ASSESSMENT OF THE QUALITY OF SOAPS PRODUCED FROM BLENDS OF *Butyrospermum parkii* FRUIT OIL (SHEA BUTTER), *Sesamum indicum* (SESAME) SEED OIL AND TALLOW

BY

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APRIL, 2017

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BY

Muhammad Kabir YAHAYA, B.Sc. (ABU) 2011.
P13SCCH8035

A DISSERTATION SUBMITTED TO THE SCHOOL OF POST GRADUATE STUDIES, AHMADU BELLO UNIVERSITY, ZARIA IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF MASTER OF SCIENCE DEGREE IN ANALYTICAL CHEMISTRY

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APRIL, 2017

DECLARATION

I, Yahaya Muhammad Kabir, hereby declare that this dissertation titled **"**Assessment of the quality of soaps produced from blends of *butyrospermum parkii* fruit oil, *sesamum indicum* seed oil and tallow**"** is my own work and has not been submitted in any other form for another degree or diploma to any other University or Institution. Information derived from published or unpublished work of other researchers has been fully acknowledged in the text.

…………………………………………   …………………………………..

Yahaya Muhammad Kabir   Date

CERTIFICATION

This dissertation titled **"Assessment of the quality of soaps produced from blends of *butyrospermum parkii* fruit oil, *sesamum indicum* seed oil and tallow"** by Yahaya Muhammad Kabir meets the regulations governing the award of the degree of Masters of Science (Analytical Chemistry) of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

…………………………………… …………………

Prof. C. E. GIMBA

(Chairman, Supervisory Committee) Date

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(Dean, Post Graduate School) Date

DEDICATION

To

My parents MR. AND MRS. YAHAYA OHIKERE ISAH

ACKNOWLEDGEMENT

I express my deep sense of gratitude to my humble and able supervisors; Prof C. E. Gimba and Prof. M.S. Sallau (Assistant Dean, Faculty of Science, ABU Zaria) who did not only patiently supervised this work, but also groomed me in the field of Analytical Chemistry.

I am extremely grateful to all academic Staff of Chemistry Department, Ahmadu Bello University, Zaria as well as Mr. Omowanle Joshua for their support and encouragement.

Thanks to my lovely parents and my awesome siblings. Also, thanks to Asuku Abdulsamad M.Sc (ABU), Aliyu Usman Yakubu B.Sc (ABU) and all M.Sc students admitted during 2013/2014 academic session for their support.

ABSTRACT

The study was conducted to determine the characteristics of soap produced from different fats and oils. Three different blends of *Sesamum indicum* seed oil, *Butyrospermum parkii* fruit oil and Tallow were used; nine blends each of *Sesamum indicum* seed oil and *Butyrospermum parkii* fruit oil, *Sesamum indicum* seed oil and Tallow, *Butyrospermum parkii* fruit oil and Tallow. Cold method of soap making was employed for this research. A total of 30 blends were carried out. Each of these blends were used in producing soap as well as individual oil samples. Palm oil was used as control as well as two commercial soaps; Giv and B-29 soap. The soap samples SHS8 (Shea butter 80% - sesame 20%), SHS9 (Shea butter 90% - sesame 10%) SHT9 (Shea butter 90% - Tallow 10%) and G (Shea butter 60% - sesame 20% - Tallow 20%) with total fatty matter (75.60±0.35), (75.10±0.35), (75.80±0.28) and (75.30±0.28) respectively, gave the best soap judging by % Total Fatty Matter (TFM) while blends E (Shea butter 20% - sesame 60% - Tallow 20%) and F (Shea butter 20% - sesame 60% - Tallow 20%) with foam height (cm^3) (91.50±0.71) and (80.00±2.83) respectively gave a better soap in terms of foaming ability. The pH values observed for blended samples at 10% soap solutions were ranged from 10.50±0.14 to 11.20±0.14, while pH values for the commercial soaps used as standard at 10% soap solutions were 10.90±0.14 to 10.95±0.07. Soap sample C (100%Tallow) had the highest pH value of 12.00±0.30 which makes it harsh for the body. Thus, from the results obtained in the present study it can be concluded that due to the favorable physicochemical properties (high %TFM, high pH and high Foam height) of soap samples, some should be recommended for bathing (G, SHS8, SHS9 and SHT9) and some for laundry (D, ST8, ST7, ST9, SHS1, SHS2 and SHS3) judging by % TFM and pH values as well as from the comparison with the commercial soaps used as control.

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NOMENCLATURE

CAS = Chemical Abstract Service number

C_1 = First Control (Giv soap)

C_2 = Second control (B-29)

MF = molecular formula

MW = molecular weight

P = Palm oil

RT = retention time

S= *Sesamum indicum* (sesame) seed oil

SH = *Butyrospermum parkii* (Shea butter) fruit oil

SHS = Blend of Shea butter and Sesame

SHT= Blend of Shea butter oil and Tallow

ST = Blend of sesame oil and Tallow

TFM = Total fatty matter

CHAPTER ONE

1.0: INTRODUCTION

Soap may be defined as a chemical compound or mixture of chemical compounds resulting from the interaction of fatty acids or fatty glycerides with a metal radical or organic base (Kirk-Othman, 1963). Soaps are mainly used as surfactants for washing, bathing, and cleaning, but they are also used in textile spinning and are important components of lubricants. Soaps for cleansing are obtained by treating vegetable or animal oils and fats with a strongly alkaline solution. Fats and oils are composed of triglycerides; three molecules of fatty acids are attached to a single molecule of glycerol (Cavitch and Miller, 1994).

The alkaline solution, which is often called lye (although the term "lye soap" refers almost exclusively to soaps made with sodium hydroxide), brings about a chemical reaction known as saponification. The metals commonly used in soap making are sodium and potassium, which produce water-soluble soaps that are used for laundry and cleaning purposes (Kuntom *et al*., 1994). The qualities of soap are usually determined by the amount and composition of the component fatty acids in the starting oil.

Blends of oils can be used in both the hot and cold soap production methods. Vegetable oil blends could be obtained by mixing different vegetable oils such as the mixture of coconut oil, palm kernel oil, groundnut oil and shea butter in different proportions (Kuntom *et al*., 1996) and soaps of desirable quality can be produced by blending *butyrospermum parkii* fruit oil, tallow and *sesamum indicum* seed oil.

The quality of soap produced is usually comparable to the quality of commercially available soaps. This research work involves using various blends of *Butyrospermum parkii* fruit oil, tallow and *Sesamum indicum* seed oil for the production of various soaps reported.

1.1 **Statements of Research Problem**

There is heavy dependence on palm oil as source of fatty acids for the production of soaps by soap producers. Exploring other type of oils/fats would go a long way in reducing this total dependence on palm oil. Blending various vegetable oils of different qualities could go a long way in the production of quality soaps for laundry, bathing and general cleaning purpose.

1.2 **Aim and Objectives**

The aim of this research work is to study the quality of soap produced from blended oils. The specific objectives of this work are to:

I. Blend oil/fats from *Butyrospermum parkii* fruit oil, tallow and *Sesamum indicum* seed oil;
II. determine the physicochemical parameters of the blended fat/oils;
III. produce soaps from the individual blends (saponification) and
IV. determine the quality parameters of the produced soap.

1.3 **Justification**

Two major sources of oil for soap making are available in nature, which includes vegetable oil (from plants) and Tallow (animal fat). Vegetable oil has a unique quality of being able to form lather water but its short coming is its inability to harden properly. Animal fat on the other hand harden properly but does not readily form lather. Hence the need to blend two or more of these oils with a view to producing a soap which lathers properly in both soft and hard water as well as hardens properly.

1.4 Scope of Research Work

This work covered the procurement of some selected vegetable oils which include Shea butter oil, sesame seed oil and Tallow (animal fat) and other essential soap materials from Samaru Market and Zango in Zaria. These oils were blended together and each blend was used to produce soap. Each soap produced from these various oils were analyzed for their physicochemical parameters. Although other fat/oils exist but this work is only limited to just these three oils.

CHAPTER TWO

2.0 LITERATURE REVIEW

Soaps for cleansing are obtained by treating vegetable or animal oils and fats with a strongly alkaline solution. It consist of sodium or potassium salts of fatty acids and is obtained by reacting common oils or fats with a strong alkaline solution in a process known as saponification. Soap belongs to the family of detergents which is a substance which improves the cleaning properties of water. In saponification, the fats are first hydrolyzed into free fatty acids, which then combine with the alkali to form crude soap. Glycerol (glycerine) is liberated and is either left in or washed out and recovered as a useful byproduct, depending on the process employed (Cavitch and Miller, 1994).

Fats and oils are composed of triglycerides; three molecules of fatty acids are attached to a single molecule of glycerol (Kirk-Othman, 1963). The fatty acids; stearic acid, palmitic acid, myristic acid, lauric acid and oleic acid contribute to lathering and washing properties of the soaps (Ainie *et al*., 1996). Fats are solid esters of fatty acids and glycerin while oils are the liquid glycerol esters of fatty acids at room temperature. During saponification reaction of glycerides with alkalis, NaOH is used for solid or bar soaps and KOH is used for soft soaps (Shoge, 2011). These are characteristically different from soaps made from divalent metals such as calcium, magnesium, iron or aluminum which are not water soluble (Chalmers and Bathe, 1978). Coconut oil is a source of lauric acid (12 carbons) which can be made into sodium laurate. This soap is very soluble and will lather easily even in sea water. Fatty acids with only 10 or fewer carbons are not used in soaps because they irritate the skin and have objectionable odours (Chalmers and Bathe, 1978). For centuries, soap was the only cleansing substance available. Shortage of some raw materials makes soap to be highly priced and only wealthy people could afford it (Viorica *et al*., 2011). It became affordable to other people only after the manufacture of sodium carbonate came in handy during the manufacture of soap (Viorica *et al*., 2011). At the end of the 19th century, the first soap powder for laundry was made using sodium silicate as a binder (Viorica, 2011). Though the use of sodium or potassium carbonate leads to a hard or soft soap respectively, the chemical nature of the lipophilic part of soap plays the largest role in determining the performance of finished soap (Viorica *et al*., 2011).

2.1 **Saponification**

Saponification is the chemical process of making soap that involves an exothermic reaction between sodium hydroxide (lye) and a fat (usually oils). A simple saponification reaction is as shown in Figure 2.1:

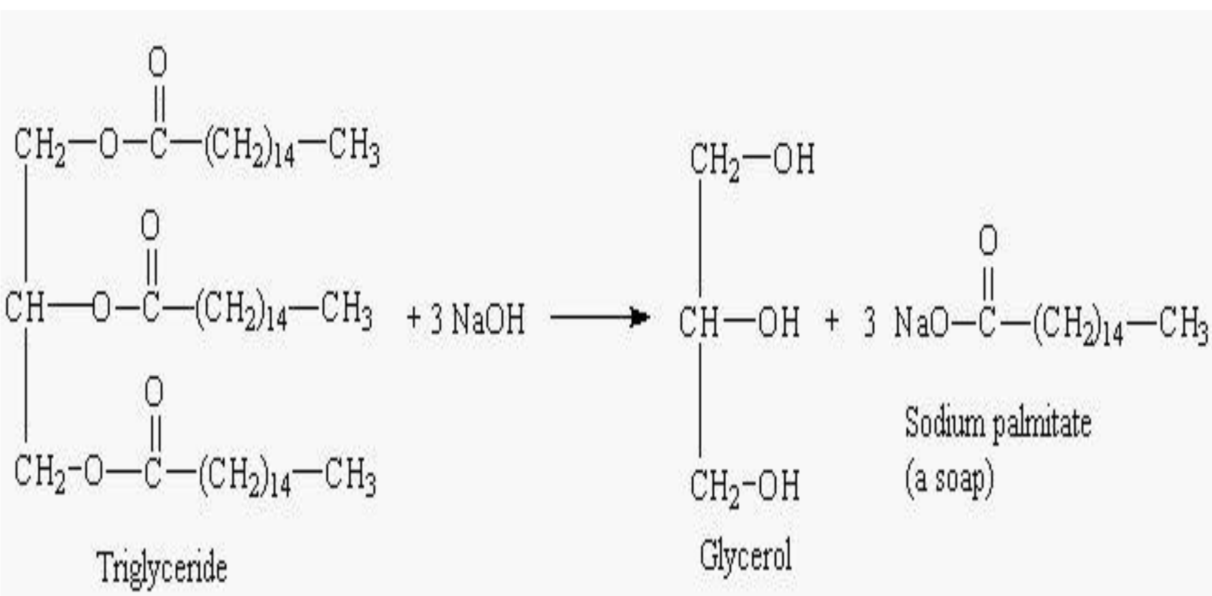

Figure 2.1: Saponification reaction

2.2 Raw Materials Used in Soap-making

Some of the raw materials used in soap making are:

2.2.1 Fats and oils

Panduranga *et al* (1971) reported that soap making oils and fats are found in animal and vegetable fats and oil (such as beef tallow, grease, coconut oil, palm oil, palm kernel oil, linseed oil, rubber seed oil, soybean oil, and corn oil), among others. These fats and oils contain various properties of the fatty acid usually having 6-22 carbon atoms in the paraffin chain. These include caprylic acid ($C_8H_{16}O_3$) lauric acid ($C_{14}H_{32}O_2$), stearic acid ($C_{18}H_{36}O_2$), and linoleic acid ($C_{18}H_{32}O_2$) among others.

