INSPECTIONS OF FINISH CHARACTERISTICS SUCH AS TOTAL FATTY MATTER (TFM), FREE CAUSTIC ALKALI (FCA), ANDFREE FATTY ACID (FFA) OF SOVEREIGN BAR SOAP @ BCC LANKA LIMITED

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This thesis is submitted in partial fulfillment of the requirement for the General Degree of Bachelor of Sciences (Applied Sciences) in Applied Chemistry

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March 2009

DECLARATION

I certify that this dissertation, which was practically, implemented by me at the BCC Lanka Limited of Sri Lanka, and the Faculty of Applied Sciences under the supervision of N. M. Wickramaratne and N. B. Senarath, I did not follow any other degree or diploma in any other university and to the best of my knowledge does not contain another person where due references is made in the text.

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Affectionately

Dedicated

To My Parents
&
To All My Teachers

ACKNOWLEDGEMENT

First, I would like to offer my sincere gratitude to my internal supervisor, Dr. (Mrs.) N. M. Wickramaratne, Senior Lecture, Department of Physical Sciences, Sabaragamuwa University of Sri Lanka; for her encouragement, guidance and she gave me to necessary instruction to complete this project successfully.

I would also like to express my grateful thanks to my external supervisor, (Mr.) N.B. Senarath, Assistant Production Manager, BCC Lanka Limited, for his advice, guidance, confidence, help and instruction to make this study success.

Next I specially acknowledge the Head of the Department of Physical Sciences Dr. C. P. Udawatta, for sharing his valuable time with us and his guidance in occasions where it was needed. Further more my sincere thanks go to all the lectures and Instructors the University of Sabaragamuwa.

Next I would like to extend my sincere gratitude to Mr. S. A. R. Maddumabandara, Chairman of BCC Lanka Limited, giving me this great opportunity to do my project in there company. Also I would like to convey my thanks to all BCC staff members for there support.

Finally I would like to express my deepest sincere thanks to everyone for their encouragement who tremendous supported me a lot for the successful completion of my project period.

ABSTRACT

Sovereign bar soap is the main product of BCC Lanka Limited and is a product of hydrolysis of Oils and fats with sodium hydroxide. In soap industries there are important quality characteristics to that must be monitor regularly such as Total Fatty Matter (TFM), Free Caustic Alkali (FCA) and Free Fatty Acid (FFA). These characteristics mainly effect to the quality of the product. Sri Lanka Standards Institution (SLSI) has provided with the standard requirements for the of Laundry soap.

The above parameters were determined for a period of 3 months to ensure the quality of the final product. TFM was analyzed using the ether extraction method Sri Lanka Standards Institution (SLSI) protocol, FCA was analyzed using ethanol method Sri Lanka Standards Institution (SLSI) protocol and FFA was analyzed as per Sri Lanka Standards Institution (SLSI) protocol.

The results obtained were analyzed using MINITAB 14. According to the Wilcoxon Signed Rank Test the test results revealed that Total Fatty Matter (TFM) – 59.31%, Free Caustic Alkali (FCA) – 0.043%, Free Fatty Acid (FFA) – 0.56%.

The TFM and FCA results indicated that the products are in line with the SLSI standards, and FFA result is with that of the company standard.

CONTENT

LIST OF ABRIVIATIONS

°C	-	Degrees of Celsius
°F	-	Degrees of Faranhide
Kg	-	Kilo gram
G	-	gram
Mg	-	Milligram
m	-	Meter
ml	-	milliliter

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CHAPTER 1
INTRODUCTION

1.1 Introduction

BCC Lanka Limited was established on February 28th 2006. The main business activities are manufacturing, distribution, and marketing of coconut oil related products as detergents, toiletries and other personal care products, and produce steel drums for the purpose of storing oils greases and industrial products such as asphalt etc.

Soap productions of the company are sovereign bar soap, shaving soap, soft soap, coal tar and night & day. The main production is sovereign bar soap. It is produced by using semi-boiled process with pure coconut oil with caustic soda. Semi-boiled process does not permit the removal of waste lye contains the glycerin produced in the soap making process. Therefore sovereign bar soap also contains glycerin, but it can manufacture in short time. That process are produce soaps as batches, the company produce 12 batches with in a weak.

Soap when pure, consist of 3 substances, fat, alkali, and water in a state of more or less intimate union. Soap always contains the fat in 2 conditions, i.e mostly combined, but a small amount of free, while the alkali may be present partly free and partly combined, with the fat. In case if more elaborate analysis is required, then a complete analysis of soap is determined. Following constituents are present in the soap,
(Combined fat, free fat, combined alkali, free caustic alkali, carbonate alkali, water etc)
Total Fatty Matter (TFM), Free Caustic Alkali (FCA) and Free Fatty Acid (FFA) are some important quality characteristics of soap industries. Sri Lanka Standards Institution (SLSI) has standard for the above characteristics.

1.1.1 Analysis of TFM

Total Fatty Matter means the fatty material obtained by decomposing the soap with a strong mineral acid and extracting the separated fatty matter with diethyl ether under the operating conditions described. This term includes unsaponifiable matter, glycerides and any resinic acids contained in the soap, in addition to the fatty acids derived from the soap.
The TFM value also an indication of the quality of the product. The more has better

the quality of the soap, anything above 59% considered to be good quality product of laundry soaps. TFM value can be analysis by ether extraction method. When TFM value is high, it has high cleaning active.

1.1.2 Analysis of FCA

The caustic alkali present in soap in 2 forms, free and combined. The combined alkali is that which is combined with the fat to form the soap. The free caustic alkali is that remains in the reaction medium without react with fat. If the soap is grainy, or turbid and somewhat white, it indicates a high level of unreacted caustic. Therefore free caustic alkali value also reason for the quality of the final product.FCA value can be analysis in 2 methods, Ethanol method and Barium chloride method.

The barium chloride method should be applied to all soft potassium soaps or, mixed sodium and potassium soaps. Sovereign bar soap is manufactured by using sodium hydroxide; therefore the ethanol method is used for analysis the FCA. The FCA value should be above the 0.1% to be good quality laundry soap.

1.1.3 Analysis of FFA

In soap industry, FFA value of soap is not mainly effect to the quality of product.FFA normally expressed as oleic acid. The exceptions are coconut and palm kernel oils, which are calculated as lauric acid. Therefore in Sovereign bar soap, FFA value is determined as a lauric acid. The company stands of FFA is 1% max.FFA can be analysis under the determination of unsaponified matter.

1.2 OBJECTIVE

To determine the Total Fatty Matter (TFM), Free Caustic Alkali and Free Fatty Acid (FFA) of Sovereign bar soap product by using an acceptable chemical process.

1.3 SPECIFIC OBJECTIVE

Inspections of the Total Fatty Matter, Free Caustic Alkali results revolved that there are with in the SLSI standard, and Free Fatty Acid result is with in the company standard.

CHAPTER 2
LITERATURE REVIEW

2.1 What is soap?

Soap is a cleansing substance which makes lather in soft water. Soap is a salt of a certain fatty acid, resulting from the chemical reaction of fats and alkali. Fats and oils are primary triglycerides. Triglyceride is a molecule of glycerol linked up with three molecules of fatty acids. During the saponification the triglyceride is broken down with alkali to glycerol and fatty acids; the latter combining with sodium in the alkali to form soap molecules.

2.2 Chemistry of soap

The chemical reaction of making soap may be representing as follows;

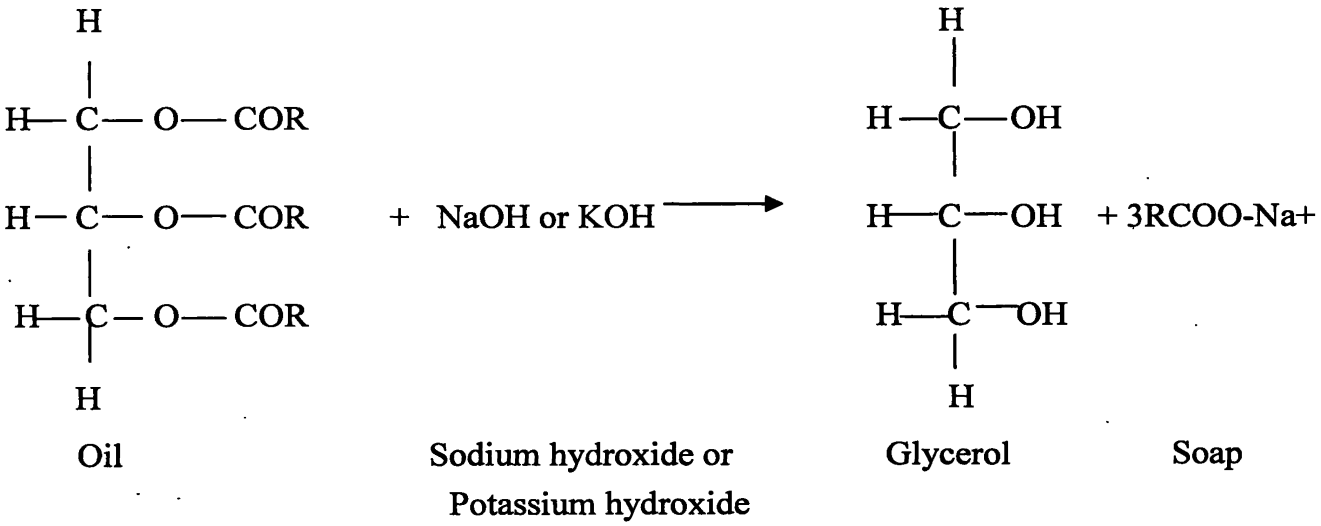


Fig 2.1 Chemical reaction of soap production

"R" long chain hydro carbon (Lauric, Oleic, Palmitic, Stearic) etc.

The above reaction is named as saponification.The reaction takes place in presence of water in stages with the formation of diglycerides, monoglycerides and finally glycerin with simultaneous neutralization of the liberated fatty acids by the alkali to form soap.

2.2.1Cleaning action of the soap

2.2.1.1 Surface tension

Water, the liquid commonly used for cleaning has a property called surface tension. In the body of the water, each molecule is surrounded and attracted by other water molecules. However, at the surface, those molecules are surrounded by other water molecules only on the water side. A tension is created as the water molecules at the surface are pulled in to the body of the water. This tension causes water to bead up on surface (glass, fabric), which slows wetting of the surface and inhibits the cleaning process. In the cleaning process, surface tension must be reduced so water can spread and wet surfaces. Chemical that are able to do this effectively are called surface active agents,or surfactants. They are said to water "wetter".
(http://en.wikipedia.org/wiki/soap-making)

2.2.1.2 Surfactants

Surfactants are compound that have a dual affinity ; They are both lipophilic & hydrophilic A surfactant molecule consist of a lipophilic tail group, which links to greasy soil, and a hydrophilic and polar head group, which renders the molecule water soluble ; this arrangement helps to disperse and rinse away greasy soil. Variations in the balance between hydrophobic and hydrophilic features determine the use of surfactant as a detergent, wetting agent or emulsifier. Surfactants are classified according to the nature of the hydrophilic head. There are four main classes: anionic, cationic amphoteric and nonionic. "Natural" soap contains an anionic surfactant. The majority of surfactants that are used in cleansing bars have anionic head groups. (http://en.wikipedia.org/wiki/soap-making)

2.2.1.3 Soaps are emulsifiers

Each soap molecule has a long hydrocarbon chain, sometimes called its "tail", with a carboxylate "head".

