$ Mean $C p x \quad t$
$=30819 \times 1.826784 \times(120-25)$
$=5348467 \mathrm{KJ}$
$Q=H s G-H(o i l)-H(N a O H)$
$=5348467-1351248-670668$
$=3326551 \mathrm{KJ}$
Steam Suppiled
From the energy balance calculated around the ractor, the quantity of steam supplied per batch is given by
$q=Q$
latent heat of steam at $12^{\circ} \mathrm{C}$
$=\frac{3326551}{2202}=1511 \mathrm{~kg} /$ batch

But a batch $=6 \mathrm{hrs}$
i.e Quantity of steam supplied in 1 hour is given as

$$
\frac{1511}{6}=252 \mathrm{~kg} / \mathrm{hr} .
$$

27. 

5.7 .0

## EQUIPMENT SIZING



The diagram above is that of a reactor, where DT $=$ Diameter of the reactor
$\mathrm{hT}=$ height of the reactor
$\mathrm{VR}=$ Volume of reactor
VT $=$ Capacity of the reactor
Sf = Factor of safety $=10 \%$
$V R=V T+(V T \times$ factor of safety)
$=\mathrm{VT}+0.1 \mathrm{VT}$
But Vt $=\frac{\mathrm{D}_{\mathrm{T}}^{2} \mathrm{~L} \pi}{4}+\frac{4 \pi \mathrm{ab}^{2}}{6}$
But $\mathrm{L}=1.3 \mathrm{DT}, \mathrm{a}=0.5 \mathrm{DT}, \mathrm{B}=0.5 \mathrm{DT}$

## 3

substituting these values in equation 2 ,

$$
\begin{equation*}
\text { i.e } V T=\frac{1.3 \mathrm{DT}^{3}}{4}+\frac{0.5 \pi \mathrm{D}^{3} T}{54} \tag{3}
\end{equation*}
$$

From material balance,
Mass of reactor content $=30819 \mathrm{~kg}$
Density of the reactor $=1820 \mathrm{~kg} / \mathrm{m}^{3}$
i.e Capacity of reactor $V T=\frac{30819}{1820}=16.93 \mathrm{~m}^{3}$

From equation 1;

$$
\begin{aligned}
V R & =V T+0.1 V T \\
& =16.93+0.1(16.93) \\
& =18.63 \mathrm{~m}^{3}
\end{aligned}
$$

From equation 3,

$$
\mathrm{VT}=\frac{1.3 \mathrm{D}^{3} \mathrm{~T} \pi}{4} \quad+\frac{0.5 \pi \mathrm{D}^{3} \mathrm{~T}}{54}
$$

i.e $16.93=\frac{1.3 D^{3} T}{4} \pi+\frac{0.5 \pi D^{3} T}{54}$

$$
=\frac{54(1.3) \pi D^{3} T+4(0.5) \pi D^{3} T}{216}
$$

i.e $16.93=\frac{(70.2+2) \pi 0^{3} T}{216}$
i.e $72.2 \pi D^{3} T=3656.88$

$$
D_{T}^{3}=\frac{3656.88}{72,2 \pi}=16.12 \mathrm{~m}
$$

DT $=$
16.12
$=2.53 \mathrm{~m}$
Vessel height $=\mathrm{hT}=\mathrm{L}=1.3 \mathrm{DT}=1.3 \times 2.53=3.29 \mathrm{~m}$
Radius of vessel $=a=0.5 \mathrm{DT}=0.5 \times 2.53=1.265 \mathrm{~m}$
$\mathrm{b}=0.5 \mathrm{DT}$
3
$\frac{1.265}{3}$ $=0.422 \mathrm{~m}$
5.7 .1

Agiltator/Reactor dimension
Height of reactor $=1.3 \mathrm{DT}=1.3 \times 2.53=3.289 \mathrm{~m}$
Height of content in reactor $=H=D T=2.53 \mathrm{~m}$ $E=$ distance between paddle and reactor base
$\frac{\mathrm{E}}{\mathrm{DT}}$
$=$


$$
\frac{2.53}{3}=0.843 \mathrm{~m}
$$

Width of paddle $\mathrm{W}=0.2 \mathrm{DT}=0.2 \times 2.53=0.506 \mathrm{~m}$
Diameter of paddle $=1 / 3 \mathrm{DT}=1 / 3 \times 2.53=0.843 \mathrm{~m}$

### 6.0 DISCUSSION OF RESULTS <br> \&.! 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.

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

Results also showed that about 10 g of saponin can be extracted from the clear deep red solution treated with 10 g of normal hexane.