All animal fats and vegetable oils intended for soap-making should be as free as possible from unsaponifiable matter, of a good colour and appearance, and in a sweet, fresh condition (Simmons and Appleton, 2007). Fats and oils used to make soap come from animal or vegetable sources. Oil derived from another sources such as mineral oil, cannot be used. Soap can be made by using only one kind of fat or oil, by blending animal and vegetable oil or blending more than one vegetable oil. Other oils that can be used are olive, cottonseed, maize, soybean, groundnut, safflower, sesame and linseed. Coconut and palm oils are very good for soap making. The animal or hard fats are generally used to make soap are tallow and lard. Tallow is the fat from beef or lamb. Lard is the fat from hogs. Butterfat from cow milk is acceptable. However, chicken fat is not a hard fat, and is regarded as oil (Francioni and Callings, 2002).

Some other vegetable oils are very rich in minerals and Vitamins that are good for the skin. Vegetable oils such as Almond Oil, Aloe Vera Butter, Aloe Vera Oil, Apricot Kernel Oil, Avocado Butter, Avocado Oil, Babassu Nut Oil, Beeswax Animal, Borage Oil, Candelilla Wax, Canola Oil, Castor Bean Oil, Cherry Kernel Oil, Cocoa Butter, Coconut Oil, Jojoba Seed Oil, Neem Tree Oil, Olive Oil, Palm Kernel Oil, Peanut Oil, Pumpkin Seed Oil and Soybean Oil, among several others. These oils, when used in bathing soap nourish the skin, and cure many skin infections and diseases.

2.2.1.1 Olive oil

Olive oil is made from pressing the olive fruit. This oil is an excellent moisturizer because it attracts external moisture, holds the moisture close to the skin, and forms a breathable film to prevent loss of internal moisture. It is also known for healing properties (Kenam and Melek, 2014).

2.2.1.2 Palm oil

Palm oil is known for moisturizing with a creamy rich lather. It is so important to use sustainable palm oil as it saves the environment (Kenam and Melek, 2014).

2.2.1.3 Coconut oil

Coconut oil is considered a gift. Its discovery has contributed to higher grade soaps in addition to its rich, moisturizing lather. If used in too high of portions it can be dried. Many of these natural oils have replaced lard in high quality soaps (Kenam and Melek, 2014).

2.2.1.4 Jojoba

Jojoba is a perennial woody shrub grown primarily in the desert regions of the southwestern United States and Northwestern Mexico. Native Americans have long used jojoba to help heal sores and wounds. Today, jojoba oil is still most commonly used for cosmetic purposes, particularly for the maintenance of healthy skin. Jojoba oil helps promote healing of the skin in many ways. It has antimicrobial properties, which means it actually discourages the growth of some bacterial and fungal microbes that attack the skin. In addition, the chemical composition of jojoba closely resembles that of the skins natural sebum, so it is easily absorbed and rarely causes allergic reactions, even in the most sensitive individuals (Kenam and Melek, 2014).

- Acid value is the most frequently determined property of Natural Jojoba. Total acid value of jojoba is typically less than 1.0 which can be reduced with refining methods.
- Peroxide value is used as a measure of its relative oxidation state at a single point in time. Typically, jojoba's peroxide value at time of packaging is less than 5.0.
- Iodine value is a measure of unsaturation (number of double bonds). Natural Jojoba is almost exclusively composed of "diene" making its Iodine Value similar to that of oleic acid. Iodine values are typically in the range between 70 and 90.
- Saponification value also is constant and ranges from 85- 100

2.2.1.5 Shea butter

This is a byproduct of nuts obtained from shea tree also known as *Vitellaria Paradoxa* (Maranza *et al*., 2004*)* or *Butyrospermum parkii* which belongs to the *Sapotaceae* family. It constitutes an important source of fat in food and cosmetics (Okollu *et al*., 2010) Its fatty matter has been used for years in Africa for different purposes, ranging from food and soap processing, to healthcare and other medicinal uses (Coulibaly *et al*., 2010) Global interest in the product stems from its use as a cocoa butter equivalent in the pharmaceutical and cosmetics industries (Elias *et al*., 2006) It is also used as an unguent for the skin and has anti-microbial and soothing properties, which give it a place in herbal medicine. It is also used as an important raw material and/or a precursor for the manufacture of candles (Adomako, 1985). The fat obtained from the shea kernel is referred to as shea butter and it is the most valued product from the shea tree (Hall *et al*., 1996).

Shea Butter is from the nut of the Shea Tree, also known as “The Tree of Life”. The Shea tree (locally known as ori in Yoruba Language) only grows in the savannah region of Africa. It grows up to 60 feet and can live up to 200 years. The Shea Tree produces its fruits once a year. The nut in the center of the fruit when crushed and traditionally processed by boiling and extracting the oil, is what yields the vegetable fat known as Shea Butter. Shea butter contains vitamins A, E, and F, and fatty acids, which provide healing abilities superior to cocoa butter. This natural butter has proven cell regenerative, moisturizing, and anti-aging properties, which restore shine and beauty to skin and hair. Effective in preventing stretch marks, wrinkles, inflammation, eczema, and dryness from razor irritation. Shea butter has been used for centuries in Africa for cooking, and also as skin treatment for its amazing ability to renew, repair and protect the skin against harsh climates. African healers and beauties like Cleopatra have known about shear butter for thousands of years. Shea butter is also called “women’s gold”, because extracting the butter from the nuts gives income to hundreds of thousands of rural African women.

2.2.1.6 Mango butter

Mango butter is extracted from the shelled fruit kernel of the mango tree, which is a tropical evergreen plant. This butter is highly emollient, softening and soothing to the skin. Mango butter has protective effects against UV radiation and also helps treat skin rash, eczema, insect bites, and poison ivy. Mango butter can help protect and heal skin from the damage caused by sunburn and frostbite. While mango butter is excellent for skin, it is often mixed with other ingredients because it is much harder than Shea butter (Kenam and Melek, 2014).

2.2.1.7 Aloe vera butter

Aloe Vera Butter is a combination of coconut oil and aloe vera. It is a soft, white butter with a low melting point. Aloe Vera Butter has similar qualities to Aloe Vera Gel in that it soothes and helps heal dehydrated skin conditions such as eczema, psoriasis, sunburn and windburn. It can be added to moisturizing soaps, creams, lotions, lip balms and body butters, and can be used on its own as a body balm or after-sun treatment (Kenam and Melek, 2014).

2.2.1.8 Cocoa butter

Cocoa butter is obtained from the same bean as chocolate and cocoa. Cocoa butter lays down protective layer that holds moisture to the skin, making it a good skin softener (Kenam and Melek, 2014).

2.2.1.9 Calendula flower

Calendula flower used in creams and washes are used to disinfect minor wounds and to treat infections of the skin. The antibacterial and immune stimulant properties of the plant make it extremely useful in treating slow-healing cuts and cuts in people who have compromised immune systems. The herb stimulates the production of collagen at wound sites and minimizes scarring. Gargling calendula water may ease sore throat (Kenam and Melek, 2014).

2.2.1.10 Castor oil

Castor oil is derived from the bean produced from the castor plant. Like olive oil and jojoba oil, castor oil acts as humectants by attracting and retaining moisture to the skin (Kenam and Melek, 2014).

2.2.1.11 Sweet almond oil

Sweet almond oil is known for its many skin benefits. While it is used for all skin types, sweet almond oil is particularly effective for dry skin that is prone to itching and irritation. The oil is a nice choice for the natural oil cleansing method because it is light, moisturizing and does not leave a greasy residue on the skin once it absorbs.

2.2.1.12 Sunflower oil

Sunflower Oil is very high in essential fatty acids. It helps to moisturize, regenerate and condition the skin. Sunflower oil is good oil for mature, sensitive, dry or damaged skin. Non oily feel in lotions, saves, and lip balms. Excellent moisturizer and massage/baby oil. (DMBB, 2013)

2.2.2 Alkali

Alkalis are basic (high pH) substances that cause the desired chemical reaction. Common alkalis used are caustic soda (sodium hydroxide) and potash (potassium hydroxide). The alkalis use in soap making can be from two sources:

I. Lye, caustic soda, or potash, purchased at the market: or
II. Lye obtained by leaching or washing water through the ashes of plants.

2.2.3 Other soap ingredients

Borax, soda, ammonia, kerosene, naptha, and rosin are sometimes added to increase the quantity of suds and improve the appearance of the soap, however they may not be necessary. Perfumes are added to make soap smell more pleasant. Either essential oils or artificial perfumes can be used. Some essential oils commonly used are lavender, geranium, winter green, citronella, clover lemon, rose, almond, caraway, and banana. The oil or perfume is stirred into the soap just before the soap is poured into molds. The quantity of perfume used depends on the size of the batch of soap being produced, the intensity of the perfuming agent, and the strength of the desired final scent. The amount used can vary from a few drops to 15 grams or more. Coloring matter can also be added. For example, vegetable or aniline dyes can be used to make soap. They are stirred evenly into the soap mixture before pouring it into molds. The color obtained from aniline dyes has a tendency to fade slightly when soap is exposed to bright light (Francioni and Callings, 2002).

2.3 Fatty Acids and Soap Making.

Soap is a chemical substance, created by combining fatty acids, an alkali and heat in a water medium. Fatty acids are chemical components of oils ("oils" includes fats and butters). There are various fatty acid varieties, each differing in molecular composition. When used in soap making, each fatty acid variety will make a soap that has its own unique characteristics. Different oils used in soap making have differing proportions of these various fatty acids. Therefore, chosen "recipe" combinations of specific oils mixed together in soap making can result in combined fatty acids compositions tailored to make soaps with desired characteristics. An understanding of this ability to tailor the oils mix and thereby control the fatty acids mix is essential in designing successful soap recipe formulas.

2.3.1 Some fatty acids and their resulting soap characteristics

Lauric Acid ($C_{12}H_{24}O_2$): Hard bar, excellent cleansing, lots of fluffy lather, can be drying to skin

Linoleic Acid ($C_{18}H_{32}O_2$): Conditioning, silky feel.

Myristic Acid ($C_{14}H_{28}O_2$): Hard bar, cleansing, fluffy lather.

Oleic Acid ($C_{18}H_{24}O_2$): Conditioning, slippery feel, stingy lather, kind to skin

Palmitic Acid ($C_{16}H_{32}O_2$): Hard bar, cleansing, stable lather.

Riconoleic Acid ($C_{18}H_{34}O_3$): Softer bar, conditioning, moisturizing, lots of fluffy, stable lather, kind to skin.

Stearic Acid ($C_{18}H_{36}O_2$): Hard, long lasting bar, stable lather.

2.4 Types of Soap Based on Process, Properties and Compositions

2.4.1 Hard and soft soaps; Often, hardness is achieved through the addition of hardening agents, so many natural soaps tend to be softer. Hard soap can be produced by repeat regrinding and re-forming, known as triple milling. Keeping the moisture content low helps the hardness of the soap, but this is often achieved by accelerated curing in very low humidity conditions. Glycerine is a normally produced during the soap making process. Glycerine is humectants, which means it attracts water. Therefore soaps which contain glycerine in them tend to make the skin feel moister. At the same time glycerine soaps tend to have more water in them and attract water from the atmosphere, therefore 'sweating' in humid conditions (DMBB, 2013).

2.4.2 Transparent soap

This soap uses slightly different components and usually some form of alcohol to alter the process. The process is also conducted at higher temperatures. All these facts (alcohol and higher temperature) change the process and hence the resulting product. Not all transparent soaps are glycerine soaps. Sugar syrup (sugar in distilled water) is an alternative ingredient to replace alcohol but not as effective as alcohol. Some of the soap producer use already made soap base to make their transparent soap.

2.4.3 Liquid soaps

Liquid soaps are actually quite difficult to make and many of the commercial liquid soaps are just in fact detergents. They are in liquid state. Making liquid soap is more complicated than bar soap. Liquid soaps are usually made with the hot process method. It becomes liquid form due to a different type of lye (potassium hydroxide instead of sodium hydroxide) used in the making process and also due to more water added during the soap making process.

2.4.4 Tablet or bar soaps

These are generally solid soaps.