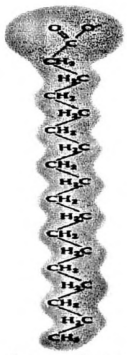


Fig 2.2 Hydrocarbon chain "tail" and carboxylate "head" of soap molecule

In water, the sodium or potassium ions float free, leaving a negatively charged head. Soap is an excellent cleanser because of its ability to act as an emulsifying agent. An emulsifier is capable of dispersing one liquid in to another immiscible liquid. This means that while oil (which attract dirt) dose not naturally mix with water, soap can suspend oil/dirt in such a way that it can be removed. The organic part of a natural soap is a negatively charged, polar molecule. Its hydrophilic (water loving) carboxylate group (-CO2) interacts with water molecules via ion dipole interactions and hydrogen bonding. The hydrophobic (water fearing) part of a soap molecule, its long nonpolar hydrocarbon chain, does not interact with water molecules. The hydrocarbon chains are attracted to each other by dispersion forces and cluster together,forming structure called micelles.
(http://chemistry.about.com/library/weekly/aa081301a.htm)

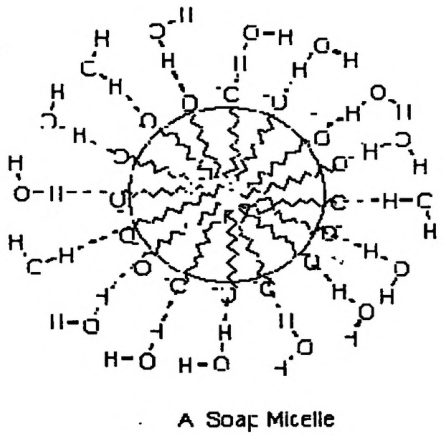


Figure 2.3 A soap micelle

In these micelles, the carboxylate groups form a negatively charged spherical surface, with the hydrocarbon chains inside the sphere. Because they are negatively charged, soap micelles repel each other and remain dispersed in water. Grease and oil are nonpolar and insoluble in water. When soap and soiling oils are mixed, the nonpolar hydrocarbon portion of the micelles break up the nonpolar oil molecules. A different type of micelles then forms, with nonpolar soiling molecule in the center. Thus, grease and oil and the 'dirt' attached to them are caught inside the micelle and can be rinsed away. (http://chemistry.about.com/library/weekly/aa081301a.htm)
Although soaps are excellent cleansers, they do have disadvantages. As salts of weak acids, they are converted by mineral acids in to free fatty acids;

$$3RCOO^{-}Na^{+} + HCl \longrightarrow 3RCOOH + Na^{+} + Cl^{-}$$

These fatty acids are less soluble than the sodium or potassium salts and form a precipitate or soap scum, because of this; soaps are ineffective in acidic water. Also,

soaps form insoluble salts in hard water, such as water containing magnesium, calcium or iron. (http://chemistry.about.com/library/weekly/aa081301a.htm)

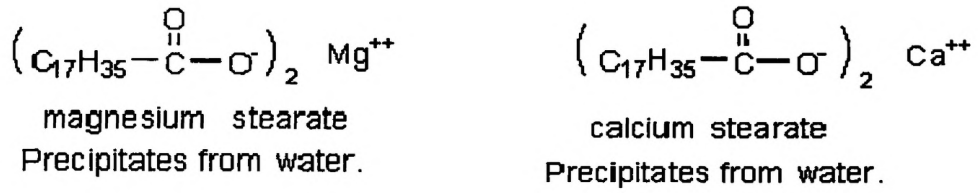


Figure 2.4 Magnesium and Calcium stearate precipitates from water

To achieve the same washing or cleaning action, more soap must be added.

2.3 Manufacturing Technology of soaps

There are three common methods and principles which are usually employed in the soap manufacturing units. These processes are Cold process, Semi- boiled process and Full- boiled process.

2.3.1 Cold Process:

This is the simplest method of saponification requiring little capital equipment. Cold process basically involves mixing and stirring. Caustic alkali / soda is dissolved in a suitable container, checking for final concentration or solution strength using hydrometer. A suitable amount of oil is poured into the reaction vessel. Caustic soda is poured on the oil charge and the whole stirred continuously. The whole essence of soap making in the cold is to keep the oil and water phases in an intimate mix. Colors and perfumes are added. The mixture thickens by the process, and it is the consistency that serves as indicator for reaction completeness. When molten soap is cooled, the sides of the frame are detached to facilitate the removal of the cooled, solid soap block. In lathe course of the cooling process, saponification continues for a day or so, during which time there is a temporary rise in temperature prior to normal cooling. It is desirable to conserve this heat so as to effect as complete a saponification as is possible, to ensure that no free caustic remains in the final product.

2.3.2 Semi-Boiled Process

This is merely a modification of the cold process. In brief, the method involves much the same operations as the cold process, except that the temperature of the reaction mix lies between 70^0C and 89^0C.The liquid fat is first raised to the required temperature either by steam pipe (large scale) or merely by placing the reaction vessel over a source of heat. Pre-heated caustic is added to the fat with continuous stirring until the reaction is complete. The time taken to produce soap by this method is less than the time involved in the cold process. The perfume and coloring is added at a later stage to prevent loss of these materials by evaporation. The hot soap mix can be cooled in frames. It is then allowed to stay for 24-36 hours to harden.

The process, although not suitable for the production of toilet soaps, can be used to produce laundry and all types of soft and liquid soaps. The process does not permit the removal of waste lye which contains the glycerin produced in the soap making process, and hence the glycerin, which trends to decrease the hardening property of the soap but improves the cosmetic property, is retained in the finished soap. However the method has some advantage, soap filler can easily be incorporated into semi-boiled mixes. Heating provides for more complete saponification, and allows for the correction of inaccurate mixes.

2.3.3 Full-Boiled Process

The process, popularly used by large and some small soap industries is the most important commercial method of soap making. It involves the treatment of fats or oils with an appropriate amount of alkali and in this process glycerin is remove as a by product and thus it ensures good yield of high quality soap. The process produces hard and firm soaps, though it takes a longer time than the semi-boiling process, and is mostly used for the production of laundry soap and soap bases for toilet soaps.

The process consists of four stages

I. Saponification of the oil with alkali
II. Graining out of the soap
III. Boiling on strength (or clear boiling)
IV. Fitting

2.4 Raw Materials for manufacturing of soap

2.4.1 Characteristics and composition of raw material

2.4.1.1 Fats and Oils:

The fat and oil use in soapmaking come from plant or animal sources. The quality of the soap product directly depends on the fatty oil. Each fat or oil is made up of a distinctive mixture of several different triglycerides. There are many types of triglycerides; each type consists of its own particular combination of fatty acid.

Fatty acids are the components of fats and oils that are used in making soap. They are weak acids composed of two parts;

A carboxylic acid group consists of one hydrogen (H) atom, two oxygen (O) atoms, and one carbon(C) atom, plus a hydrocarbon chain attached to the carboxylic acid group. Generally, it is made up of a long straight chain of carbon(C) atoms each carrying two hydrogen (H) atoms.

General formula for saturated fatty acid is $C_nH_{2n}O_2$. Other unsaturated fatty acids or oils may be shown by the formula $C_nH_{2n-2}O_2$; $C_nH_{2n-4}O_2$ and $C_nH_{2n-2}O_3$. (http://www.chemistry.nus.edu.sg/2500/soap2.htm)

Table 2.1 Fatty acids that are commonly used in soap making

Acid Name	Formula	Double Bonds	Sources
Caproic Acid	$C_6H_{12}O_2$	0	Butterfat
Caprylic Acid	$C_8H_{16}O_2$	0	Coconut oil
Capric Acid	$C_{10}H_{20}O_2$	0	Coconut oil
Lauric Acid	$C_{12}H_{24}O_2$	0	Coconut oil
Myristic Acid	$C_{14}H_{28}O_2$	0	Palm Kernel oil
Palmitic Acid	$C_{16}H_{32}O_2$	0	Palm oil
Stearic Acid	$C_{18}H_{36}O_2$	0	Animal fat
Oleic Acid	$C_{18}H_{34}O_2$	1	Olive oil
Ricinoleic Acid	$C_{18}H_{34}O_3$	1	Castor oil
Linoleic Acid	$C_{16}H_{28}O_2$	2	Grape seed oil
Arachidic Acid	$C_{20}H_{40}O_2$	0	Peanut oil, Fish oil

2.4.1.1.1 Vegetable oils and fats

2.4.1.1.1.1Coconut oil

This oil obtained by boiling or pressing the ground oil crushed kernels of the nuts of cocos nucifera.

Table 2.2 Fatty acid Composition and percentage of Coconut oil

Acid Name	Composition	Percentage %
Caproic acid	C 6:0	0.4 - 0.6
Caprylic acid	C 8:0	4.6 - 10
Capric acid	C 10:0	5.0 - 8.0
Lauric acid	C 12:0	45.1 - 53.2
Myristic acid	C 14:0	16.8 - 21.0
Palmitic acid	C 16:0	7.5 - 10
Stearic acid	C 18:0	2.0 - 4.0
Oleic acid	C 18:1	5.0 - 10.0
Linoleic acid	C 18:2	1.0 - 2.5
Other	C 18:3 C 24:1	< 0.5

Coconut oil has the highest saponification number of all the fats hitherto examined. (S.B srivastava, Soap Detergent and Perfume industry),

It requires strong caustic lyes and forms soaps which can only be separated by concentrated brine, and then become so hard that they cannot be cut .For this reason a clear boiling to be solid would in case of coconut oil soap be entirely contrary to the end in view and very difficult (S.B srivastava, Soap Detergent and Perfume industry).

The relative density	-	(40°C/ water at 20°C) 0.908-0.921
Refractive index	-	(n_o40°C) 1.448-1.450
The saponification value (mg KOH /g oil)	-	248-265
Iodine value	-	6.3-10.6
Unsaponifiable matter	-	not more than 15g/Kg

Table 2.3 Quality characteristics of Coconut oil

Characteristics	Requirements
Clarity	A fully transparent oil at or above 30°C
Color (Platinum-cobalt scale)	50 maximum
Odour and Taste	Mild fresh coconut (free from smoky, soapy, rancid or foreign notes)
Free fatty acids, as lauric acid	0.3% maximum on packing for export
Moisture and volatile matter	0.1% maximum

2.4.1.1.1.2 Cotton seed oil

The oil is obtained from the seeds of the cotton plant. Cotton seed oils are now used for the manufacture of hard and soft soaps. Refined cotton oil is clear, bright oil of pale yellow color. It is usually free from rancidity of free acid. The soap made from this oil is comparatively easily soluble in water, and therefore lathers freely. It is characterized by the high melting point of the fatty acids obtained from it upon the saponification of the oil. The crude cotton oil has a specific gravity of from 0.928-0.930. (SIRI Board of consultants and Engineers small industry research institute, Small scale manufacture of soaps and Detergents)

Table 2.4 percentage of fatty acid of Cotton seed oil

Acid Name	Percentage %
Palmitic acid	22-26
Oleic acid	15-20
Linoleic acid	49-58
Mixture of Arachidic acid, Behenic acid and Lignoceric acid	10
Sterculic acids and Mavalic acids in the crude	1

2.4.1.1.1.3 Linseed oil

The odour and taste of linseed oil are perculiar and characteristic, not to be mistaken for any other oil. It is soluble in about forty times its own volume of alcohol at the ordinary temperature. Linseed oil is easily saponified by boiling with either caustic potash or caustic soda. Linseed oil is the basis from which nearly all the soft soaps are made and for this purpose no better oil can be used. A linseed oil soft soap is of a good bright appearance pleasing in color and free from any objectionable odour. The specific gravity of linseed oil varies some what, that average being 0.935 at 60oF.
(SIRI Board of consultants and Engineers small industry research institute,
Small scale manufacture of soaps and Detergents)

2.4.1.1.1.4 Castor oil

The oil is obtained from the seeds of the castor oil plant. This seed contains 45-55% of oil. The pure castor oil is heavy, colorless oil, possessing a slight odour and a strong nauseating taste. The oil is easily saponified with caustic soda, and yield a very soluble soap. Castor oil soap is very clear and transparent, and is therefore used in the preparation of transparent toilet soap. The specific gravity of castor oil ranges from 0.960-0.970.Viscorsity of castor oil is greater than all the fluid oils employed in soap making. (http://en.wikipedia.org/wiki/castor_oil)

Table 2.5 Average percentage range of fatty acid of castor oil

Acid Name	Average percentage range %
Ricinoleic acid	85 to 95
Oleic acid	6 to 2
Linoleic acid	5 to 1
Linolenic acid	1 to 0.5
Stearic acid	1 to 0.5
Palmitic acid	1 to 0.5
Dihydroxystearic acid	0.5 to 0.3
Others	0.5 to 0.2

2.4.1.1.1.5 Palm oil

Palm oil is obtained from the freshly outer coating of the fruit of several species of palms. The crude palm oil called "peel oil" is use in the soapery along with bone fat and other from of dark grease, for the manufacture of brown soap, but the bulk of it is bleached and employed as a substitute of tallow in making ordinary pale and mottled soaps. Palm oil has an unpleasant, sweetish odour and a neutral taste. It has an orange to chocolate – brown color which is highly characteristics, but is liable to vary considerably. When saponified, palm oil yields from 94 to 97% of fatty acid.
(S.B srivastava, Soap Detergent and Perfume industry).