### 6.2 EXTRACTION OF LEMON GRASS PERFUME

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

For processes like the soap production where only the odour is required, and purity does not matter so much, it can best be extracted by first washing and crushing the leaves into very tiny particles. This is boiled with calculated quantity of water. The greenish solution which is obtained after filtering is used as part of process water. This method is better than using $n$ - hexane for the extraction because $n$ - hexane contaminates the odour of the lemon grass.


#### Abstract

6.3 Hardness Test on Soap Samples

The results obtained from hardness test show that in 1 to 2 days of production, when the soap samples are not yet sent, the samples are soft with no remarkable difference. However, in about 7 days and above, the samples are hard and of equal hardness, i.e saponin does not increase or reduce the hardness of soap.


### 6.4. Results of Foamability Test

The results obtained on foamability test is obvious. They showed that the more quantity of saponin added, the more the foamability of soap increases. The test showed that saponin a surface active agent characterised by foaming in water, supports and increases the foaming of soap.

### 6.5 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 contradicts the interesting results obtained on foamability test. It showed that when saponin is to be used in large quantity, the soap will be corrosive and will require other additives to reduce the level of corrosiveness.

### 6.6 The Rate of Wear Test

There was no marked difference in the rate at which the soap samples would diminish when in use. This might be due to no difference in hardness when samples are properly dried. 6.7 Design Work

A plant which would produce 20000 kg ( 20 tonnes) of soap flakes per batch was designed using the data stated in 5.4. Material balances were calculated for the flow in and out each process equipment. Also energy balances were calculated for the dryer and the reactor.

Detailed design of the reactor were carried out to determine the dimensions and power requirement of the equipment.

The pH values show that saponin increases the alkalinity of soap A sharp increase in alkalinity was noticed in soap samples prepared with palm oil. In the case of samples prepared with groundnut oil, an increase is noticed but not as sharp as when palm oil is used in preparing the soap.

The pH values confirm the results obtained during corrosive test when it was discovered that saponin increases the percentage of unconverted sodium hydroxide (\% NaOH ) of soap.

## CHAPTER SEVEN

## 7.0

 CONCLUSIONThe research work has shown that saponin could be extracted from hibiscus sabdarifa, a soap plant which contain the saponin on its calyces.

Analysis on soap samples with calculated quantities of the substance showed that saponin (a foam producer on it own) increases the foamability of soap. From the pH values as recorded from a pH meter and results 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 has no remarkable effect on the rate at which soap deminishes when in use. Also, hardness test showed that saponin does not reduce or increase the hardness of soap.

### 7.1 RECOMMENDATIONS

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

1. Other solvents such as alcohol 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. This project should be repeated using other grades of oil and/or fat in place of bleached palm. What the effect of saponin will be on the soap samples prepared using these grades should be compared with that obtained in this work.
3. In this project work, saponin was obtained from hibiscus sabdarifa material: 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.

## REFERENCES

1. Encyclopedia American; International Edition; Grolier

Incorporated; volume $25,1980 \mathrm{pg}$. 117. 1 Ition; Grolier
2. The New Book of knowledge; International Edition; Grolie

International incorporated; D. Volume 4, 1980; Grolier
3. Aberuagba, K. Dr. Plant Design (CEE 515)

Engineering; 1997/98 Session. (CEE 515) note; Chemical
4. The Encyclopedia Americana, International Edition; Grolier

Incorporated; Volume 24, 1980; pg. 253 Edition; Grolier
5. Richardson and Coulsin; chemical Engineering; New Edition;
Volume 6, 1980, pg. $223-237$
6. Mc Graw-Hill, encyclopedia of Science and Technology, 5th

Edition, McGraw-Hill Company, Volume 12, 1982, pg. 488
7. Perry R,H; Perry's Chemical Engineeers Handbook, McGraw-Hill
Company, pg. 8.23
8. Parmeggiani, Luigi Dr, Encyclopedia of Occupational Health and Safety, Third (Revised) Edition, volume 1, pg. 254-259.