2.5 Types of Soap Based on Usage

Based on the ingredients used by the manufacturer or producer soap can be used for different purposes, which include cleaning, washing to bathing. The types of soap based on usage are:

2.5.1 Antiseptic soaps and bathing soaps

Antiseptic soap, sometimes called antibacterial soap or anti-fungal soap, is regular soap in liquid or solid form that contains some kind of ingredient that reduces the chance of infection when applied to the skin. These products also have antimicrobial properties, meaning they kill or inhibit the growth of microbes like bacteria, virus, or fungi. Alcohol, triclosan, and tetrasodium ethylenediaminetetraacetic acid are three antiseptics commonly used in soap, and they are all examples of antibacterial antiseptics. Other antiseptics have anti-viral properties, and some are anti-fungal and can be used to treat or prevent fungal infections like athlete's foot, ringworm, or vaginal yeast infections. Many antiseptics have a combination of these various properties. Some essential oils, such as tea tree oil, contain naturally occurring antiseptics called terpenes that have antibacterial, anti-fungal, and antiviral properties. These various kinds of antiseptic ingredients are also used in soap (WiseGEEK, 2013). Some scientists and medical professionals are critical of the increasing use of antiseptic soaps and the addition of antibacterial ingredients to so many cleaning products. They believe that this practice might lead to more strains of bacteria becoming resistant to antibacterial agents, eventually causing antibiotic medications to become less effective. This could make it more difficult to treat serious medical conditions caused by bacteria, like staph infections and pneumonia. For example, many microorganisms are already resistant to triclosan, meaning that even though it is a proven antibacterial agent, it may not be as effective as stated by some soap manufacturers because many strains of bacteria are immune to it (WiseGEEK, 2013).

2.6 Soap Making Processes

There is several different soap making processes that allow easy production of soap products. These are briefly described in sections 2.7.1-2.7.4:

2.6.1 Cold process soap making

The cold process uses just enough heat (45-50^{0}C) to ensure that all the fat is melted prior to reacting it with the base. The cold process gets its name from the general low temperatures that are used to mill this type of soap (Anne-Marie, 2003). Even in the cold soap making process, some heat is usually required; the temperature is usually raised to a point sufficient to ensure complete melting of the fat being used. The batch may also be kept warm for some time after mixing to ensure the alkali (hydroxide) is completely used up. This soap is safe to use after about 12–48 hours, but is not at its peak quality for use for several weeks.

Cold-process soapmaking requires exact measurements of lye and fat amounts and computing their ratio, using saponification charts to ensure the finished product does not contain any excess hydroxide or too much free unreacted fat. Saponification charts should also be used in hot processes, but are not necessary for the "fully boiled hot-process" soaping.

Historically, lye used in the cold process was made from scratch using rainwater and ashes. Soapmakers deemed the lye solution ready for use when an egg would float in it. Homemade lye making for this process was unpredictable and therefore eventually led to the discovery of sodium hydroxide by English chemist Sir Humphry Davy in the early 1800s.

A cold-process soap makers first looks up the saponification value for each unique fat on an oil specification sheet. Oil specification sheets contain laboratory test results for each fat, including the precise saponification value of the fat. The saponification value for a specific fat will vary by season and by specimen species (QLOEPA, 2012). This value is used to calculate the exact amount of sodium hydroxide to react with the fat to form soap. The saponification value must be converted into an equivalent sodium hydroxide value for use in cold process soapmaking. Excess unreacted lye in the soap will result in a very high pH and can burn or irritate skin; not enough lye leaves the soap greasy. Most soap makers formulate their recipes with a 2–5% deficit of lye, to account for the unknown deviation of saponification value between their oil batch and laboratory averages.

The lye is dissolved in water. Then, the oils are heated, or melted if they are solid at room temperature. Once the oils are liquefied and the lye is fully dissolved in water, they are combined. This lye-fat mixture is mixed until the two phases (oils and water) are fully emulsified. Emulsification is most easily identified visually when the soap exhibits some level of "trace", which is the thickening of the mixture. Many modern-day amateur soapmakers often use a stick blender to speed up this process. There are varying levels of trace. Depending on how additives will affect trace, they may be added at light trace, medium trace, or heavy trace. After much stirring, the mixture turns to the consistency of a thin pudding. "Trace" corresponds roughly to viscosity. Essential oils and fragrance oils can be added with the initial soaping oils, but solid additives such as botanicals, herbs, oatmeal, or other additives are most commonly added at light trace, just as the mixture starts to thicken (QLOEPA, 2012)

The batch is then poured into molds, kept warm with towels or blankets, and left to continue saponification for 12 to 48 hours. (Milk soaps or other soaps with sugars added are the exception. They typically do not require insulation, as the presence of sugar increases the speed of the reaction and thus the production of heat.) During this time, it is normal for the soap to go through a "gel phase", wherein the opaque soap will turn somewhat transparent for several hours, before once again turning opaque.

After the insulation period, the soap is firm enough to be removed from the mold and cut into bars. At this time, it is safe to use the soap, since saponification is in essence complete. However, cold-process soaps are typically cured and hardened on a drying rack for 2–6 weeks before use. During this cure period, trace amounts of residual lye are consumed by saponification and excess water evaporates.

During the curing process, some molecules in the outer layer of the solid soap react with the carbon dioxide of the air and produce a dusty sheet of sodium carbonate. This reaction is more intense if the mass is exposed to wind or low temperatures.

2.6.2 Hot process soap making

This process is named for its boiling pot method of soap milling. This method uses heat to speed the reaction resulting in fully saponified soap by the time the soap is poured into molds (Anne-Marie, 2002). Hot-processed soaps are created by encouraging the saponification reaction by adding heat to speed up the reaction. In contrast with cold-pour soap which is poured into molds and for the most part only then saponifies, hot-process soaping for the most part saponifies the oils completely and only then is poured into molds.

In the hot process, the hydroxide and the fat are heated and mixed together at 80–100 °C, a little below boiling point, until saponification is complete, which, before modern scientific equipment, the soapmaker determined by taste (the sharp, distinctive taste of the hydroxide disappears after it is saponified) or by eye; the experienced eye can tell when gel stage and full saponification has occurred. Beginners can find this information through research and classes. Tasting soap for readiness is not recommended, as sodium and potassium hydroxides, when not saponified, are highly caustic.

An advantage of the fully boiled hot process in soapmaking is the exact amount of hydroxide required need not be known with great accuracy. They originated when the purity of the alkali hydroxides were unreliable, as these processes can use even naturally found alkalis, such as wood ashes and potash deposits. In the fully boiled process, the mix is actually boiled (100+ °C), and, after saponification has occurred, the "neat soap" is precipitated from the solution by adding common salt, and the excess liquid is drained off. This excess liquid carries away with it much of the impurities and color compounds in the fat, to leave a purer, whiter soap, and with practically all the glycerine removed. The hot, soft soap is then pumped into a mold. The spent hydroxide solution is processed for recovery of glycerine (Garzena and Tadiello, 2013)

2.6.3 Hand milled soap making method

This method is also called soap re-batching in which soap can be made from soap scraps that can still be rework into a new bar of soap. To do this, soap scraps, a mold, a grater, fragrance, dyes, and other additives would be needed. The best type of soap base for this type of soap making is cold process soap (Anne-Marie, 2002).

2.6.4 Melt and pour soap making method

Out of all of the soap making methods this is perhaps the easiest for novice soap makers. It involves simply melting a soap base in a double boiler, adding in fragrance and dyes, and then placing the soap mixture into a mold (Anne-Marie, 2002).

Adding extra oil to cold or hot process soap after it has traced or cooked is called superfatting. Superfattng typically is used to make soap more moisturizing. However, soap may be superfatted regardless of whether or not there is coconut oil in the formulation. Of all the oils used in soap making coconut oil is one that is a mainstay of many formulations owing to the fact that it creates a super hard soap and produces a soap that lathers well. The downside to using coconut oil is that it has a high percentage of lauric acid, a medium chain triglyceride, which renders it a great cleaner but drying to the skin. Therefore formulations that have coconut oil - in any amounts but especially in amounts over 20% should be superfatted so that it will not be drying. The addition of the extra oil that does not take part in the saponification process but rather at the end of trace or the cook is small, usually anywhere from 1-5% of the total oils used. One other important reason to superfat natural soap especially cold process soap has to do with the soap's pH and its drying effects on the skin. Regular cold process soap, though a natural product has a high pH (9.5). Skin on the other hand has a low pH (4.5-6) in the acidic range. The difference between the low pH of skin and high pH of natural cold process and this incompatibility is what causes soap to be drying to the skin. In order to make soap more compatible with our skin's pH we should superfat natural cold process.

2.7 Physicochemical Characteristic of Fats/Oils

The physicochemical characteristic of fats/oils includes;

1. Saponification value is the amount of alkali necessary to saponify a definite quantity of the sample. It is expressed as the number of milligrams of potassium hydroxide (KOH) required to saponify one gram of the sample **(**Onyegbado *et al*., 2002**).**
2. The Iodine Value is a measure of the unsaturation of fatty acids and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample (percent iodine absorbed). The oil/fat sample taken in carbon-tetrachloride is treated with a known excess of iodine monochloride solution in glacial acetic (Wijs solution). The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulfate solution. The iodine value is a measure of the amount of unsaturation (number of double bonds) in a fat. A sample was dissolved in chloroform and then reacted, in the dark, with Wijs solution for a set amount of time **(**Onyegbado *et al*., 2002**)**.
3. The acid value is defined as the number of milligrams of potassium hydroxide required to neutralize the free fatty acids present in one gram of fat. It is a relative measure of rancidity as free fatty acids are normally formed during decomposition of oil glycerides. The value is also expressed as per cent of free fatty acids calculated as oleic acid. The acid value is determined by directly titrating the oil/fat in an alcoholic medium against standard potassium hydroxide/sodium hydroxide solution **(**Onyegbado *et al*., 2002**).**

4. Specific gravity is the ratio of the density of a substance to the density of a reference substance (usually water); equivalently, it is the ratio of the mass of a substance to the mass of a reference substance for the same given volume.

2.8 Physicochemical Characteristic of Soap

The physicochemical properties of soaps determine their quality and hence determine their efficiency and their cleansing properties. The physicochemical characteristic of soap depends on several factors which include the strength and purity of alkali, the kind of oil used and completeness of saponification. Such physicochemical characteristics include moisture content, total fatty matter (TFM), pH, free caustic alkalinity, foam ability and total alkali (Roila *et al*.,2001). The metals commonly used in soap making are sodium and potassium, which produce water-soluble soaps (Chalmers and Bathe, 1978). Good quality soap for cleansing purpose is one that strikes a balance in all the mentioned physicochemical parameters. The levels of the physicochemical properties in the Nigerian soaps remain obscure and thus the quality of individual soaps also remains unknown. Herein we report the levels of physicochemical parameters of soaps made from shear oil and sesame oil and some selected bar soaps that are used for cleansing purposes and that are manufactured and sold in Nigerian Markets.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials used in this work include the following:

I. conical flask
II. measuring cylinder
III. beaker
IV. burette
V. pipette
VI. water bath
VII. soap mold
VIII. stirrer
IX. reflux condenser
X. Agilent 7090B gas chromatography
XI. Agilent 5977A MSD mass spectroscopy detector system
XII. Tallow (Beef fat), Sesame seed oil and Shea butter.
XIII. Sodium hydroxide pellets (NaOH)

3.2 Methodology

3.2.1 Sample collection and preparation

Shea butter oil and sesame oil were purchased in Samaru market, Zaria in September 2016.

3.2.2 Preparation of tallow from animal (cow) fat

The preparation of fats for soap making consist of cleaning the fats to free them from impurities contained in it the process is called rendering. To render the fat, all meat tissues were removed. The fat was then placed in a stainless steel pot containing 500 cm^3 of distilled water. The mixtures were boiled until the fat melted. The solution was allowed to cool down and left overnight. The next day the fat had solidified and floated to the top forming a layer of clean fat. All the impurities being denser settled at the bottom as the fat remained on top of the water (Chalmers and Bathe, 1978).

3.3 Blending of the Oils/Fats

Blending of the fats and oil was carried out as described by kautom *et al*. (1996). Palm oil was used as a control oil in this research. The blending was done in four ways as follows;

I. *Butyrospermum parkii* fruit oil, *sesamum indicum* seed oil and animal fat were blended together,

II. *Butyrospermum parkii* fruit oil and *sesamum indicum* seed oil were blended together

III. *Sesamum indicum* seed oil and animal fat oil were blended together in different proportion

IV. *Butyrospermum parkii* fruit oil and animal fat oil were also blended together as well also in different proportions. These proportions are as stated in Tables 3.1, 3.2, 3.3 and 3.4 respectively.

Table 3.1: Blends of *butyrospermum parkii* fruit oil, tallow and *sesamum indicum* seed oil

The table contains the blending ratio for the three oils.