Table 2.6 Percentage of fatty acid of Palm oil

Acid Name	Percentage %
Myristic	1.2 – 2.4
Palmitic	41.0 – 43.0
Stearic	4.4 – 6.3
Oleic	38.0 – 40.2
Linoleic	9.9 – 11.2

2.4.1.1.1.6 Palm-Kernel oil

This oil is obtained from the kernels of the palm fruits. The percentage of oil varies from 35 - 50 percent. The melting point of fresh palm- kernel oil is at from 77-79 °F.That of old rancid oil being somewhat higher. Its saponification number is about 247.6 and that of the separated fatty acids 265.8.The iodine number of the palm kernel oil ranges from 13.4 -13.6 and that of the separated fatty acid 12.07.
(S.B srivastava, Soap Detergent and Perfume industry).

Table 2.7 Percentage of fatty acid of Palm- Kernel oil

Acid Name	Percentage %
Caprylic	3.0-4.0
Capric	3.0-7.0
Lauric	46.0 – 52.0
Myristic	14.0-17.0
Palmitic	6.0-9.0
Stearic	1.0-2.5
Oleic	13.0-19.0
Linoleic	0.5-2.0

2.4.1.1.1.7 Sunflower oil

Sunflower oil is the non-volatile oil expressed from sunflower seed. Sunflower oil is liquid at room temperature. It is a yellowish, but nearly colorless, limpid oil, possessing little odour and taste. Its specific gravity is usually about 0.925 at 15°C.The density of sunflower oil is 917Kg/m3 at25°C and refractive index ~1.473 at 25°C.(http://en.wikipedia.org/wiki/sunflower_oil)

Table 2.8 Percentage of fatty acid of Sunflower oil

Acid Name	Percentage %
Linoleic acid	48-74 in triglyceride form
Palmitic acid	4-9
Stearic acid	1-7
Oleic acid	14-40

2.4.1.1.1.8 Rapeseed oil

This is a yellowish or greenish- brown oil, possessing in the crude state, a perculiar strong odour and taste and is expressed from seeds of the "Brassica-compestris" and "Brassica-napus". (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.9 Peanut oil or Earth – nut oil

It is obtained from the fruits of "Arachis hypogoea", by drying and pressing. It contains about 45 percent oil by weight. The cold drawn oil is used for eating instead of olive oil, the remaining proteins serving for soap making. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.10 Malabar Tallow

It is obtained from the seeds of an ever green tree known as Vateria Indica.The seeds contain about 25 percent of oil. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.11 Marotli oil

It is obtained from the seeds of the tree known as Hydnocarpus Wightiana.The seeds contain about 60 percent of oil.The soap obtained from this oil is fairly hard with good lathering properties. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.12 Sesame oil

It is obtained by crushing the seeds of sesame orientale; commonly called till-seed. The seeds contain 47-56 percent of oil. Only lastly extracted oil or oil extracted from spoiled seeds is used in the filed of soap manufacture. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.13 Mahua oil

Mahua oil is obtained from the seeds of the three different species of Bassia.The kernels of the seed contain about 50-55 percent oil.The oil is semi-solid at ordinary temperature. It is dirty yellow in color and has a perculiar but not pleasant smell. The soap made out of this oil is easily grained and is hard in appearance. It has a good washing property with creamy lather. When mixed with other oils Mahua give a good texture and hardness to the soap. It saponifies very readily. It is also used for edible purposes. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.1.14 Neem seed oil

It is obtained from the seed of "Melia Azadirachta".The kernels of the seed contain

about 40-45 percent of oil. It has a greenish color and bitter taste with disagreeable odour.It saponifies readily and gives a hard grained soap with good lathering properties. The soap is used as an antiseptic also. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2 Oils and Fats of animal origin

2.4.1.1.2.1 Whale oil or Train oil

This oil, obtained by boiling the blubber of the whale in water and skimming off the oil from the surface is also used for the manufacture of soft soap. It is brown-colored fishy smelling oil, having a specific gravity of about 0.925 and containing a characteristic glyceride of valeric acid $C_5H_{10}O_2$. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2.2 Curriers Grease

A considerable amount of the refuse grease remaining after the treatment of leather with cod oil, tallow etc; in the process of currying is employed in the manufacture of brown and other soaps. It is a softish fat varying in color from pale brown to nearly black and smelling like leather. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2.3 Menhaden oil

This fish oil is now often used in the manufacture of soft soap, in addition to or as a substitute for linseed and cotton oils. Menhaden oil is obtained from Alosa Menhaden and is brown somewhat viscous and fishy –smelling oil having a specific gravity of from 0.927-0.933 at 15°C. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2.4 Tallow

The fat of oxen and sheep is largely employed in conjunction with rosin, in the manufacture of hard yellow or prime-rose soap. It is, when pure a white nearly tasteless solid fat and consists largely of a glycerides of stearic acid, but contains also glycerides of palmitic and oleic acids. The tallow obtained by the soap maker is seldom in a state of purity, being generally of a yellow or brownish color and often slightly rancid. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2.5 Bone oil

Bone oil is obtained from the bones of various animals by treatment with steam under pressure in an iron cylinder, is use for soap making. The fat is usually of a brown

color and often possesses a disagreeable rancid smell. This fat is generally employed for the manufacture of brown soaps and the cheaper kind mottled and also in the fabrication of soap for manufacturers' use soap made from bone grease alone is apt to be deficient in body and firmness. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.1.2.6 Lard

This is the fat of the pig, separated from animal tissue and other impurities by being melted and stained. The amount of both lard and lard oil, used in soap making is however comparatively very small but they are valuable in account of the high class character of the soap made from them. Lard oil soap is very hard, white and in odorous and the fat is mostly employed in the fabrication of the better class toilet soap. (S.B srivastava, Soap Detergent and Perfume industry).

Table 2.9 Soap-making properties of few important oils and fats

Soap made from	Consistency of soap	Lathering Property	Cleaning action
Coconut oil and Palm - kernel oil	Very hard and brittle	Quick forming large unstable bubbles	Very good in both cold and warm water
palm oil	Very hard	Foams rather slowly , small stable bubbles	Very good
Animal Tallow	Very hard	similar to palm oil soap	Good especially in hot water
Greases	Hard	Creamy stable lather	Good
Olive oil	Not very hard	Abundant close greasy stable foam	Good
Cotton seed oil	Somewhat soft	Quick,Aboundant,Greasy stable form	Good
Castor oil	Soft	Very little forming	Fair

2.4.1.2 Alkali

An alkali is a soluble salt of an alkali metal like sodium or potassium.Originally, the alkalis used in soap making were obtained from the ashes of plants, but they are now made commercially. The term alkali describes a substance that chemically is a base and that reacts with and neutralizes an acid. The common alkalis used in soap making are sodium hydroxide (NaOH), also called caustic soda; and potassium hydroxide (KOH),and also called caustic potash.
(http://www.chemistry.nus.edu.sg/2500/soap2.htm)

2.4.1.2.1 Caustic Soda (NaOH):

Caustic soda is a creamy in appearance and fibrous in structure. It readily absorbs moisture and carbonic acid from the air to form sodium carbonate. Due to its hydroscopic property of absorbing moisture, it must not be exposed to the air. Pure sodium hydroxide is a white solid; available in pellets, flakes, granules and as a 50% saturated solution. It is very soluble in water with liberation of heat .commercially; caustic soda is produced by the electrolysis of brine (sea water). (http://en.Wikipedia.Org/sodium hydroxide)

2.4.1.2.2 Caustic Potash (KOH):

It is used in the manufacture of some classes of soft soaps, liquid soaps and as an ingredient of shaving soap, high class textile and phamaceutical soaps. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.3 Other raw materials for soap making

In addition to fats oils and alkalis, a lot of other chemicals are used in comparatively small quantities to give various characteristics to soaps. These chemicals include builders, fillers and various other additives. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.4.1.3.1 Builders

Building agents are essentially used to "build up" i.e. increase the detergent power of soaps. Some alkali builders commonly used in soap making follows;(B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.4.1.3.1.1Sodium Carbonate (Na_2CO_3)

It is added in the form of solution of required density to soap before it is framed for cooling. It is also used as a solid ingredient of soap powders and cleaning compounds.

(S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.3.1.2 Sodium Silicate

For use in soap making, silicate of soda is usually made in to a thin solution of the required density and clutched in the soap which may be fitted cold process or grained one. (S.B srivastava, Soap Detergent and Perfume industry).

2.4.1.3.2 Fillers

Fillers are used to add weight to the soap without in any way adding to the detergent property of the soap. They are not, however, used in good quality genuine soaps. A number of filling agents are used as fillers, but the most popular ones are clay, kaolin, talc, starch, common salt, chalk and magnesium carbonate. Soda ash and sodium silicate are also used in large quantities as fillers. Sodium silicate when used, also acts as an antioxidant to protect the soap from going rancid. It also improves the smoothness, binding, transparency and hardness of the soap. ;(B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.4.1.3.3 Common Salt

Brine (a saturated solution of salt) is very important in soap manufacture. It is needed to grain out the soap in a full boiled process, in order to separate out glycerin and excess caustic from the genuine soap. Salt used in graining has to be pure, i.e. free from compounds of iron (Fe), calcium (Ca) and magnesium (Mg), otherwise they will cause the deterioration of the soap and introduce impurity in the form of their insoluble soaps. ;(B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.4.1.3.4 Colors

Colors are used in soap to give it attractive appearance. In selecting colors care should be taken that they are not acted upon by alkalis which may be present in the free State in the soap. Various mineral colors such as ultramarine, vermilion chrone green, cadmium yellow, caranel etc. are used in drying soaps. The other consideration is that the color should not have any deleterious action on the perfume. The earlier dye stuff used in soap was of water soluble nature but new they are replaced by oil soluble colors. The following are the important colors used in soap manufacture.

Blue	-	Methylene Blue, Ultramarine
Red	-	Crolein Scarlet, Safframine, Cardinal Red "B"

Violet	-	Methyl Violet
Brown	-	Carmel.Umbets, Burnt Sienna
Green	-	Ultramarine Green, Chrone Green, Alizarine
Pink	-	Scarlet Vermilon
Yellow	-	Soap Yellow, Quinolene Yellow, Metanil Yellow, Napthol Yellow, Uramine
White	-	Titanium Dioxide, Zinc Oxide
Scarlet	-	Red and Yellow colors are mixed together

(K.C Dhingra, Hand book of soap detergent and glycerin)

2.4.1.3.5 Perfume

Some oils and fats produce soaps of unpleasant odour, which need to be perfumed. However, in the use of these perfumes, consideration must be given to the action of the particular perfume on the soap. Some synthetic perfume and essential oils cause soap to darken rapidly on keeping, e.g.Clove oil, and Vanillin, while others decompose the soap. Lemon grass oil, Citronella oil, oil of lavender and bergamot oil are suitable oils. Perfumes are added to soaps at low temperature as they are very volatile at high temperature. (S.B srivastava, Soap Detergent and Perfume industry)

2.4.1.3.6 Chemical Bleaching Agent

These are added to washing to carry out washing and bleaching simultaneously. In the recent period the trend is towards the use of fluorescent bleaching or whitening agents. (S.B srivastava, Soap Detergent and Perfume industry)

2.5 The important factors affecting to the quality of raw materials for soap production

2.5.1 Saponification value

This very important constant helps to determine the quantity of caustic soda or potash required to neutralize (saponify) a given quantity of oil or fat chosen for the making of soap.Saponification values are highly significant in the making of soap. It is important that the sap value is just right too high and the soap might contain too much alkali even though there is sufficient soapiness that it would react with skin whilst a sap value too small the fatty acid salts will not be sufficient enough to remove or saponify the fat or oil and less soapiness. Most of fats and oils use of soap making have saponification value ranging from 185 to 205. (Peter Donkor, (1986) Small – scale soap making)

Table 2.10 Saponification values for some common soapmaking oils and fats (http://wiki.answer.com/Q/what_is_thesignificance_of_saponification_value)

Oil/Fat	Saponification Value
Palm Oil	200-205
Coconut Oil	251-264
Palm Kernel Oil	248
Cotton Seed Oil	193-195
Animal Tallow	190-199
Vegetable Tallow	192-195
Lard	195
Neem Oil	186-204
Sunflower Oil	189-194
Castor Oil	181

2.5.2 Iodine Number

The iodine value of an oil or fat is defined as the mass of iodine absorbed by 100g of the sample. The number, in fact, indicates the presence of unsaturated acids in the oil or fat. The iodine number thus indicates the hardness of the soap, the lower the number the harder the soap produced. Cotton seed oil has an iodine number of 109 while Coconut oil has an iodine number of 9. This explains why Coconut oil produces hard soaps. (Peter Donkor, (1986) Small – scale soap making)

Table 2.11 Iodine numbers for some common soapmaking oils and fats

Oil/ Fat	Iodine number
Palm Oil	49-59
Coconut Oil	8-10
Palm Kernel Oil	11-15
Cotton Seed Oil	108-110
Animal Tallow	35-46
Vegetable Tallow	40
Lard	59-63
Neem	69.0
Sunflower Oil	126
Castor Oil	84.1

(Peter Donkor, (1986) Small – scale soap making)

2.5.3 Iodine Number Saponification (I. N. S.) Factor

This factor forecasts about the hardness of soap. Iodine value and Saponification value which are important chemical constants of oil may be related as follows:

I.N. S. Factor = Saponification Value – Iodine Number

Hardness of soap is directly proportional to I.N. S. factor. It is ranges from 15 to 250 for soap making oils and fats. (Peter Donkor, (1986) Small – scale soap making)

Table 2.12 INS factors of common oils and fats used in soapmaking

Oil/ Fat	INS Factor
Coconut Oil	250
Palm Kernel Oil	235
Vegetable tallow	165
Mutton tallow	155
Beef tallow	150
Palm Oil	146
Lard	137
Olive Oil	108
Cotton Seed Oil	85
Linseed Oil	15

(B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.4 Solubility Ratio (S. R.)

It denotes lathering and solubility property of soap. (S.B srivastava, Soap Detergent and Perfume industry)

2.5.5 Moisture

Moisture can be checked by visual examination. If necessary the amount present can be determine by Karl Fischer titration or Dean and Stark distillation. The sample should be well mixed prior to weighing as moisture tends to settle rapidly to the bottom of the container. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.6 Free Fatty Acid – Acid value

The Free Fatty Acid content or acid value of fats and oils is a measure of the free acid present produced by decomposition of the oil or fat. For fatty acids, the acid value, in conjunction with the saponification value, can be use to give a measure of the amount of neutral fat present. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.7 Titre

The titre is the melting point of the fatty acid component of a fat or oil and must not be confused with the melting point of the fat. In general, the melting point of a fat is not a well defined characteristic as the amount of free fatty acid present can vary quite considerably.

The titre gives an indication of the hardness and the solubility in water of the sodium soap made from that particular fat. This is a generalization and is not true in every case although at one time it was considered to be so. Tallow has high titre which gives a hard soap with poor water solubility and low foam. Coconut oil has a low titre and gives a hard soap, easily soluble in water with copious foam. Oleic acid has a low titre and gives a soft, easily soluble soap with low foam. In spite of this, the titre does give an idea of how a particular soap will perform, especially when mixture of fats are being considered. The titre of a mixture of fats cannot be calculated, it must be determined. The fat must be converted to the fatty acids before the titre can be determined. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.8 Color and Bleachability

Color is measured by heating the sample to about 10oC above is melting point, placing it in a colorimeter and reading the value. The color is usually expressed in Lovibond units. If an instrument other than a Lovibond Tintometer is being used, it can be calibrated using either iodine or potassium dichromate solutions. The latter is an alternative manner of expressing color. So ,by using the graph, the color of any fat or oil can be converted to whichever standard is required using any colorimeter or spectrophotometer. There are variations in normal conversions of Lovibond scales but the order of magnitude can still be determined. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.9 Hydroxyl Value

This value is used to characterize castor oil. It is defined as the number of milligrams of potassium hydroxide required to esterify the hydroxyl (-OH) groups.

As the hydroxyl value is inversely related to the molecular weight, the higher the molecular weight, the smaller the hydroxyl value. (B. M. Milwidsky and D. M. Gabriel, (1982) Detergent Analysis, A hand Book for cost-effective Quality control)

2.5.10 Specific Gravity

Specific gravity of the soap is an important characteristic which gives good quality soap. It is determined by the specific gravity bottle or tube. It also measure by hydrometer and by specific gravity balance method. The bottle is carefully filled with the oil to be tested at a temperature of 60°F, taking care to avoid the formation of air bubbles , stopper is carefully insert , the outside nipped clean and dry , then whole weighed, also weight of water at 60°F. (Peter Donkor, (1986) Small – scale soap making)

Table 2.12 Specific Gravity of fatty oils at 60°F

Fat/Oil	Specific Gravity
Tallow	0.912
Lard	0.912
Coconut oil	0.925
Castor oil	0.964
Cotton seed oil	0.923
Linseed oil	0.932
Olive oil	0.915
Palm oil	0.940

(Peter Donkor, (1986) Small – scale soap making)

2.6 Highly considerable conditions

The quality and properties of the soap to be produced depend to a large extent on the type of oil used; hence the choice of suitable oil is very necessary for the production of good soap.

2.6.1 TFM Analysis

TFM says how much fat substance the soap has, therefore TFM is a main quality character to gain a high quality product. The more has better the quality of the soap , anything above 59% of laundry soap.

2.6.1.1 Ether extraction method

When analyze TFM it was extracted in to organic phase that is mainly diethyl ether, here should be careful because of evaporation. Diethyl ether is highly flammable. In ether extraction method should be wearing safety equipments, also careful the boiling ethanol because of somewhat flammable.

When make solutions should be standardize. In this method time is highly consume.

2.6.2 FCA Analysis

FCA value also affecting to the quality of product. The better quality of the soap it should be below 0.1% for laundry soap. While if the soap is grainy or turbid, and somewhat white, it indicates high level of unreacted caustic.

2.6.2.1 Ethanol method

The 5g of soap must be weigh approximately to an accuracy of 0.01g.Before neutralize and titration the temperature should be maintain 70 °C, and careful the end point. The solution must be standardizing. Here also wearing safety equipment.

2.6.3 FFA Analysis

FFA is not highly effect to the quality of the product, but it should less than 1% in sovereign bar soap (company stands).

When boiling ethanol should be careful because of flammable. Before neutralizing and throughout the titration the temperature should be 70 °C.Here also solution must be standardize and careful the end point when titration.

2.7 Equipment for Sovereign Bar soap making

2.7.1 Caustic soda storage tank

This is the tank in which caustic soda solution of the required strength is prepared and stored for use when needed. It is cylindrical shape and made of a strong and thick material to withstand attack from caustic soda. At one time it can be added 500Kg of caustic soda.

2.7.2 Scale tank

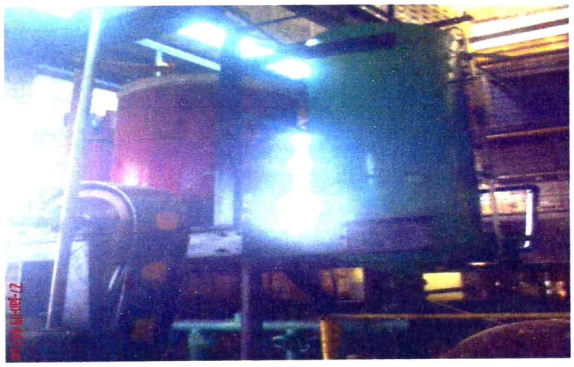


Figure 2.5 Scale tanks

Oil, Caustic soda and Water are storage in the scale tank, requirement quantity.

2.7.3 Soap boiling tank

Figure 2.6 Soap boiling tank

These pans may vary in size and material depending on the maximum volume of soap to be boiled and the process being used, but it made of mid steel or cast iron or galvanized materials of suitable gauge. It has a cylindrical body with truncated conical base (to effect easy drainage of the hot soap).At the bottom of the frame is hole fitted through which the boiled soap is discharged into the soap frame. The total volume of the boiling tank 342 liters and it is capable of boiling half a tonne of soap per batch.

2.7.4 Soap Frame

Figure 2.7 Soap frames

These are metal frames in to which the boiled soap is run for cooling. These boxes are rectangular in shape. The sides of the frames can be made to be taken apart for easy removal of the dried soap or can be permanently nailed. A box designed to give 350 bars of soap.