Fatty Blend	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Palm oil	**100**	**-**	**-**	**-**	**-**	**-**	**-**
***Butyrospermum parkii* fruit oil (%)**	**-**	**100**	**-**	**-**	**20**	**20**	**60**
Tallow (%)	**-**	**-**	**100**	**-**	**20**	**60**	**20**
***Sesamum indicum* seed oil (%)**	**-**	**-**	**-**	**100**	**60**	**20**	**20**

Table 3.2: Blends of *Butyrospermum parkii* fruit oil and Tallow

The table contains blending ratio of Shea butter oil and tallow

Blends	Percentage composition	
	SH (%)	T (%)
SHT_1	10	90
SHT_2	20	80
SHT_3	30	70
SHT_4	40	60
SHT_5	50	50
SHT_6	60	40
SHT_7	70	30
SHT_8	80	20
SHT_9	90	10

SH= Shea butter oil

T= Tallow fat

Table 3.3: Blends of Tallow and *Sesamum indicum* seed oil

The table contains blending ratio for tallow and sesame seed oil

Blend	Percent Composition	
	S (%)	T (%)
ST_1	10	90
ST_2	20	80
ST_3	30	70
ST_4	40	60
ST_5	50	50
ST_6	60	40
ST_7	70	30
ST_8	80	20
ST_9	90	10

S = Sesame seed oil

T = Tallow

Table 3.4: Blends of *Butyrospermum parkii* fruit oil and *Sesamum indicum* seed oil

The table contains blending ratio for shea butter oil and sesame oil

Blends	Percentage composition	
	SH (%)	S (%)
SHS_1	10	90
SHS_2	20	80
SHS_3	30	70
SHS_4	40	60
SHS_5	50	50
SHS_6	60	40
SHS_7	70	30
SHS_8	80	20
SHS_9	90	10

SH = Shea butter oil

T= Tallow

3.4 Physicochemical Parameters of Blended Oil

After the blending which was done by weighing the oils in their different proportions as indicated in Tables 3.1, 3.2, 3.3 and 3.4 which was followed by continues stirring and heating for 5 minutes in order to achieve a thorough mixture, physicochemical parameters which include saponification value, iodine value, acid value and specific gravity were determined for the individual blends obtained**.**

3.4.1 Determination of saponification value of the fats/oils

A sample is refluxed in 0.5M ethanolic KOH for 1.5 hours and titrated using 0.5M HCl.

The sample was melted and mixes thoroughly to ensure homogeneity. A Pipette was used to transfer 50 cm^3 of 0.5M KOH into a 250 cm^3 conical flask, some boiling stones were added, and reflux for 1.5 hours. A blank was prepared and ran simultaneously with the samples by pipetting 50 cm^3 of 0.5 M KOH into an empty 250 cm^3 conical flask, adding some boiling stones, and using a reflux condenser alongside the samples. After 1.5 hours of refluxing, the inside of the condensers was rinsed with about 25 cm^3 of distilled water. The flasks were removed from the condensers and the sample solutions were allowed to cool to room temperature. To each flask, 3 to 5 drops of phenolphthalein indicator was added and stirred and titrated, while mixing, with 0.5M HCl until the pink color just disappears. The respective titration volumes used to reach each endpoint was recorded. The Saponification values of the samples analyzed were calculated using the formula below and the results reported to one decimal place.

$$\text{SAP VALUE} = \frac{(cm^3\ \text{Blank} - cm^3\ \text{Sample})(\text{M of HCl})(56.1)}{(\text{wt.of oil/fat sample})} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.3.1$$

3.4.2 Determination of iodine value of the fats/oils

KI and deionized water were added to the flask and the solution was titrated with 0.1N sodium thiosulfate.

An appropriate amount of oil sample (10 g) was weighed into a tarred 250 cm^3 Erlenmeyer flask. The weight was recorded and the flask labeled accordingly. The sample was dissolved by adding 25 cm^3 of chloroform and the flask was swirled. The flask was heated (to about 50-60 ^{0}C) on a steam bath to completely dissolve the sample. After the sample solution has cooled to room temperature, 25 cm^3 of Wijs solution was pipetted into the flask, the contents were swirled till thoroughly mixed. A blank sample was prepared by pipetting 25 cm^3 of chloroform and 25 cm^3 of Wijs solution into an empty 250 cm^3 Erlenmeyer flask and the flask labeled accordingly. The flasks were stoppered with a ground glass stopper. About 5 cm^3 of the 15% KI Solution was pipetted into the stopper as well. Using a timer, the flask was stored in a dark place for 30 minutes to allow the reaction to take place completely. After the reaction was completed, all of the flasks was removed from the dark at the same time. About 20 cm^3 of the 15% KI solution and 75 cm^3 of distilled water was added to each of the flasks. A stir bar was added to each flask and mix as well. Using 0.1M sodium thiosulfate, the blank sample was titrated to a pale yellow endpoint. About 2 cm^3 of the starch indicator solution was added to the flask and continued titrating until the blue color just disappeared (usually a white endpoint). This titration was repeated for each of the samples. Using Equation 3.2, the iodine value was calculated and the values reported to one decimal place

$$\text{Iodine Value (I.V)} = \frac{(cm^3 \text{ Blank} - cm^3 \text{ Sample})(\text{N})(12.69)}{\text{sample wt.}} \quad \ldots\ldots 3.2$$

3.4.3 Determination of acid value of fat/oils

Ethyl alcohol (100 cm^3) was heated with 10 g of oil or fat sample in a 250 cm^3 beaker until the mixture begin to boil. The heating was stopped and the solution was titrated with KOH solution, using two drops of phenolphthalein as indicator with consistent shaking for which a permanent pink colour was obtained at the end point. The acid value was calculated using the equation 3.3

Acid value $= \frac{56.1 \ X \ M \ X \ V}{W}$ ……………………………………………………………………….3.3

Where,

V = Volume in cm^3 of standard 0.1 M sodium hydroxide used

M = Concentration of the Sodium hydroxide solution in mole/ dm^3

W = Mass in g of the sample

3.4.4 Determination of specific gravity (SG) of fats/oils

A 10 cm^3 of each oil blend was weighed in an analytical weighing balance as well as that of same 10 cm^3 of reference (water) and the weight recorded. And the specific gravity was calculated using equation 3.4:

SG = $\frac{\text{weight of oil sample (g)}}{weight\ of\ reference(water)(g)}$…………………………………………………………3.4

3.5 GC-MS Analysis

The analysis of the fatty acids in the principal oils, tallow shea butter and sesame oil was done at Multi-User laboratory Ahmadu Bello University, Zaria Nigeria. An Agilent 7090B gas chromatography coupled with an Agilent 5977A MSD mass spectroscopy detector system was used. The mass spectra were compared with NIST14.L mass spectra library (NIST, 2012) and the results are shown in the appendix. And the summary of the fatty acids present in the oil sample are shown in Tables 4.11, 4.12 and 4.13.

3.6 Preparation of Soap Samples (Saponification)

The amount of oil used for the saponification was 100 g and each oil blend required a different amount of base to react completely owing to the difference in their saponification value. The amount of NaOH required with the different oil blends was calculated according to saponification values. The calculated amount of NaOH for each of the oil blends was dissolved in 35 cm^3 of distilled water. The oil was warmed gently and poured into the beaker followed by the alkali solution to form an intimate mix and then stirred frequently for 1 min using stirring rod until reaction reached equilibrium which was observed when trails of the mixture was seen on the surface upon lifting the stirrer and this stage is called ‘trace’. The saponification mixture was then poured into mold and allowed to dry (cure) for 5 weeks (Eke *et al*., 2004). The cold process method was used for the saponification and the soap produced did not contain any additive.

3.7 Analysis of the Physicochemical Properties of Soap

The physicochemical properties analyzed in the soaps are:

3.7.1 pH determination

A 5 g sample of the soap shavings were weighed and dissolved with distilled water in a 100 cm^3 volumetric flask. The electrode of the pH meter was inserted into the soap solution and the pH reading was recorded (Warra *et al*., 2012)

3.7.2 Moisture content

For the determination of moisture content, 5 g of soap samples was accurately weighed using analytical balance of sensitivity 0.1 mg into dried tarred moisture dish in an oven for 2 hrs and temperature of 101˚C and repeated until a constant weight was reached. The percentage moisture was calculated using the following equation 3.5.

% moisture = $\frac{c_{s-}c_l}{c_{s-}c_w}\ x\ 100$……………………………………………………………3.5

Where; *CW* = Weight of crucible, *CS* = Weight of crucible + sample, *CL* = Weight of crucible + sample after floating (AOCS, 1997).

3.7.3 Free caustic alkali

Free caustic alkali was determined by the method described by (Milwidsky and Gabriel, 1994). According to this method, 5 g of finished soap was weighed and dissolved in 30 cm^3 of ethanol. Few drops of phenolphthalein indicator and 10 cm^3 of 20% $BaCl_2$ was added. The resulting solution was titrated against 0.05 M H_2SO_4 (aq).

Free Caustic Alkali was calculated using equation 3.6:

$$FCA = \frac{0.31}{w}\ X\ V_A \qquad \text{.....3.6}$$

Where; *VA* = Volume of acid, *W* = Weight of soap.

3.7.4: Total alkali

The total alkali was determined by titrating excess acid contained in the aqueous phase with standard volumetric NaOH solution. Ten grams of finished soap was weighed and 100 cm^3 of neutralized alcohol was added to it. About 5cm^3 of 0.5M H_2SO_4 solution was added to the mixture and heated till the soap sample dissolved. Test solution was titrated against 1 M NaOH using phenolphthalein as indicator (Mak-Mensah and Firempong, 2011). The total alkali was obtained using equation 3.7

$$\text{Total alkali} = \frac{V_{A-}v_B}{W} X\ 3.1 \qquad \text{.....3.7}$$

where V_A = volume of acid (cm^3)

V_B = volume of base (cm^3)

W = weight of soap (g)

3.7.5 Determination of total fatty matter (TFM)

The total fatty matter test was carried out by reacting soap with acid in the presence of hot water and measuring the fatty acids obtained. About 10 g of finished soap was weighed and 150 cm^3 distilled water was added and heated. The soap was dissolved in 20 cm^3 of 15 % H_2SO_4 while heating until a clear solution was obtained. Fatty acids on surface of the resulting solution was solidified by adding 7g of candlewax and reheated (Roila *et al*., 2001) The set up was allowed to cool to form cake. Cake was removed and blotted to dry and weighed to obtain the total fatty matter using equation 3.9:

$$\%TFM = \frac{A-X}{W} X\ 100 \quad \text{.....................................} 3.9$$

Where A= weight of wax + oil (g)

X = weight of wax (g)

W = weight of soap (g)

3.7.6 Foam ability test

The soap (2g) was added to a 250 cm^3 measuring cylinder containing 50 cm^3 of distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for 10 seconds, the cylinder was allowed to stand for 10 min. The height of the foam in the solution was measured and recorded (Warra *et al*., 2012).

CHAPTER FOUR

4.0 RESULTS

The results obtained for the physicochemical properties of both oil and soaps produced as well as the GC-MS analysis carried out on the fat/oil are presented in Tables 4.1, 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9, 4.10, 4.11, 4.12 and 4.13.

Table 4.1 contains the result of the saponification values of the fat/oil blends. Saponification value is used to estimate the quantity of sodium hydroxide required to saponify the fats/oils. High values of saponification value denotes a low molecular weight and high amount of alkali to neutralize the fat/oil. So from table 4.1, the saponification values (mgKOH/g) ranged from 172.50±3.97 – 215.29±2.98 with sample B having the lowest and sample G having the highest value. These values implies that sample B would require the least amount of NaOH to neutralize it while sample G would require the highest amount of NaOH to neutralize it.

Table 4.2 contains the result of the acid values of the fat/oil blends. Acid value is the weight of KOH in mg needed to neutralize 1g of fat/oil and it's the measure of the free fatty acids present in the fat/oil. The acid values (mgKOH/g) obtained for the oils/fat ranged between 9.45±0.13 – 43.30±1.23 with palm oil (standard oil) having the least value which implies it contains less fatty acids compared to the remaining oil while sample SHS1 having the highest acid value implies that it contains the highest number of fatty acids.

Table 4.3 contains the results of the iodine values of the fat/oil blends. Iodine value gives information on the degree of unsaturated fatty acids. High iodine values implies high degree of saturated fatty acids and vice versa. The iodine values ($gI_2/100g$) of the fat/oils ranged between 42.20±1.35 - 193.21±2.11. Sample C has the lowest value which implies high degree of unsaturation while SHT2 with the highest value implies high degree of saturation.