2.7.5 Slabbing

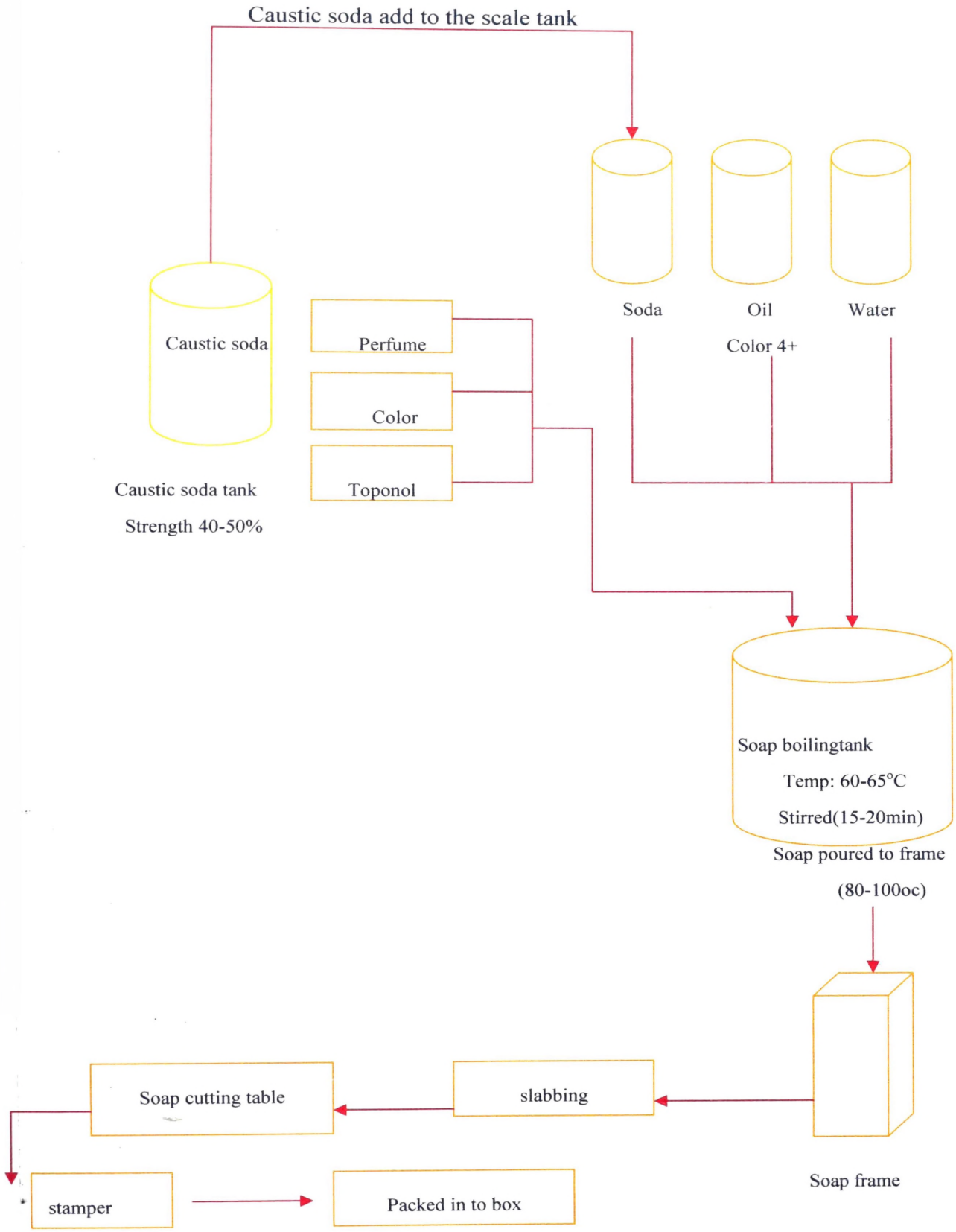
After the soap is hardened and taken from the soap frame, it is in the form of a solid rectangular block, which needs to be slabbed by using slabbing machine.

2.7.6 Soap cutting table

When solid blocks are slabbed and further cut in to bars .This is done using a cutting table.

2.8 Soap Manufacturing Process

Caustic soda is stored in caustic soda tank with 40-50% strength. Then caustic soda, water and fat are stored in scale tanks. First oil is poured to the soap boiling tank. It is heated at 60-65oC temperature. Then caustic soda and water are poured in to the boiling tank with continues stirring until reaction is complete. Then perfume and coloring is added. The hot soap mix poured to the soap frames for cooling. Then it is allowed to stay for 24-36 hours to harden. After hardened it is slabbed by using slabbing machine. Then it is cut in to bars and stamped. Finally it is packed in to boxes.


Caustic soda add to the scale tank
Soda
Oil
Water
Color 4+
Caustic soda
Perfume
Color
Toponol
Caustic soda tank
Strength 40-50%
Soap boilingtank
Temp: 60-65°C
Stirred(15-20min)
Soap poured to frame
(80-100oc)
Soap frame
slabbing
Soap cutting table
stamper
Packed in to box

2.9 The follows condition are maintained during soap manufacturing process

Process	Condition
Caustic soda is stored in caustic soda tank	Strength – 40-50%
Caustic soda, Oil and Water are stored in scale tank	Oil – Color - 4^{+} Iodine value -7.5-9.5 FFA - 1.5
Oil is sent to the boiling tank	Temp: 60-65°C
Add soda + water	Temp:65°C (15-20 min)
After mining add perfume+color+toponol	-
Unloading the mixture to soap frame	80-100 °C
Stay the soap to cooling in frame	24-36 hours
Solid soap block cut into the required sizes	TFM - 59 % FCA - 0.1% FFA – 1%

2.10 The follows are factors that are imported in soap manufacturing process

1) Raw materials such as oils, fats, alkalies and other auxiliary materials which ever be undergoing use must be pure individually.

2) Goggles should be used during the soap preparation because soda lye may harm the eyes.

3) Every constituent should be used in its exact proportion.

4) Soap concentrate should run in to frames only after complete saponification.

5) Besides Mahua oil tallow, Castor oil, Neem oil, Rosin and Palm oil also make the soap concentrate denser and hard. Diluting oils are Coconut oil, Groundnut oil, Cottonseed oil, Mustard oil, Linseed oil and Sesamum oil. The ratio of hard and soft oil should be 60; 40 during use, although it may not be true for the soaps using salt, flour-fine and water.

6) Silicate soap concentrate should fill in the frames only when it is fully saponified and is dense otherwise silicate may be filtered out of the soap.

7) If soap concentrated be harder, it should not be filled in to the frames, but is boiled with additional water so that the concentrate would be homogeneous enough for forming.

8) Coconut oil, gives good and creamy lather to the soap. So one tenth to one fifth part of the oil should be Coconut oil in the manufacturing of the soap.

9) Castor oil yields brightness to the soap so it should be used in the ratio of 1/8 to 1/10 of the total oil content.

10) Al and Zn should not be used as the metal of construction because soap or alkalis easily corrode these metals.

11) Silicates must not be dissolved in either the salt water or washing soda water.

CHAPTER 3
MATERIALS AND METHODOLOGY

3.1 Materials

3.1.1 Materials for determination Total Fatty Matter (TFM)

3.1.1.1 Reagent

- Soap product sample
- Distilled water
- Diethyl Ether (pure)
- Ethanol, 95 %(v/v)
- 0.5M Sulphuric acid solution
- Sodium Chloride Solution, 10g of sodium chloride dissolved in 100ml of distilled water
- Sodium hydroxide, (analytical grade) 0.5M ethanolic solution (recently standardized)
- Methyl orange indicator, 0.2g in 100ml of distilled water
- Phenolphthalein indicator, 1g in 100ml of ethanol

3.1.1.2 Apparatus

- Analytical balance (+/-0.0001g)

3.1.2 Materials for determination Free Caustic Alkali

3.1.2.1 Reagent

- Soap product sample
- Ethanol, absolute
- Potassium hydroxide, 0.1M ethanolic solution
- Hydrochloric acid 0.1M ethanolic solution
- Phenolphthalein indicator, 1g in 100ml of 95% ethanol (v/v)

3.1.2.2 Apparatus

- Analytical balance (+/-0.0001g)

3.1.3 Materials for determination Free Fatty Acid (FFA)

3.1.3.1 Reagent

- Soap product sample
- Ethanol 95% (v/v)
- Potassium hydroxide, 0.1M ethanolic solution
- Phenolphthalein indicator, 0.5 %(w/v) solution in 95% (v/v) ethanol

3.1.3.2 Apparatus

- Analytical balance (+/-0.0001g)

3.2 Method

3.2.1 Method for determination Total Fatty Matter (TFM)

About 5g of soap was weighted and it was dissolved in about 150ml of hot distilled water in a beaker of about 200 ml capacities. This hot aqueous solution was poured in to a separating funnel and the beaker was rinsed with small quantities of hot distilled water. A few drop of methyl orange was added to the solution and the acid solution was added from a burette so that an excess of about 5 ml was added.

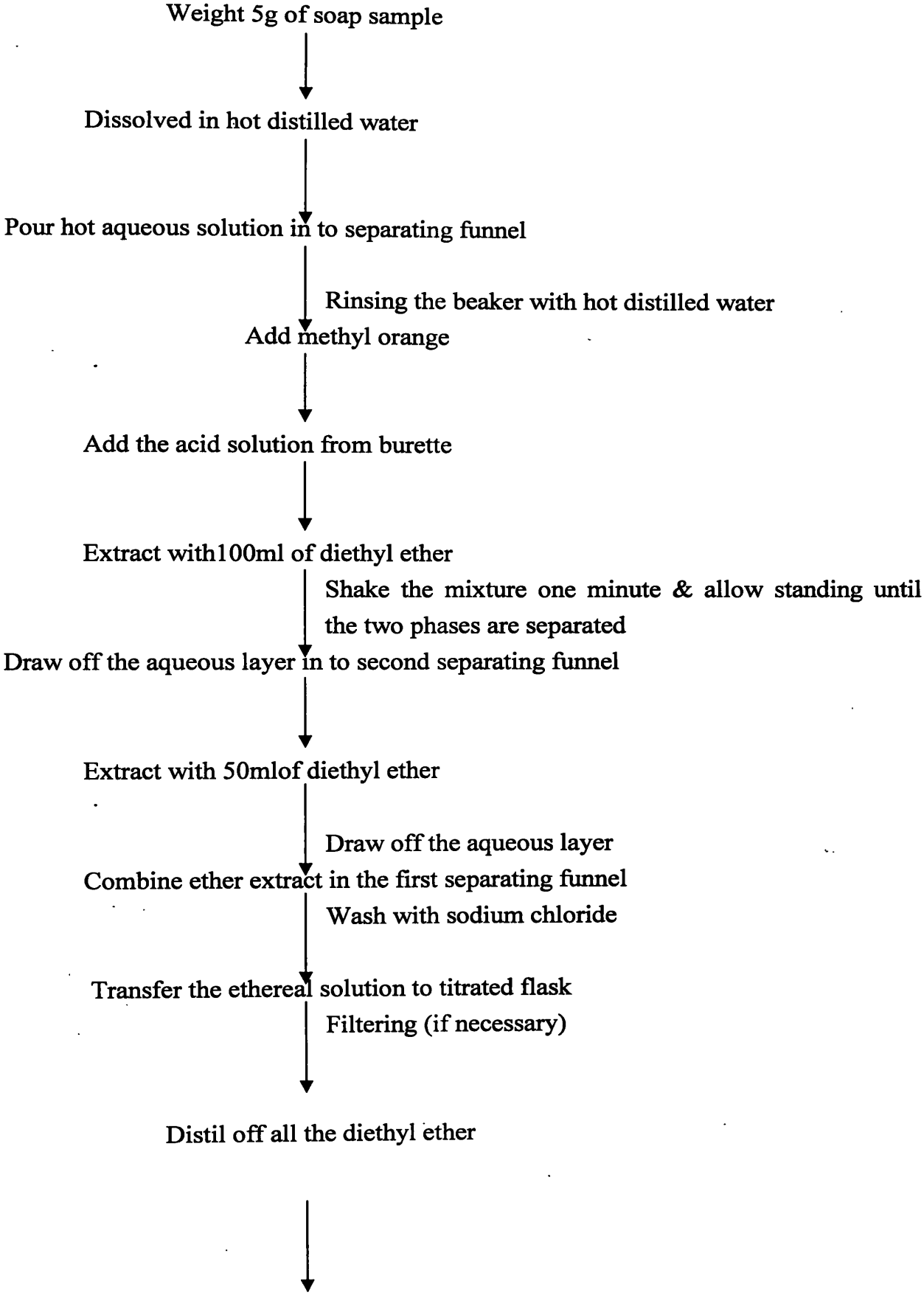
100 ml of diethyl ether was added. The mixture was shaken vigorously for one minute, and allowed to stand until the two phases are completely separated. The aqueous layer was drowned off to a second separating funnel and re- extracted with 50 ml of diethyl ether. The aqueous layer was drowned off. It was combined with ether extract in the first separating funnel. It was washed with 50 ml portions of the Sodium chloride solution, until the washings methyl orange was neutral. Usually three washings were sufficient.