4.1 Physicochemical Properties of the Oil Blends

Table 4.1: Saponification values of oil blends

S/no	Oil samples	Oil blends (%)	Saponification value (mgKOH/g)
1	A	P : 100	204.61±2.27
2	B	SH: 100	172.50±3.97
3	C	T : 100	196.50±3.50
4	D	S : 100	187.92±2.01
5	E	SH:20, T:20, S: 60	193.20±6.44
6	F	SH:20, T:60, S: 20	181.98±1.49
7	G	SH:60, T:20, S: 20	215.29±2.98
8	ST1	S: 10, T: 90	195.00±1.20
9	ST2	S: 20, T: 80	180.32±1.41
10	ST3	S: 30, T: 70	181.98±1.49
11	ST4	S: 40, T: 60	187.11±2.08
12	ST5	S: 50, T: 50	172.50±3.23
13	ST6	S: 60, T: 40	190.00±1.50
14	ST7	S: 70, T: 30	191.00±1.70
15	ST8	S: 80, T: 20	185.00±2.01
16	ST9	S: 90, T: 10	192.00±1.41
17	SHT1	SH: 10, T: 90	193.03±2.70
18	SHT2	SH: 20, T: 80	193.21±2.11
19	SHT3	SH: 30, T: 70	191.73±2.11
20	SHT4	SH: 40, T: 60	190.20±2.19
21	SHT5	SH: 50, T: 50	187.11±0.99
22	SHT6	SH: 60, T: 40	185.31±0.70
23	SHT7	SH: 70, T: 30	181.23±1.19
24	SHT8	SH: 80, T: 20	177.27±1.20
25	SHT9	SH: 90, T: 10	173.11±1.10
26	SHS1	SH: 10, S: 90	201.00±2.33
27	SHS2	SH: 20, S: 80	193.11±2.11
28	SHS3	SH: 30, S: 70	190.27±0.10
29	SHS4	SH: 40, S: 60	185.20±1.31
30	SHS5	SH: 50, S: 50	180.21±2.50
31	SHS6	SH: 60, S: 40	177.21±1.17
32	SHS7	SH: 70, S: 30	177.33±1.23
33	SHS8	SH: 80, S: 20	175.00±1.19
34	SHS9	SH: 90, S: 10	173.89±2.00

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii* ; S= *Sesamum indicum*

Table 4.2: Acid values of oil blends

s/no	Oil samples	Oil blends (%)	Acid value (mgKOH/g)
1	A	P : 100	9.45±0.13
2	B	SH: 100	11.53±0.18
3	C	T : 100	10.69±0.12
4	D	S : 100	42.07±1.60
5	E	SH:20, T:20, S: 60	32.93±0.55
6	F	SH:20, T:60, S: 20	14.20±0.23
7	G	SH:60, T:20, S: 20	10.97±0.12
8	ST1	S: 10, T: 90	11.00±3.13
9	ST2	S: 20, T: 80	13.33±2.00
10	ST3	S: 30, T: 70	17.12±2.23
11	ST4	S: 40, T: 60	31.3±2.773
12	ST5	S: 50, T: 50	35.00±2.33
13	ST6	S: 60, T: 40	37.23±2.11
14	ST7	S: 70, T: 30	37.97±2.70
15	ST8	S: 80, T: 20	39.27±3.10
16	ST9	S: 90, T: 10	40.03±2.70
17	SHT1	SH: 10, T: 90	9.20±2.30
18	SHT2	SH: 20, T: 80	9.50±1.60
19	SHT3	SH: 30, T: 70	10.30±1.90
20	SHT4	SH: 40, T: 60	10.50±0.32
21	SHT5	SH: 50, T: 50	11.03±0.93
22	SHT6	SH: 60, T: 40	11.57±0.15
23	SHT7	SH: 70, T: 30	11.90±1.97
24	SHT8	SH: 80, T: 20	12.32±2.10
25	SHT9	SH: 90, T: 10	13.50±2.30
26	SHS1	SH: 10, S: 90	43.30±1.23
27	SHS2	SH: 20, S: 80	37.17±1.11
28	SHS3	SH: 30, S: 70	33.29±1.09
29	SHS4	SH: 40, S: 60	30.23±2.17
30	SHS5	SH: 50, S: 50	27.11±3.10
31	SHS6	SH: 60, S: 40	20.35±2.11
32	SHS7	SH: 70, S: 30	19.29±3.23
33	SHS8	SH: 80, S: 20	15.73±0.90
34	SHS9	SH: 90, S: 10	12.30±1.10

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii ;* S= *Sesamum indicum*

Table 4.3: Iodine values of the oil blends

S/no	Oil samples	Oil blends (%)	Iodine value ($gI_2/100g$)
1	A	P : 100	44.66±2.35
2	B	SH: 100	60.28±2.69
3	C	T : 100	42.20±1.35
4	D	S : 100	102.99±5.66
5	E	SH:20, T:20, S: 60	75.51±4.50
6	F	SH:20, T:60, S: 20	26.02±2.69
7	G	SH:60, T:20, S: 20	40.93±0.45
8	ST1	S: 10, T: 90	40.00±1.10
9	ST2	S: 20, T: 80	49.17±2.30
10	ST3	S: 30, T: 70	50.23±1.11
11	ST4	S: 40, T: 60	63.77±3.10
12	ST5	S: 50, T: 50	73.11±2.11
13	ST6	S: 60, T: 40	77.11±1.17
14	ST7	S: 70, T: 30	85.73±2.90
15	ST8	S: 80, T: 20	86.32±3.30
16	ST9	S: 90, T: 10	97.37±3.90
17	SHT1	SH: 10, T: 90	40.13±2.30
18	SHT2	SH: 20, T: 80	43.97±1.90
19	SHT3	SH: 30, T: 70	49.27±0.30
20	SHT4	SH: 40, T: 60	52.77±2.70
21	SHT5	SH: 50, T: 50	57.92±2.90
22	SHT6	SH: 60, T: 40	58.85±2.11
23	SHT7	SH: 70, T: 30	60.30±3.15
24	SHT8	SH: 80, T: 20	62.19±3.00
25	SHT9	SH: 90, T: 10	63.17±3.90
26	SHS1	SH: 10, S: 90	100.23±1.10
27	SHS2	SH: 20, S: 80	91.23±2.10
28	SHS3	SH: 30, S: 70	87.37±3.70
29	SHS4	SH: 40, S: 60	81.10±2.70
30	SHS5	SH: 50, S: 50	75.11±2.70
31	SHS6	SH: 60, S: 40	73.27±1.41
32	SHS7	SH: 70, S: 30	70.33±1.20
33	SHS8	SH: 80, S: 20	67.34±1.90
34	SHS9	SH: 90, S: 10	63.61±2.13

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii* ; S= *Sesamum indicum*

Table 4.4: Specific gravity values of oil blends

S/no	Oil samples	Oil blends (%)	Specific gravity
1	A	P : 100	0.940
2	B	SH: 100	0.900
3	C	T : 100	0.954
4	D	S : 100	0.902
5	E	SH:20, T:20, S: 60	0.910
6	F	SH:20, T:60, S: 20	0.922
7	G	SH:60, T:20, S: 20	0.936
8	ST1	S: 10, T: 90	0.913
9	ST2	S: 20, T: 80	0.932
10	ST3	S: 30, T: 70	0.911
11	ST4	S: 40, T: 60	0.921
12	ST5	S: 50, T: 50	o.917
13	ST6	S: 60, T: 40	0.907
14	ST7	S: 70, T: 30	0.943
15	ST8	S: 80, T: 20	0.927
16	ST9	S: 90, T: 10	0.930
17	SHT1	SH: 10, T: 90	0.913
18	SHT2	SH: 20, T: 80	0.925
19	SHT3	SH: 30, T: 70	0.933
20	SHT4	SH: 40, T: 60	0.919
21	SHT5	SH: 50, T: 50	0.972
22	SHT6	SH: 60, T: 40	0.910
23	SHT7	SH: 70, T: 30	0.936
24	SHT8	SH: 80, T: 20	0.973
25	SHT9	SH: 90, T: 10	0.909
26	SHS1	SH: 10, S: 90	0.913
27	SHS2	SH: 20, S: 80	0.927
28	SHS3	SH: 30, S: 70	0.942
29	SHS4	SH: 40, S: 60	0.900
30	SHS5	SH: 50, S: 50	o.929
31	SHS6	SH: 60, S: 40	0.911
32	SHS7	SH: 70, S: 30	0.903
33	SHS8	SH: 80, S: 20	0.928
34	SHS9	SH: 90, S: 10	0.940

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii* ; S= *Sesamum indicum*

Table 4.5 contains the fatty acids present in *sesamum indicum* seed oil when GC-MS analysis was carried out on the oil. Their respective mass spectral are giving in the appendix A.

From table 4.5, there are 6 saturated fatty acids and 2 unsaturated fatty acids. Nonanoic acid having least molecular weight of 158.2 and stearic acid having the highest molecular weight of 284.5.

Table 4.5 Fatty Acids present in *Sesamum indicum* seed oil

S/NO	Name of fatty Acid	MF	MW	RT	Ref. Number	CAS Number	LIBRARY
1	Dodecanoic Acid	$C_{12}H_{24}O_2$	200.3	25.717	207455	1000159-98-0	D:\MassHunter\library\NIST14.L
2	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270.5	20.839	130813	112-39-0	D:\MassHunter\library\NIST14.L
3	Linoleic Acid	$C_{18}H_{32}O_2$	280.4	23.348	140138	60-33-3	D:\MassHunter\library\NIST14.L
4	Myristic Acid	$C_{14}H_{28}O_2$	228.4	19.041	91415	544-63-8	D:\MassHunter\library\NIST14.L
5	Nonanoic Acid	$C_9H_{18}O_2$	158.2	12.476	31238	112-05-0	D:\MassHunter\library\NIST14.L
6	Oleic Acid	$C_{18}H_{34}O_2$	282.5	21.100	142070	112-80-1	D:\MassHunter\library\NIST14.L
7	Palmitic Acid	$C_{16}H_{32}O_2$	256.4	21.363	117419	57-10-3	D:\MassHunter\library\NIST14.L
8	Stearic Acid	$C_{18}H_{36}O_2$	284.5	21.552	144271	57-11-4	D:\MassHunter\library\NIST14.L

S/NO = Serial number

MF = Molecular Formula

MW = Molecular Weight

CAS NUMBER = Chemical Abstract Service Registry Number

Ref. NUMBER = Reference Number

RT = Retention Time

Table 4.6 contains the fatty acids present in *butyrospermum parkii* fruit oil when GC-MS analysis was carried out on the oil. From the table, the oil contains one saturated fatty acid and one unsaturated fatty acid.

Table 4.6 Fatty Acids present in *Butyrospermum parkii* fruit oil

S/NO	Name of fatty Acid	MF	MW	RT	Ref. Number	CAS Number	LIBRARY
1	Linoleic Acid	$C_{18}H_{32}O_2$	280.4	16.269	140138	60-33-3	D:\MassHunter\library\NIST14.L
2	Palmitic Acid	$C_{16}H_{32}O_2$	256.4	21.283	117419	57-10-3	D:\MassHunter\library\NIST14.L

S/NO = Serial number

MF = Molecular Formula

MW = Molecular Weight

CAS NUMBER = Chemical Abstract Service Registry Number

Ref. NUMBER = Reference Number

RT = Retention Time

Table 4.7 contains the fatty acids present in the tallow fat upon GC-MS analysis. The fatty acids present in the tallow are all unsaturated.

Table 4.7 Fatty Acids Present in Tallow (Cow Fat)

S/NO	Name of fatty Acid	MF	MW	RT	Ref. Number	CAS Number	LIBRARY
1	Linoleic Acid	$C_{18}H_{32}O_2$	280.4	18.194	140138	60-33-3	D:\MassHunter\library\NIST14.L
2	Oleic Acid	$C_{18}H_{34}O_2$	282.5	21.261	142070	112-80-1	D:\MassHunter\library\NIST14.L
3	Trans-13-octadecanoic acid	$C_{18}H_{34}O_2$	282.5	18.270	142094	693-71-0	D:\MassHunter\library\NIST14.L

S/NO = Serial number

MF = Molecular Formula

MW = Molecular Weight

CAS NUMBER = Chemical Abstract Service Registry Number

Ref. NUMBER = Reference Number

RT = Retention Time

4.2 Physicochemical Properties of the Manufactured Soaps from the Oil Blends

Table 4.8, 4.9, 4.10, 4.11, 4.12 and 4.13 gives the physicochemical properties of the produced soap samples. Percent total fatty matter, percent moisture content, total alkali, free caustic alkali, pH and foam ability were the major physicochemical properties deduced. Other properties includes hardness and colour of the soap samples.

Table 4.8 contains the total alkali value for the soap samples produced as well as for the controls. And the values ranged from 0.47±0.22 – 0.62±0.44 with the first control (C1) having the highest value amongst the control with value of 0.78±0.22.

Table 4.9 contains percent total fatty matter of the soap samples produced as well as for the controls. High values implies the soap would give the softness that is expected of a soap and vice versa. From the table, the values of the produced soap ranged between 41.30±0.29 - 75.80±0.28 with sample D having the lowest value while sample SHT9 had the highest value. The control soap C2 had the value (95.70±0.14) and the palm oil soap produced gave the least value (76.90±0.14) for the controls.

Table 4.10 contains pH values for the soap samples produced as well as for the controls. Control sample A has the highest pH value of 11.00±0.14 amongst the controls. And for the produced soap samples, the pH values ranged between 10.50±0.14 – 12.00±0.30. Samples SHS4 and B, having the lowest value while sample C has the highest value.