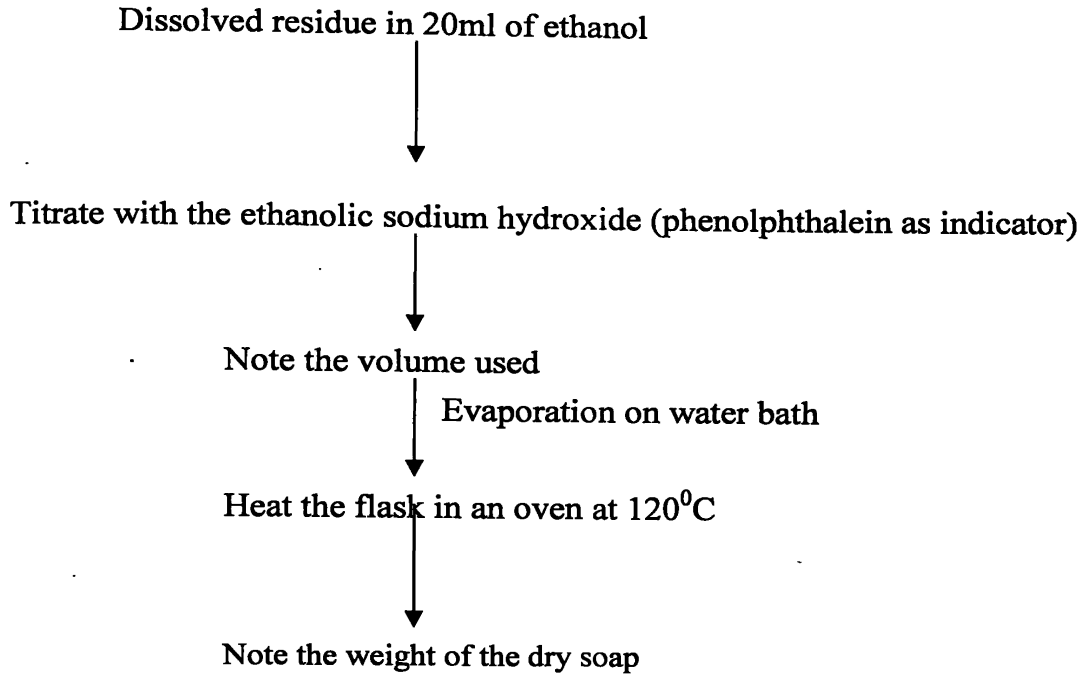
The ethereal solution was transferred to a titrated flask, it was filtered if necessary. The filter was washed with small portion of the diethyl ether. All the diethyl ether was distilled off nearly by boiling gently.

The residue was dissolved in 20 ml of the ethanol. The ethanolic solution of fatty acids were titrated with the ethanolic sodium hydroxide solution, 2-3 drops of phenolphthalein was used as indicator. The volume used was noted.

The ethanol was removed by evaporation on a water bath. The flask was heated in an oven at 120 ^{0}C until the difference in weight, after drying in the oven for an additional 15 minutes did not exceed 5mg.The weight of the dry soap (W2) was noted.

Flow diagram of determination of Total Fatty Matter (TFM)





The equation can be written as follows;

Total fatty matter, per cent by weight, in the soap = [W2-(V*0.5*0.022)] * 100/W1

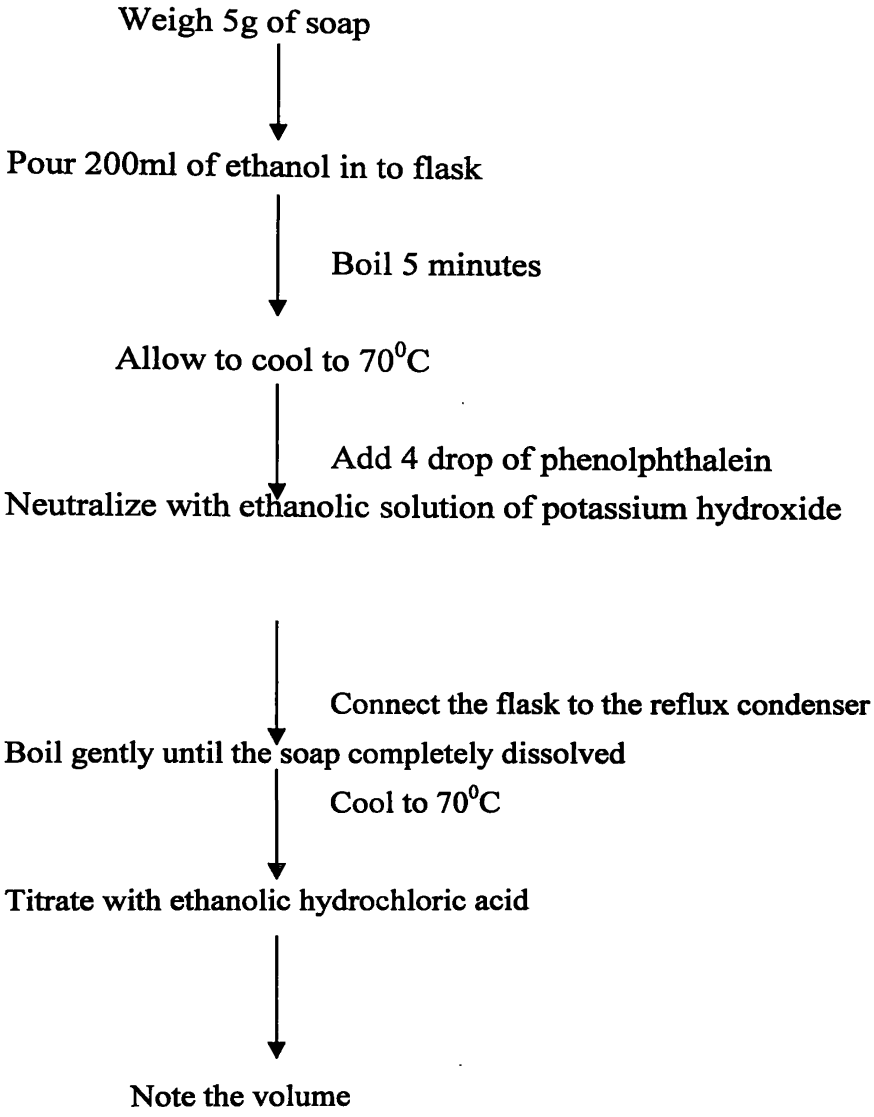
Where W1 = The weight in grammes of the test portion
W2 = The weight in grammes of dry soap
And V = The volume, in milliliters of ethanolic sodium hydroxide solution used.

3.2.2 Method of determination of Free Caustic Alkali (FCA)

Approximately 5g of soap was weighted to an accuracy of 0.01g.200 ml of ethanol was poured in to a flask and it was connected to a reflux condenser. It was gentle boiled for 5 minutes in order to remove carbon dioxide. The solution was allowed to cool to about 70^{0}C. 4 drops of phenolphthalein indicator was added, neutralized exactly with the ethanolic solution of potassium hydroxide, until the indicator was just turned pink.

The test portion was placed in the flask containing the neutralized ethanol. The flask was connected to the reflux condenser and boiled gently until the soap had completely dissolved. It was cooled to about 70^{0}C. The solution was titrated with the ethanolic solution of hydrochloric acid until the color was just perceptibly pink, identical with that obtained when the ethanol was neutralized.

Flow diagram of determination of Free Caustic Alkali (FCA)



The equation can be written as follows

Free Caustic Alkali per cent by weight, expressed as

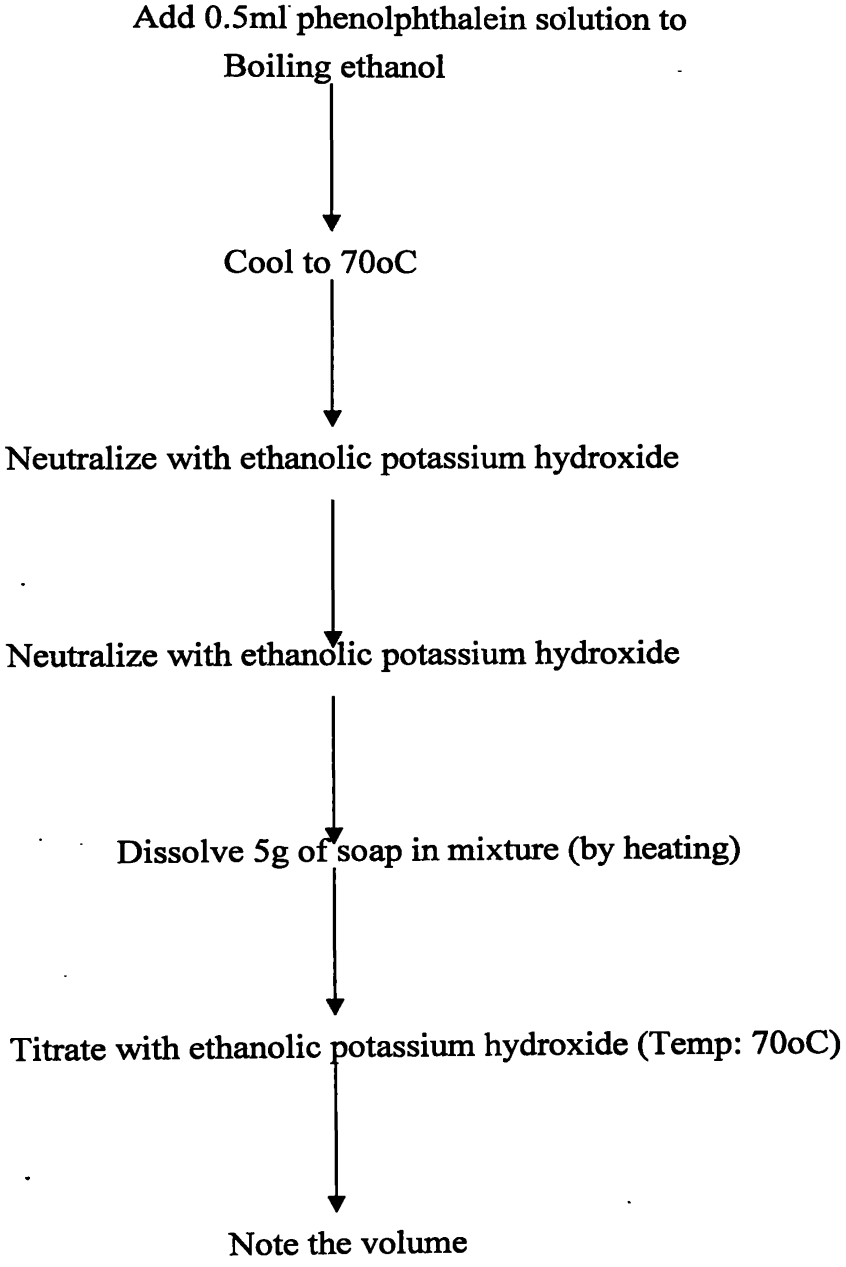
$$Na_2O = 0.31*V/W$$

Where W = The weight in grammes of the test portion
And V = The volume in milliliters of 0.1M ethanolic hydrochloric acid solution used

3.2.3 Method for determination of Free Fatty Acid (FFA)

0.5 ml of the phenolphthalein solution was added to 100ml of boiling ethanol in a suitable beaker, it was allowed to cool to 70°C and neutralized at this temperature with the ethanolic potassium hydroxide solution. 5g of the soap was dissolved in this solution as quickly as possible by heating. If the solution was not alkaline, it was titrated with the ethanolic potassium hydroxide solution until the faint pink color persists for 15 seconds; the temperature was maintained at 70°C throughout the titration.

Flow diagram of determination of Free Fatty Acid (FFA)



The equation can be written as follows

Free Fatty Acid, per cent by weight,

Expressed as lauric acid = 2.00*V/W

Where V = Volume in milliliters, of 0.1M ethanolic potassium hydroxide required
And W = Weight, in grammes, of the test portion taken

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Result

4.1.1 Results of Total Fatty Matter (TFM)

23 soap samples out of 32 had 59 or more than 59.As a percentage is 72% and rest of the sample less than 59, it is 28% of whole.

Table 4.1 Quantity of TFM range of sample

TFM Range	No: of sample
59<	23 out of 32 sample
59>	9 out of 32 sample

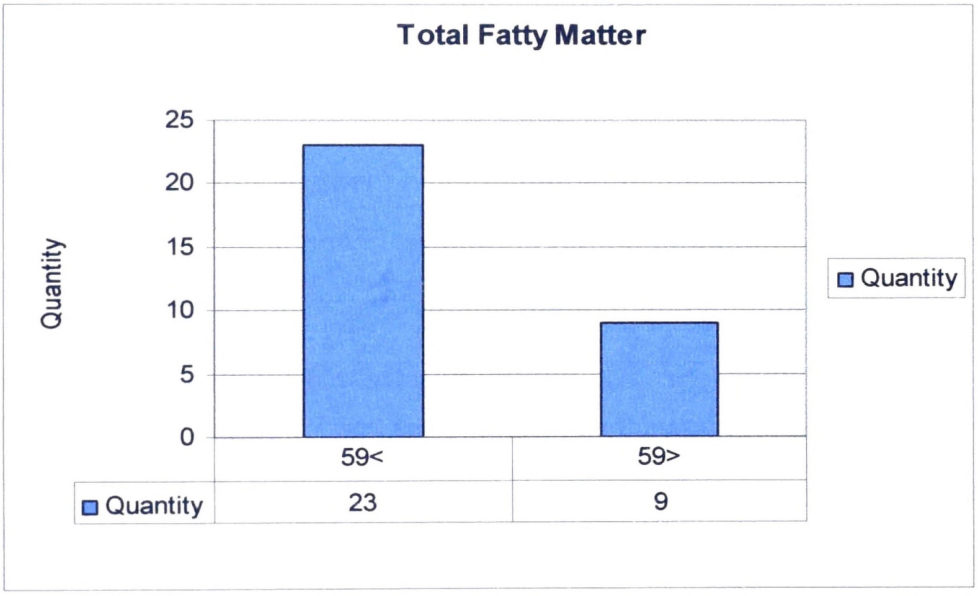


Figure 4.1 Graph of quantity of TFM range in a sample

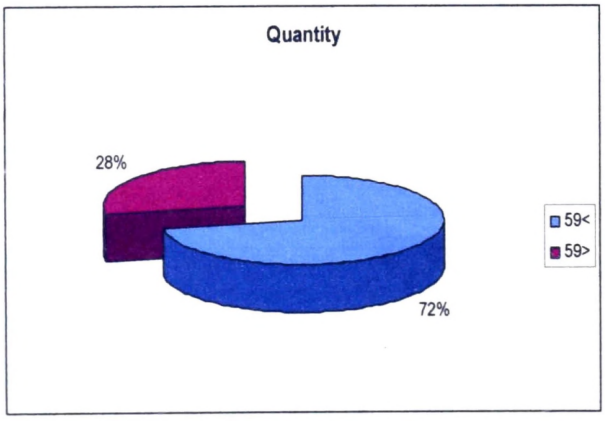


Figure 4.2 Percentage quantity of TFM range in a sample

Statistical analysis was done by using the statistical software (MINITAB 14),

4.1.1.1 Results of TFM statistical analysis,

Wilcoxon Signed Rank Test: TFM

```
Test of median = 59.00 versus median > 59.00

            N
            for    Wilcoxon                 Estimated
      N  Test  Statistic      P     Median
TFM  32    32      327.0  0.121      59.31
```

Wilcoxon Signed Rank CI: TFM

```
                                  Confidence
          Estimated    Achieved    Interval
      N      Median  Confidence  Lower  Upper
TFM  32       59.31        94.9  58.70  59.69
```

4.1.2 Results of Free Caustic Alkali (FCA)

15 soap samples out of 17 had a 0.1 or less than 0.1 as a percentage it is 88% and rest of the sample more than 0.1, it is 12% of whole.

Table 4.2 Quantity of FCA range of sample

FCA Range	No: of sample
0.1<	2 out of 17 sample
0.1>	15 out of 17 sample

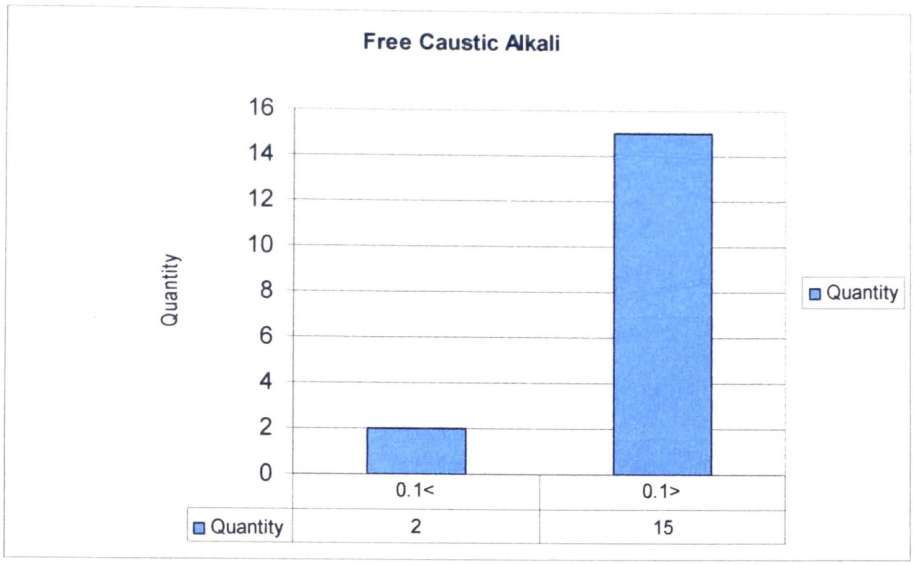


Figure 4.3 Graph of quantity of FCA range in a sample

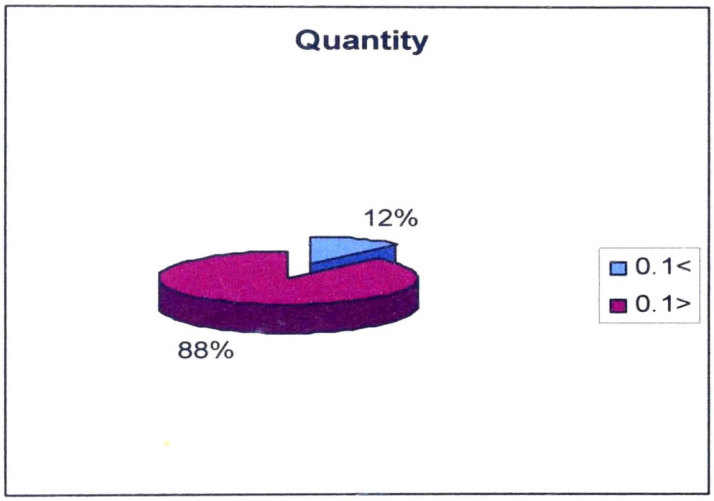


Figure 4.4 Percentage quantity of FCA range in a sample

4.1.2.1 Results of FCA statistical analysis,

Wilcoxon Signed Rank Test: FCA

```
Test of median = 0.1000 versus median < 0.1000

            N
          for   Wilcoxon          Estimated
      N  Test  Statistic      P     Median
FCA  17    17       26.0  0.009    0.04330
```

Wilcoxon Signed Rank CI: FCA

	N	Estimated Median	Achieved Confidence	Confidence Interval Lower	Upper
FCA	17	0.0433	94.8	0.0340	0.0558

4.1.3 **Results of Free Fatty Acid (FFA)**

13 soap samples out of 15 had a 1 or less than 1 as a percentage it is 87% and rest of the sample more than 1, it is 13% of whole.

Table 4.3 Quantity of FFA range of sample

FFA Range	No: of sample
1<	2 out of 15 sample
1>	13out of 15 sample

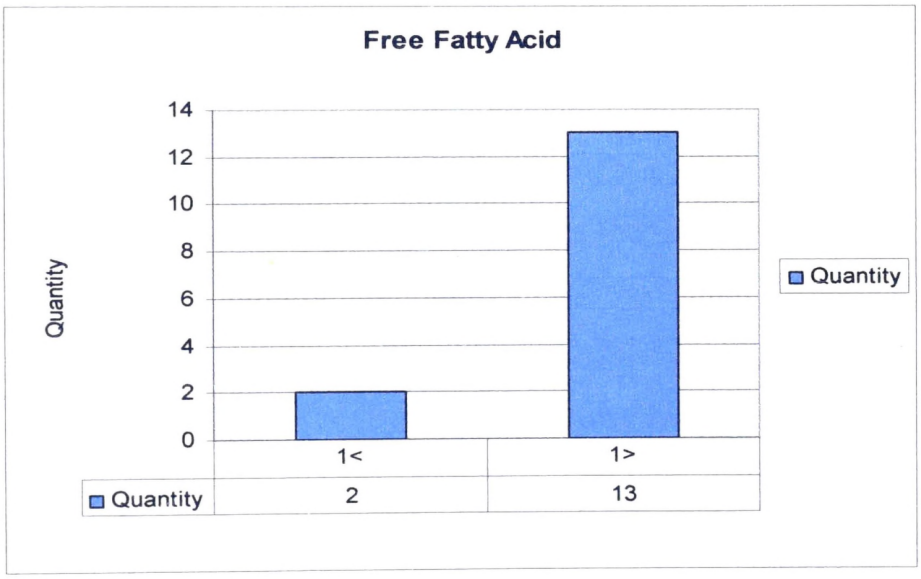


Figure 4.5 Graph of quantity of FFA range in a sample

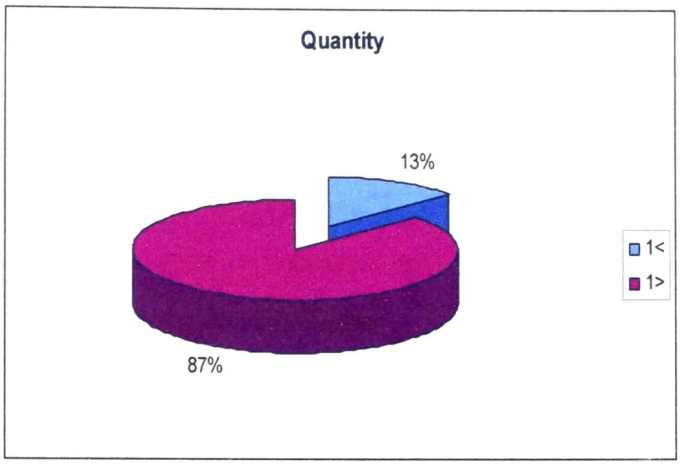


Figure 4.6 Percentage quantity of FFA range in a sample

4.1.3.1Results of FFA statistical analysis,

Wilcoxon Signed Rank Test: FFA

```
Test of median = 1.000 versus median < 1.000

            N
          for    Wilcoxon                Estimated
      N  Test  Statistic      P     Median
FFA  15    15       15.0  0.006     0.5590
```

Wilcoxon Signed Rank CI: FFA

```
                                    Confidence
          Estimated    Achieved     Interval
      N     Median  Confidence  Lower  Upper
FFA  15      0.559        95.0  0.419  0.769
```

4.2 Discussion

According to the observations and calculations, following information and results can be identified,

This research project analyzes TFM, FCA, and FFA of sovereign bar soap .The minimum standard level (SLSI) of TFM is 59% .The results revolved of 23 soap samples out of 32 had 59% or more than 59%. The percentage is 72% .Rest of the samples out of 32 had less than 59% of TFM value; it is a 28% of whole. (Figure 4.1 and Figure 4.2)

As a results of data was not normally distributed (APPENDIX IV), therefore used nonparametric (Wilcoxon Signed Rank Test) analysis test for further details. According to above test the median value of TFM was 59.31%, and the confident interval was 58.70% to 59.69% under 95% confident levels. The lower level of TFM was less than the standard value of SLSI.According to results sovereign bar soap has good TFM value.