Table 4.11 contains the percent moisture content values for the produced soap as well as for the controls. The values for the produced soap samples ranged between 1.30±0.07 – 11.32±0.014. Sample C has the lowest value of 1.30±0.07 while sample B has the highest value. Amongst the three control used for the analysis, control C1 has the lowest value of 3.9±0.014 while control A has the highest value of 7.00±0.07

Table 4.8 Total alkali values of the soap samples produced

S/no	Samples	% composition of oil blends	Colour	Texture	Values
1	C1		WHITE	VERY FIRM	0.78±0.22
2	C2		WHITE	VERY FIRM	0.47±0.22
3	A	P : 100	YELLOW	FIRM	0.62±0.44
4	B	SH: 100	CREAM	VERY SOFT	0.47±0.22
5	C	T : 100	WHITE	VERY FIRM	0.47±0.22
6	D	S : 100	L-CREAM	FIRM	0.47±0.22
7	E	SH:20, T:20, S: 60	CREAM	FIRM	0.47±0.22
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	0.47±0.22
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	0.62±0.44
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	0.47±0.22
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	0.47±0.22
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	0.47±0.22
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	0.62±0.44
14	ST5	S: 50, T: 50	L-CREAM	FIRM	0.47±0.22
15	ST6	S: 60, T: 40	CREAM	FIRM	0.62±0.44
16	ST7	S: 70, T: 30	CREAM	FIRM	0.62±0.44
17	ST8	S: 80, T: 20	CREAM	FIRM	0.62±0.44
18	ST9	S: 90, T: 10	CREAM	FIRM	0.78±0.22
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	0.47±0.22
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	0.47±0.22
21	SHT3	SH: 30, T: 70	WHITE	FIRM	0.47±0.22
22	SHT4	SH: 40, T: 60	WHITE	FIRM	0.47±0.22
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	0.47±0.22
24	SHT6	SH: 60, T: 40	WHITE	SOFT	0.62±0.44
25	SHT7	SH: 70, T: 30	WHITE	SOFT	0.47±0.22
26	SHT8	SH: 80, T: 20	CREAM	SOFT	0.47±0.22
27	SHT9	SH: 90, T: 10	CREAM	SOFT	0.62±0.44
28	SHS1	SH: 10, S: 90	CREAM	HARD	0.62±0.44
29	SHS2	SH: 20, S: 80	CREAM	HARD	0.47±0.22
30	SHS3	SH: 30, S: 70	CREAM	HARD	0.47±0.22
31	SHS4	SH: 40, S: 60	CREAM	SOFT	0.62±0.44
32	SHS5	SH: 50, S: 50	CREAM	SOFT	0.62±0.44
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	0.47±0.22
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	0.47±0.22
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	0.47±0.22
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	0.47±0.22

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii ;* S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

Table 4.9 Values for Percent Total Fatty Matter of the Soap Samples Produced

S/no	Samples	% composition of oil blends	Colour	Texture	Values (%)
1	C1		WHITE	VERY FIRM	83.40±0.28
2	C2		WHITE	VERY FIRM	95.70±0.14
3	A	P : 100	YELLOW	FIRM	76.90±0.14
4	B	SH: 100	CREAM	VERY SOFT	73.80±0.28
5	C	T : 100	WHITE	VERY FIRM	70.15±0.21
6	D	S : 100	L-CREAM	FIRM	41.30±0.29
7	E	SH:20, T:20, S: 60	CREAM	FIRM	74.80±0.28
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	70.60±0.14
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	75.30±0.28
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	73.60±0.14
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	70.60±0.35
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	70.40±0.28
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	69.30±0.40
14	ST5	S: 50, T: 50	L-CREAM	FIRM	74.50±0.71
15	ST6	S: 60, T: 40	CREAM	FIRM	71.80±0.14
16	ST7	S: 70, T: 30	CREAM	FIRM	47.37±0.14
17	ST8	S: 80, T: 20	CREAM	FIRM	45.00±0.14
18	ST9	S: 90, T: 10	CREAM	FIRM	46.31±0.35
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	71.60±0.35
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	70.60±0.14
21	SHT3	SH: 30, T: 70	WHITE	FIRM	74.60±0.21
22	SHT4	SH: 40, T: 60	WHITE	FIRM	70.50±0.28
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	72.00±0.25
24	SHT6	SH: 60, T: 40	WHITE	SOFT	70.02±0.35
25	SHT7	SH: 70, T: 30	WHITE	SOFT	69.40±0.35
26	SHT8	SH: 80, T: 20	CREAM	SOFT	71.60±0.14
27	SHT9	SH: 90, T: 10	CREAM	SOFT	75.80±0.28
28	SHS1	SH: 10, S: 90	CREAM	HARD	44.90±0.35
29	SHS2	SH: 20, S: 80	CREAM	HARD	49.30±0.13
30	SHS3	SH: 30, S: 70	CREAM	HARD	50.15±0.35
31	SHS4	SH: 40, S: 60	CREAM	SOFT	70.60±0.14
32	SHS5	SH: 50, S: 50	CREAM	SOFT	65.60±0.28
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	65.40±0.21
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	71.00±0.14
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	75.60±0.35
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	75.10±0.35

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii ;* S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

Table 4.10 pH Values of the Soap Samples Produced

S/no	Samples	% composition of oil blends	Colour	Texture	Values
1	C1		WHITE	VERY FIRM	10.90±0.14
2	C2		WHITE	VERY FIRM	10.95±0.07
3	A	P : 100	YELLOW	FIRM	11.00±0.14
4	B	SH: 100	CREAM	VERY SOFT	10.50±0.14
5	C	T : 100	WHITE	VERY FIRM	12.00±0.30
6	D	S : 100	L-CREAM	FIRM	11.00±0.14
7	E	SH:20, T:20, S: 60	CREAM	FIRM	10.50±0.14
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	10.70±0.14
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	10.95±0.07
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	10.80±0.14
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	10.90±0.14
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	10.95±0.07
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	10.90±0.14
14	ST5	S: 50, T: 50	L-CREAM	FIRM	10.90±0.14
15	ST6	S: 60, T: 40	CREAM	FIRM	10.90±0.14
16	ST7	S: 70, T: 30	CREAM	FIRM	11.00±0.14
17	ST8	S: 80, T: 20	CREAM	FIRM	11.15±0.07
18	ST9	S: 90, T: 10	CREAM	FIRM	11.15±0.07
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	10.90±0.14
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	10.80±0.14
21	SHT3	SH: 30, T: 70	WHITE	FIRM	10.80±0.14
22	SHT4	SH: 40, T: 60	WHITE	FIRM	10.80±0.14
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	10.80±0.14
24	SHT6	SH: 60, T: 40	WHITE	SOFT	10.95±0.07
25	SHT7	SH: 70, T: 30	WHITE	SOFT	10.95±0.07
26	SHT8	SH: 80, T: 20	CREAM	SOFT	10.90±0.14
27	SHT9	SH: 90, T: 10	CREAM	SOFT	10.90±0.14
28	SHS1	SH: 10, S: 90	CREAM	HARD	11.20±0.14
29	SHS2	SH: 20, S: 80	CREAM	HARD	11.00±0.14
30	SHS3	SH: 30, S: 70	CREAM	HARD	11.15±0.07
31	SHS4	SH: 40, S: 60	CREAM	SOFT	10.50±0.14
32	SHS5	SH: 50, S: 50	CREAM	SOFT	10.80±0.14
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	10.80±0.14
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	10.90±0.14
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	10.80±0.14
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	10.80±0.14

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii* ; S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

Table 4.11 Percent Moisture Content of the Soap Samples Produced

S/no	Samples	% composition of oil blends	Colour	Texture	Values
1	C1		WHITE	VERY FIRM	3.90±0.014
2	C2		WHITE	VERY FIRM	6.40±0.07
3	A	P : 100	YELLOW	FIRM	7.00±0.07
4	B	SH: 100	CREAM	VERY SOFT	11.32±0.014
5	C	T : 100	WHITE	VERY FIRM	1.30±0.07
6	D	S : 100	L-CREAM	FIRM	10.03±0.07
7	E	SH:20, T:20, S: 60	CREAM	FIRM	6.31±0.07
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	3.51±0.014
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	7.35±0.06
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	I.87±0.031
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	3.39±0.014
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	3.97±0.073
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	4.31±0.021
14	ST5	S: 50, T: 50	L-CREAM	FIRM	4.95±0.07
15	ST6	S: 60, T: 40	CREAM	FIRM	5.13±0.07
16	ST7	S: 70, T: 30	CREAM	FIRM	7.35±0.06
17	ST8	S: 80, T: 20	CREAM	FIRM	8.13±0.056
18	ST9	S: 90, T: 10	CREAM	FIRM	10.19±0.07
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	3.52±0.014
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	4.96±0.07
21	SHT3	SH: 30, T: 70	WHITE	FIRM	5.31±0.014
22	SHT4	SH: 40, T: 60	WHITE	FIRM	8.32±0.07
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	9.12±0.07
24	SHT6	SH: 60, T: 40	WHITE	SOFT	9.31±0.014
25	SHT7	SH: 70, T: 30	WHITE	SOFT	9.73±0.056
26	SHT8	SH: 80, T: 20	CREAM	SOFT	9.98±0.017
27	SHT9	SH: 90, T: 10	CREAM	SOFT	10.92±0.014
28	SHS1	SH: 10, S: 90	CREAM	HARD	9.32±0.06
29	SHS2	SH: 20, S: 80	CREAM	HARD	8.39±0.014
30	SHS3	SH: 30, S: 70	CREAM	HARD	5.92±0.07
31	SHS4	SH: 40, S: 60	CREAM	SOFT	7.62±0.014
32	SHS5	SH: 50, S: 50	CREAM	SOFT	8.52±0.06
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	7.32±0.07
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	9.35±0.07
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	7.13±0.014
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	10.32±0.056

Values are expressed as mean and ± standard deviation of double determinations P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii ;* S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

Table 4.12 contains values of free caustic alkali present in the soap samples produced as we as those of the controls. From the table, samples with high ratio of Tallow in the oil blend contains small amount of the caustic alkali, but absent in the remaining soap samples as well as for the controls. Sample C has the highest value (0.35 ±0.02) while sample SHT5 has the least value (0.097±0.01).

Table 4.13 contains values for foam height (cm^3) of each of the soap samples and those of the controls. Control C1 has the highest value of 110.00±1.41 while C2 has the lowest value (90.00±3.50). The values ranged between 25.00±2.83 - 91.50±0.71 for the produced soap samples.

Table 4.12 Values of Free Caustic Alkali of the Soap Samples Produced.

S/no	Samples	% composition of oil blends	Colour	Texture	Values
1	C1		WHITE	VERY FIRM	NIL
2	C2		WHITE	VERY FIRM	NIL
3	A	P : 100	YELLOW	FIRM	NIL
4	B	SH: 100	CREAM	VERY SOFT	NIL
5	C	T : 100	WHITE	VERY FIRM	0.35 ±0.02
6	D	S : 100	L-CREAM	FIRM	NIL
7	E	SH:20, T:20, S: 60	CREAM	FIRM	NIL
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	NIL
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	NIL
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	0.21±0.031
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	0.137±0.02
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	0.087±0.01
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	NIL
14	ST5	S: 50, T: 50	L-CREAM	FIRM	NIL
15	ST6	S: 60, T: 40	CREAM	FIRM	NIL
16	ST7	S: 70, T: 30	CREAM	FIRM	NIL
17	ST8	S: 80, T: 20	CREAM	FIRM	NIL
18	ST9	S: 90, T: 10	CREAM	FIRM	NIL
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	0.312±0.02
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	0.29±0.014
21	SHT3	SH: 30, T: 70	WHITE	FIRM	0.273±0.03
22	SHT4	SH: 40, T: 60	WHITE	FIRM	0.157±0.02
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	0.097±0.01
24	SHT6	SH: 60, T: 40	WHITE	SOFT	NIL
25	SHT7	SH: 70, T: 30	WHITE	SOFT	NIL
26	SHT8	SH: 80, T: 20	CREAM	SOFT	NIL
27	SHT9	SH: 90, T: 10	CREAM	SOFT	NIL
28	SHS1	SH: 10, S: 90	CREAM	HARD	NIL
29	SHS2	SH: 20, S: 80	CREAM	HARD	NIL
30	SHS3	SH: 30, S: 70	CREAM	HARD	NIL
31	SHS4	SH: 40, S: 60	CREAM	SOFT	NIL
32	SHS5	SH: 50, S: 50	CREAM	SOFT	NIL
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	NIL
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	NIL
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	NIL
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	NIL

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii ;* S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

Table 4.13 Values of Foam Ability (Height) of the Soap Samples Produced

S/no	Samples	% composition of oil blends	Colour	Texture	Values(cm^3)
1	C1		WHITE	VERY FIRM	110.00±1.41
2	C2		WHITE	VERY FIRM	90.00±3.50
3	A	P : 100	YELLOW	FIRM	100.00±1.32
4	B	SH: 100	CREAM	VERY SOFT	25.00±2.83
5	C	T : 100	WHITE	VERY FIRM	65.50±0.71
6	D	S : 100	L-CREAM	FIRM	40.00±2.83
7	E	SH:20, T:20, S: 60	CREAM	FIRM	91.50±0.71
8	F	SH:20, T:60, S: 20	L-CREAM	VERY FIRM	80.00±2.83
9	G	SH:60, T:20, S: 20	CREAM	SLIGHTLY SOFT	50.00±1.41
10	ST1	S: 10, T: 90	WHITE	VERY FIRM	50.00±1.41
11	ST2	S: 20, T: 80	WHITE	VERY FIRM	55.00±2.83
12	ST3	S: 30, T: 70	WHITE	VERY FIRM	55.00±2.83
13	ST4	S: 40, T: 60	WHITE	VERY FIRM	50.00±1.41
14	ST5	S: 50, T: 50	L-CREAM	FIRM	55.00±2.83
15	ST6	S: 60, T: 40	CREAM	FIRM	40.00±2.83
16	ST7	S: 70, T: 30	CREAM	FIRM	50.00±1.41
17	ST8	S: 80, T: 20	CREAM	FIRM	40.00±2.83
18	ST9	S: 90, T: 10	CREAM	FIRM	64.00±1.41
19	SHT1	SH: 10, T: 90	WHITE	VERY FIRM	75.00±1.41
20	SHT2	SH: 20, T: 80	WHITE	VERY FIRM	75.00±1.41
21	SHT3	SH: 30, T: 70	WHITE	FIRM	75.00±1.41
22	SHT4	SH: 40, T: 60	WHITE	FIRM	30.00±2.12
23	SHT5	SH: 50, T: 50	L-CREAM	FIRM	50.00±1.41
24	SHT6	SH: 60, T: 40	WHITE	SOFT	25.00±2.83
25	SHT7	SH: 70, T: 30	WHITE	SOFT	25.00±2.83
26	SHT8	SH: 80, T: 20	CREAM	SOFT	25.00±2.83
27	SHT9	SH: 90, T: 10	CREAM	SOFT	25.00±2.83
28	SHS1	SH: 10, S: 90	CREAM	HARD	25.00±2.83
29	SHS2	SH: 20, S: 80	CREAM	HARD	25.00±2.83
30	SHS3	SH: 30, S: 70	CREAM	HARD	25.00±2.83
31	SHS4	SH: 40, S: 60	CREAM	SOFT	25.00±2.83
32	SHS5	SH: 50, S: 50	CREAM	SOFT	25.00±2.83
33	SHS6	SH: 60, S: 40	CREAM	VERY SOFT	40.00±2.83
34	SHS7	SH: 70, S: 30	CREAM	VERY SOFT	50.00±1.41
35	SHS8	SH: 80, S: 20	CREAM	VERY SOFT	50.00±1.41
36	SHS9	SH: 90, S: 10	CREAM	VERY SOFT	40.00±2.83

Values are expressed as mean and ± standard deviation of double determinations

P = Palm Oil; T= Tallow; SH= *Butyrospermum parkii;* S= *Sesamum indicum.;* L-CREAM = LIGHTER CREAM; C1= CONTROL 1; C2= CONTROL 2

CHAPTER FIVE

5.0 DISCUSSION

5.1 Physicochemical Properties of Oil

From Table 4.1, the saponification value (mgKOH/g) for samples B, ST5, SHT8, SHT9, SHS7, SHS8 and SHS9 ranged between the values 172.5±3.97 – 177.33±1.23. These values are lower than that obtained for canery melon seed oil with value 233.62±0.01 mgKOH/g (Warra et al., 2015) but greater than that of tallow with saponification value of 140.3 mgKOH/g (Warra et al., 2010). These values obtained are lower than the remaining oil blends and this implies they contain long chains of fatty acids and requires less amount of NaOH to saponify them. So since the saponification value falls between those of tallow and canery melon seed oil saponification values, these blends can as well be used for soap making. Also, the saponification values for samples D, F, ST2, ST3, ST4, ST8, SHT5, SHT6, SHT7, SHS4 and SHS5 ranged between the values 180.21±2.52 – 187.92±2.01. These values are less than that of the palm oil used as standard (204.61±2.27). The values are lower than that of neem seed oil 213 mgKOH/g (Akpan, 1999) and also lower than that of coconut oil 253.2 mgKOH/g (Oshinowo, 1987) but greater than that of crude canerium fruits (Ayoade *et al*., 2015). The high values implies they would require high amount of NaOH to saponify them as high saponification value gives a good soap. The saponification values of oil samples C, E, ST1, ST6, ST7, SHT1, SHT2, SHT3, SHT4, SHS2 and SHS3 ranged between 190.00±1.50 – 196.50±3.50. These values are high compared to those mentioned already and less than that of palm oil control which has a value of 204.61±2.27 mgKOH/g. these values are less than 220.19±0.4 mgKOH/g for *citrullus lanatus* seed oil (Edidiong *et al*., 2013) but greater than that of African pear oil 142.76 mgKOH/g (Ikhuoria and

Maliki, 2007). Samples G and SHS1 with saponification values 215.29±2.98 and 201.00±2.33 respectively are the highest saponification values amongst all the oil blends. These oil blends require high amount of NaOH to neutralize (saponify) it. The values are all greater than those of crude castor seed oil 185.85 mgKOH/g (Akpan *et al*., 2006) and less than that of *citrullus lanatus* seed oil 222.19±0.4 (Edidiong *et al*., 2013). These high values implies these blends would give a good quality soap. There are anomalies observed for the saponification values. Sample G having a high saponification value of 215.29±2.98 is greater than that of each components oils in this mixture. This might be as a result of the double determination carried out instead of replicate determination. This anomaly is also observed in samples SHS1, SHS2 and SHS3. These anomaly can be corrected if replicate determination is carried out instead of double determination.

As shown in table 4.2 which contains the acid values foe the oil samples, the highest value 43.3±1.23 (SHS1) contains more free fatty acids compared to the remaining oil blends. Also, samples B, C, F, G, ST1, ST2, ST3, SHT1, SHT2, SHT3, SHT4, SHT5, SHT6, SHT7, SHT8, SHT9, SHS7, SHS8 and SHS9 ranged between 9.20±2.30 – 19.29±3.23 mgKOH/g. These values are low compared to the remaining oil blends. The values are greater than that obtained for palm-kernel oil 3.60 mgKOH/g (Eke *et al*., 2004). These low values implies they contain less free fatty acids. Also, samples SHS6 and SHS5 has acid values of 20.35±2.11 and 27.11±3.10 respectively. These values are greater than that of palm oil control whose value is 9.45±0.13. these high values implies these oil samples will have a high content of fatty acids and the more the fatty acid present in an oil, the better the soap it will produce. Samples E, ST4, ST5, ST6, ST7, ST8, ST9, D, SHS1, SHS2, SHS3 and SHS4 has acid value ranged between 30.23±2.17 – 43.30±1.23. The high values also implies high content of fatty acids. These values are greater than those of Albizia Lebbech seed oil 10.69±0.21 mgKOH/g (Hassan *et al*., 2005), 3.83 mgKOH/g for groundnut (Warra *et al*., 2010) and canery melon seed oil 6.35±0.01 mgKOH/g (Warra *et al*., 2015) and *Hyptis Spiciger* seed oil (Ladan *et al*, 2010). Anomalies are also observed in the acid value tables. Sample SHS1 has a high acid value of 43.30±1.23 which is greater than that of each of the components that made up the blend (SH: 11.53±0.18 – S: 42.07±1.60). This might be as a result be as a result of the double determination. The anomaly is also observed for samples SHT8 and SHT9. These anomalies can be corrected by replicate determination instead of double determination.

As shown in table 4.3, sample D has the highest Iodine value of 102.99±5.66 I_2/100g and the lowest is sample F with value 26.02±2.69 I_2/100g. High iodine values implies high degree of saturated fatty acid and low value of unsaturated fatty acids. From table 4.3, samples SHT1-SHT9, C, B, F, G, ST1, ST2, ST3, ST4, SHS8 and SHS9 all have iodine values ranging between 26.02±2.69 – 67.34±1.90 I_2/100g. These values are very low compared to the remaining oil blends. These low values are greater than that obtained for palm kernel oil 17.0 I_2/100g (Adel *et al*.,1998) and *chryosphyllum albidum* seed oil with value 35.00 I_2/100g (Ayoade *et al*., 2012). Samples D, E, ST5, ST6, ST7, ST8, ST9, and SHS1-SHS7 all have an iodine value ranging between 70.33±1.20 – 102.99±5.66 I_2/100g. These values are greater than those of Shea nut oil 64.1 I_2/100g (Warra *et al*., 2010) and less than that of canery melon seed oil with a value of 135.6 ±0.07 (Warra *et al*., 2015) and 153.31 for wild *corchorusolitorius* seed oil (Ibrahim and Fagbohun, 2011). These high values implies these oil blends have a high quantity/amount of saturated fatty acids in them thereby making the oil blends suitable for soap making.

Anomalies are also observed in table 4.3. Samples SHT8 and SHT9with iodine values of 62.19±3.00 and 63.17±3.90 respectively are higher than the iodine value for each individual oil/fat that made up the blend. This anomaly might be as a result of the double determination carried out during the research instead of replicate determination.

The GC-MS spectral results and comparison of results with library search successfully enabled the identification of eight (8) fatty acids in *sesamum indicum* seed oil namely; nonanoic acid, myristic acid, palmitic acid, stearic acid, dodecanoic acid and hexadecanoic acid methyl ester oleic acid and linoleic acid, two (2) fatty acids in shea butter which are palmitic acid and linoleic acid and two (2) fatty acids present in tallow (animal fat) (oleic acid, linoleic acid and trans-a3-octadecenoic acid).

Sesamum indicum seed oil which contains 8 fatty acids has 6 saturated fatty acids which include nonanoic acid, myristic acid, palmitic acid, stearic acid, dodecanoic acid and hexadecanoic acid methyl ester as well as two unsaturated fatty acids which includes oleic acid and linoleic acid.

Butyrospermum parkii fruit oil (shea butter) contains one saturated fatty acid (palmitic acid) and one unsaturated fatty acid (linoleic acid).

Animal fat (tallow) on the other hand contains three (3) unsaturated fatty acid which includes oleic acid, linoleic acid and trans-13-octadecenoic acid.

The mass spectrum of the first fatty acid showed the molecular ion peak at m/z 158, corresponding to molecular formular $C_9H_{18}O_2$ (Nonanoic acid) indicating one degree of unsaturation. Also, the mass spectrum of the second, third, fourth, fifth, sixth, seventh, eighth and ninth fatty acid showed the molecular ion peak at m/z 228.4 (), 270.5 ($C_{17}H_{34}O_2$), 282.5($C_{18}H_{34}O_2$), 256.4($C_{16}H_{32}O_2$), 280.4($C_{18}H_{32}O_2$), 282.5($C_{18}H_{34}O_2$), 200.3($C_{12}H_{24}O_2$) and 284($C_{18}H_{36}O_2$) respectively.

Palmitic acid ($C_{16}H_{32}O_2$) is one of the most abundant and widespread natural saturated acid present in plants, animals and microorganisms (Frank *et al*., 2007) and was identified in sample. It is amongs the fatty acids that is used in the concentration of cosmetics (Liebert, 1987)

Stearic acid a saturated fatty acid with an 18-carbon chain and the IUPAC name octadecanoic acid was also found. Stearic acid is mainly used in the production of detergents, soaps and cosmetics such as shampoo and shaving creams products (Warra *et al*., 2015)

Oleic acid was also identified. On the basis of available data, it was concluded that oleic acid amongst others are safe in present practices of use and concentration in cosmetics (Liebert, 1987). Oleic acid are responsible for the lathering and washing properties of soaps (Ainie *et al*., 1996) and all the oil samples contains these four fatty acids which make them a good source of triglycerides when blended together for quality soap making.

Lauric acid on the other hand adds to the hardness of the bar. It also promotes a fluffy lather as well as cleans properly (SMR, 2008).

Linoleic acid adds conditioning and moisturizing properties to the bar soap. Also, the oil samples which contains myristic acid adds hardness to the bar soap (SMR, 2008). It also offers good cleaning properties and also adds a nice fluffy leather.

5.2 Physicochemical Properties of Soap Samples.

Table 4.8 contains the values for the total alkali contains in all the soap samples as well as the two controls: Giv soap (C1) and B – 29 soap (C2). From the table, samples G, ST6, ST7, ST8, SHT6, SHT9, SHS1, SHS4, SHS5 and ST4 had a total alkali value of 0.62±0.44 which is greater than that of C2 whose value is 0.47±0.22 but less than that of C1 and had the same value with palm oil which is the control (0.62±0.44) as well as less than that of Jatropha oil soap with value 0.76 (Ladan *et al*., 2010) but greater than that of neem oil soap with value 0.24±0.01 (Mak – Mensah and Firempong, 2011).