According to SLSI standard level the maximum level of FCA should be 0.1% .The 15 soap samples out of 17 had a 0.1% or less than 0.1%. Remaining 2 samples had a more than 0.1% of FCA value (Figure 4.3). Because of not normally distributed (APPENDIX V) of data the wilcoxon sign rank test was carried out for further analysis of data. According to that result estimated medium value was 0.043% and confident interval 0.034% to 0.056% at 95% confident interval. According to results sovereign bar soap has good FCA value.

The company standard level of FFA is 1% as maximum value .The13 soap samples out of 15 had a 1% or less than 1%. Rest of other soap samples (02) had more than 1% of FFA value. As a percentage 1% or less than 1% FFA was 87% (Figure 4.5 and Figure 4.6). The gain data was not normality distributed (APPENDIX VI) so that wilcoxon sign rank test was carried out .The estimated Median level of FFA was 0.565% and confident level was 0.42% to 0.77%. Sovereign bar soap has good FFA value.

The data collected soap samples were not completely homogenious.Because the soap production was carried out as a 12 batches per week. The raw materials were mixed batch wise. I was collected the data within 4 weeks.

CHAPTER 5

5.1 CONCLUSION

To be the good quality soap product the TFM value should be 59% or grater than 59%, the FCA should be less than 0.1%.Those are the SLSI standards for laundry soap, but there is no any SLSI standards value for FFA,but the company standard for FFA is 1%.

According to the results the FCA and FFA values of sovereign bar soap at the proper range that mean less than 0.1% and 1% respectively.

Within some soap samples TFM values were less than its proper quality level, that mean below 59%.As a percentage it was 28% from whole.

So to achieve the high quality soap the TFM,FCA and FFA level should be standardize and try to keep with in specification limit , by the organization.

5.2 SUGGESTIONS FOR FURTHER STUDIES

- Study and make the company quality specification limits for TFM, FCA and FFA with engaging SLSI standard.
- To certify the organization from SLSI certification.

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APPENDIX I

Observation and result for TFM

Sample no:	TFM %	Counter
1	63.82	1
2	59.42	1
3	60.09	1
4	62.67	1
5	57.04	0
6	58.11	0
7	59.78	1
8	59.43	1
9	59.19	1
10	61.17	1
11	60.00	1
12	60.29	1
13	54.23	0
14	59.31	1
15	59.29	1
16	59.23	1
17	58.54	0
18	59.41	1
19	59.38	1
20	59.58	1
21	57.13	0
22	59.2	1
23	59.31	1
24	58.02	0
25	60.03	1
26	53.88	0
27	55.69	0
28	58.53	0

TFM	Quantity
59<	23
59>	9

29	62.85	1
30	59.27	1
31	59.96	1
32	59.19	1

(59 <) -· More than 59

(59 >) - Less than 59

APPENDIX II

Observation and result for FCA

Sample no:	FCA%	Counter
1	0.0123	1
2	0.0433	1
3	0.0557	1
4	0.0247	1
5	0.0434	1
6	0.0309	1
7	0.0433	1
8	0.1671	0
9	0.0433	1
10	0.0433	1
11	0.0557	1
12	0.0434	1
13	0.0124	1
14	0.1795	0
15	0.0247	1
16	0.0558	1
17	0.0372	1

FCA	Quantity
0.1<	2
0.1>	15

(0.1 <) -. More than 0.1

(0.1 >) - Less than 0.1

APPENDIX III

Observation and result for FFA

Sample no:	FFA%	Counter
1	0.479	1
2	0.479	1
3	1.759	0
4	0.639	1
5	0.719	1
6	0.399	1
7	0.439	1
8	0.519	1
9	0.359	1
10	0.159	1
11	0.399	1
12	0.838	1
13	0.279	1
14	1.037	0
15	0.818	1

FFA	Quantity
1<	2
1>	13

(1 <) - More than 1

(1 >) - Less than 1

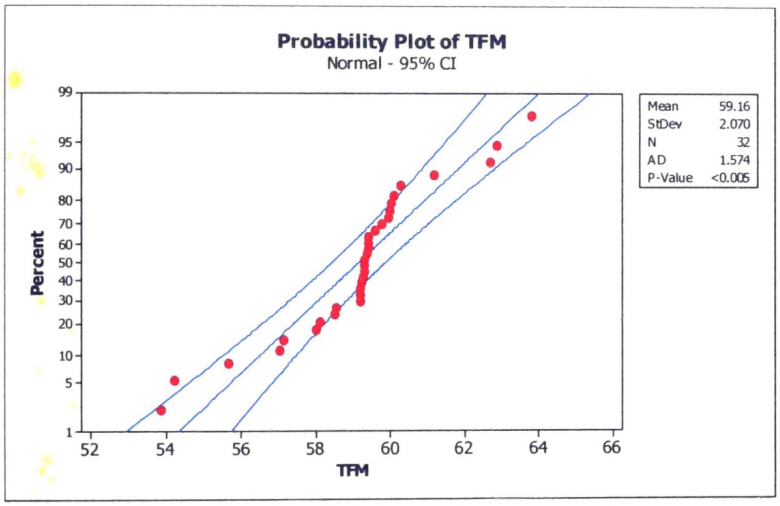
APPENDIX IV

The data was analyzed using MINITAB statistical software for the data obtained on 2/18/2009.The analysis was done at a significance level of 5%.

Descriptive Statistics: TFM

Variable	Mean	StDev	Variance	Median
TFM	59.158	2.070	4.286	59.310

Probability Plot of TFM



P-Value < 0.05

The data was not normality distributed at a significance level of 5%.

There fore used non parametric (wilcoxon signed rank test) analyzed the data.

Wilcoxon Signed Rank Test: TFM

Test of median = 59.00 versus median > 59.00

	N	N for Test	Wilcoxon Statistic	P	Estimated Median
TFM	32	32	327.0	0.121	59.31

Wilcoxon Signed Rank: TFM

	N	Estimated Median	Achieved Confidence	Confidence Interval Lower	Confidence Interval Upper
TFM	32	59.31	94.9	58.70	59.69

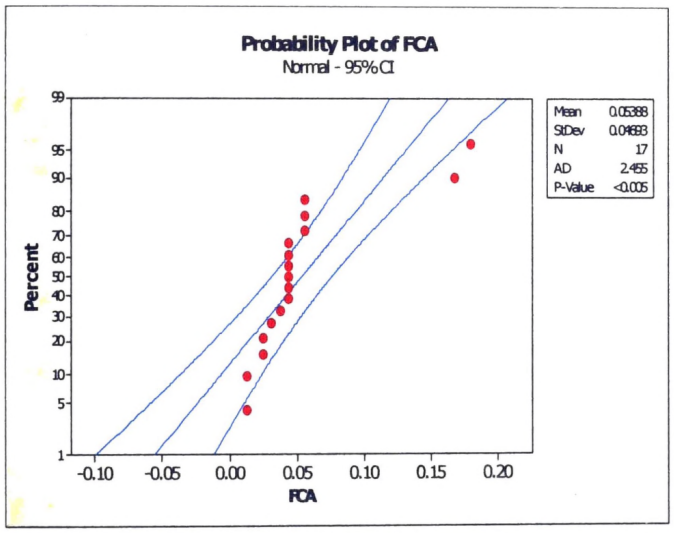
APPENDIX V

The data was analyzed using MINITAB statistical software for the data obtained on 2/18/2009.The analysis was done at a significance level of 5%.

Descriptive Statistics: FCA

Variable	Mean	StDev	Variance	Median
FCA	0.0539	0.0469	0.00220	0.0433

Probability Plot of TFM



P-Value < 0.05

The data was not normality distributed at a significance level of 5%.

There fore used non parametric (wilcoxon signed rank test) analyzed the data.

Wilcoxon Signed Rank Test: FCA

Test of median = 0.1000 versus median < 0.1000

	N	N for Test	Wilcoxon Statistic	P	Estimated Median
FCA	17	17	26.0	0.009	0.04330

Wilcoxon Signed Rank CI: FCA

	N	Estimated Median	Achieved Confidence	Confidence Interval Lower	Upper
FCA	17	0.0433	94.8	0.0340	0.0558

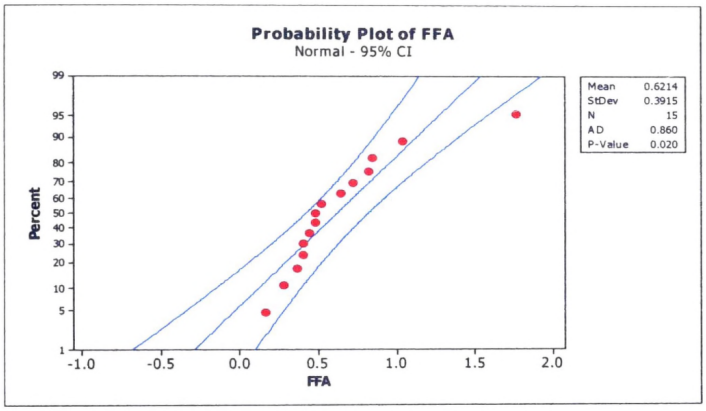
APPENDIX VI

The data was analyzed using MINITAB statistical software for the data obtained on 2/18/2009.The analysis was done at a significance level of 5%.

Descriptive Statistics: FFA

Variable	Mean	StDev	Variance	Median
FFA	0.621	0.391	0.153	0.479

Probability Plot of TFM



P-Value < 0.05

The data was not normality distributed at a significance level of 5%.

There fore used non parametric (wilcoxon signed rank test) analyzed the data.

Wilcoxon Signed Rank Test: FFA

Test of median = 1.000 versus median < 1.000

	N	N for Test	Wilcoxon Statistic	P	Estimated Median
FFA	15	15	15.0	0.006	0.5590

Wilcoxon Signed Rank CI: FFA

	N	Estimated Median	Achieved Confidence	Confidence Interval Lower	Upper
FFA	15	0.559	95.0	0.419	0.769

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