Also, from Table 4.8, Soap Samples B, C, D, E, F, ST1, ST2, ST3, ST5, SHT1, SHT2, SHT3, SHT4, SHT5, SHT7, SHT8, SHS2, SHS3, SHS6, SHS7, SHS8, SHS9 all have total alkali value of 0.47±0.22 which is less than that of C1 with value 0.78±0.22 and equal to that of C2 with value 0.47±0.22. The Total Alkaline value of 0.78±0.22 is less than that of onion seed oil soap with value of 0.92±0.02 (Warra and Sheshi, 2015) but greater than 0.76 reported for *jatropha* oil soap (Rangwala and Sarasan 2014). This implies that the soaps would not be corrosive to the skin since its values are very because according to According to Bureau of Indian Standards (BIS), good quality soaps must have less than 5% of alkali content (BIS, 2011) whereas according to ISO specification, soaps should have only below 2% of alkali content (ISO, 1975).
Table 4.8 also shows that Soap Samples ST9 have a total alkali value of 0.78±0.22 same with the C1 (0.78±0.22) but greater than 0.47±0.22 for C2. This value 0.78±0.22 is greater than 0.24±0.01 for neem oil soap (E.E. Mak – Mensah *et al*., 2011) but less than that obtained for onion seed oil soap 0.92±0.02 (Warra and Sheshi, 2015) and also 0.92±0.02 for canary melon seed oil soap (Warra *et al*, 2015).

Table 4.9 contains value of percent total fatty matter of all the soap samples. The TFM is how much fat substance the Soap has, i.e. it is an indication of soap quality. The more the value the better the quality of the soap. It is a measure of its suitability for bathing and washing of materials. According to International Standards (ISO), good quality bathing soaps and laundry must have TFM above 70% and below 70% respectively (ISO, 1975). Soap samples C, F, ST2, ST3, SHT2, SHT4, SHT6, SHS4 with percent TFM values of 70.15±0.212, 70.60±0.14, 70.6±0.35, 70.40±0.28, 70.60±0.14, 70.50±0.28, 70.02±0.35 and 70.6±0.14 respectively are in close agreement with each other. These values are greater than 36.66±0.02 for canary melon seed oil soap (Warra *et al*; 2015), but less than that of palm – kernel oil (PKO) soap which has a value of 83.6% (Eke *et al*., 2004). These high value of the samples shows that they would be suitable for bathing soap. According to Warra *et al*, (2015) soaps with 70% - 80% fatty matters are considered best one in quality. If the TFM lowers, then it would not give the softness that is expected of a soap.

Also, from Table 4.9, samples SHS5 and SHS6, both has a %TFM values of 65.6±0.28 and 65.4±0.21 respectively which are in close agreement with each other but less than those of the two controls; C1 and C2 with values 83.4±0.28 and 95.7±0.14 respectively. These values are greater than 63.75±0.07 for neem oil soap (Mak – Mensah and Firempong, 2011).but less than 74.4 for palm – kernel oil (Eke *et al*., 2004).

Table 4.6: Samples G, SHT9, SHS8 and SHS9 with %TFM values of 75.30±0.28, 75.80±0.28, 75.60±0.35 and 75.10±0.35 respectively are in close agreement with each other.

These values are all less than the values for the two commercial standards soaps; C1 and C2 with TFM values of 83.40±0.28 and 95.70±0.14 respectively and also less than the palm oil soap which is control with value 76.9±0.14. These values are greater than 36.60±0.02 for *canary* melon seed soap (Warra *et al*., 2015). High value of %TFM implies the soaps are of good quality.

Samples B and ST1 with %TFM values 73.8±0.28 and 73.6±0.14 respectively are in close agreement with each other. Their values are greater than 58±2.30 for Shea nut fat (Warra and Komo, 2014) but less than the two commercial standard soaps C1 (85.20±0.28) and C2 (95.70±0.14). These high values implies they are good for bathing.

Samples ST4 and SHT7 with %TFM values 69.3±0.40 and 69.40±0.35 respectively are all less than that of the two commercial soaps but greater than that of neem seed oil soap 63.75±0.007 (Mak – Mensah *et al*; 2011).

Samples E, STS and SHT3 with %TFM values of 74.8±0.28, 74.50±0.71 and 74.6±0.21 are all in close agreement with each other. Their values are all less than those of the commercial ones but greater than 36.66±0.02 onion seed oil (Warra and Fatima, 2015). These values are within the %TFM range 70 – 80% (Warra *et al*., 2015).

Samples SHT8, ST6, SHT5, SHT1 and SHS7 with %TFM values 71.6±0.14, 71.8±0.14, 72.00±0.35, 71.6±0.35 and 71.00±0.14 respectively are in close agreement with each other and the values are all less than that of the two controls C1 (83.40±0.28) and C2(95.70±0.14) and also greater than that of neem seed oil soap with %TFM value of 63.75±0.07 (Mak – Mensah *et al*., 2011) and also greater than that of 36.66±0.02 for canary melon seed oil soap (Warra *et al*., 2015).

Lastly, Samples D, ST8, ST9, ST7, SHS1, SHS2 and SHS3 with %TFM values of 41.30±0.29, 45±0.14, 46.31±0.35, 47.37±0.14, 44.9±0.35, 49.3±0.13 and 50.15±0.35 respectively are very low when compared with the other soaps made from other blends as well as those of the control C1 (83.40±0.28) and C2 (95.70±0.14) but are greater than 36.66±0.02 for onion seed oil soap (Warra and Sheshi., 2015). These low values of %TFM is due to presence of unreacted NaOH thereby suggesting that the soaps from these blends be used for laundry purposes instead of bathing (Toilet) soap (ISO, 1975). Low TFM make the soap harder, harsher on the skin and with greater tendency to become 'mushy' in water and so low TFM matter is usually associated with hardness and lower quality.

Table 4.10 which contains the pH values for the soap samples, Samples D, ST8, ST9, ST7, SHS1, SHS2 and SHS3 with values 11.00±0.14, 11.10±0.07, 11.15±0.07, 11.00±0.14, 11.20±0.14, 11.00±0.14, 11.15±0.007 and 11.20±0.14 respectively are all very high compared to the controls C1 (10.90±0.14) and C2 (10.95±0.07) and also less than 10.4±0.04 for neem oil soap (Mak – Mensah and Firempong, 2011). The value is also greater than that of Jatropha oil soap (Rangwala and Sarasam., 2014). This high value is due to incomplete alkali hydrolysis resulting from the saponification process. This indicates that, the prepared soap would be corrosive to the skin, so therefore, these samples would be best used as laundry soaps and especially for white clothes.

Samples B, E and SHS4 both with pH values 10.5±0.14 is higher than 10.4 for neem soap (Mak – Mensah and Firempong, 2011).but less than those of the two commercial soaps used as control A (10.90±0.14) and B (10.95±0.07) and also less than that of 11.03±0.02 for canary melon seed oil soap (Warra *et al*., 2015) and also higher than 8.6 reported for Jatropha seed oil soap (Rangwala and Serasan, 2014). The pH values are also less than that of the palm oil soap control with value 11.00±0.14.

Sample C with a very high pH of 12.00±0.30 greater than the controls C1 (10.9±0.07), C2 (10.9±0.14) and palm oil soap (11.00±0.14) would be very harsh / corrosive to the skin. Therefore, recommended for laundry soap especially clothes with stubborn stains. Samples G, ST3, SHT6 and SHT7 each with pH value of 10.95±0.07 are in close agreement with one of the control B (10.95±0.07) but greater than that of control A (10.90±0.14) and less than the palm oil soap control (11.00±0.14). This value 10.95±0.07 is less than 11.03±0.02 for *canary* melon seed oil soap (Warra *et al*., 2015) but greater than 8.6 reported for *Jatropha* oil soap (Rangwala and Serasan, 2014) as well as that of neem seed oil soap 10.4±0.04 (Mak – Mensah and Firempong, 2011).

Samples ST1, SHS5, SHS6, SHS8, SHS9, SHT2, SHT3, SHT4, and SHT5 all have the same pH value of 10.80±0.14. This pH value is less than the controls C1 (10.9±0.14), C2 (10.95±0.7) and palm oil soap (11.00±0.14). The pH value of 10.80±0.14 is less than that of 11.03±0.02 for onion seed oil (Warra and Fatima, 2015) but greater than 9.0 for palm oil (Beetseh and Anza, 2013).

Table 4.7: samples ST2, ST4, ST5, ST6, SHS7, SHT1, SHT8 and SHT9 with same pH value of 10.9±0.14 is in agreement with the pH value of one of the control C1(10.90±0.14) but less than the other controls C2 (10.95±0.07) and palm oil control (11.00±0.14). Since this value is between the two controls, it implies that these samples with pH 10.90±0.14 are ok for use for both as laundry soap and bathing soap. But the value is greater than 9.0 for black soap (Beetseh and Anza, 2013) but less than 11.03±0.02 for canary melon seed oil soap (Warra *et al*., 2015).

Sample F has a pH value of 10.7±0.14 less than those of the controls C1 (10.9±0.14), palm oil soap (11.00±0.14) and C2 (10.95±0.07). With this pH lower than the 3 controls; control

C1(10.90±0.14), control C2(10.95±0.07) and palm oil control (11.00±0.14),it's a good soap for both laundry and toilet soap.

High pH values are as a result of incomplete hydrolysis resulting from saponification process. It may be overcome by adding excess fat or oil or any other superfatting agent to reduce harshness. Higher pH values would neutralize the body's protective acid mantle that acts as a barrier against bacteria and viruses.

Table 4.11 contains values of percent moisture content of all the soap samples. From Table 4.11, samples ST2, ST3, SHT1 and sample F with percent moisture content of 3.39±0.014, 3.99±0.073, 3.52%±0.014 and 3.51%±0.014 respectively are all in close agreement with each other. These values are all less than that of the control C2 (6.4±%0.07) as well as that of C1(3.9±0.014). But only sample ST3 has a percent moisture content value less than that of C1. Also the values are less than 12.63±0.04 for neem oil soap (Mak – Mensah, 2011) as well as that of palm– kernel oil soap (8.20) as reported by (Eke *et al*., 2004). These low values might be due to the high percent of tallow oil in the oil samples used for the production of the soap samples. Moisture content is a parameter that is used in assessing the shelf – life of a soap.

Samples ST4, ST5, SHT2 with %MC values 4.31±0.021, 4.95±0.07 and 4.96±0.07 respectively are all in close agreement with each other. These values are all greater than that of the first control C1 with percent moisture content value 3.90±0.014 but all less than that of the B control (6.4±0.07). The low percent moisture values might also be as a result of the high percentage of Tallow present in the oil samples used for the production of each of the soap samples. These low values would not allow hydrolysis of the soap samples upon storage.

Samples ST1 and C with %MC values 1.87±0.031 and 1.30±0.07 respectively are in close agreement with each other. These low values are due to the high concentration of the tallow oil in used in the production of the soap. All these values are less than the two controls C1 (3.9±0.014) and C2 (6.4±0.07) used for the research. Samples ST6, SHT3 and ShS3 with %MC values of 5.13±0.07, 5.31±0.014 and 5.92±0.07 respectively. These values are all less than that of palm oil soap used as control 7.00±0.014% as well as the control C2 (6.4±0.07) but all less than that of C1 with values 3.9±0.014.

Samples ST7, SHS4, SHS6, SHS3 and G with %MC values 7.35±0.06, 7.62±0.014, 7.32±0.07, 7.13±0.014 and 7.35±0.06 respectively are all in close agreement with each other. These values are all greater than the two controls C1 (3.9±0.014) and C2 (6.4±0.07) as well as the standard oil (palm oil) soap (7.00±0.07). Also, these values are less than that of 9.53 for black soap produced from coconut oil (Ogunsuyi and Akinnawo, 2012) as well as 8.2 for palm kernel oil (Eke *et al*., 2004). Samples ST8, SHT4, SHS2 and SHS5 with %MC values 8.13±0.056, 8.32±0.07, 8.39±0.014 and 8.52±0.06 respectively are all in close agreement with each other. These values are all greater than the two controls C1 (3.9±0.014) and C2 (6.4±0.07) and also greater than the standard palm oil soap (7.00±0.07). These values are all less than that of neem seed oil soap with value 12.63±0.04 (Mak – Mensah and Firempong, 2011). These values are higher than those samples with high proportion of Tallow in the oil sample.

Samples SHT5, SHT6, SHT7, SHT8, SHS1 and SHS7 with %MC values 9.12±0.07, 9.31±0.014, 9.73±0.056, 9.98±0.017, 9.32±0.06 and 9.32±0.07 respectively are all in close agreement with each other. All these values are greater than 8.20 for palm kernel oil (Eke *et al*., 2004) but less than that of neem seed oil soap 12.63±0.04 (Mak – Mensah and Firempong, 2011). All these values are greater than the two controls C1 (3.9±0.014) and C2 (6.4±0.07) as well as the standard palm oil soap (7.00±0.07). These values high compared to the rest soap samples. The implication of high moisture content in soap is that the excess water could possibly react with any unsaponified neutral fat to give free fatty acid and glycerol in a process called hydrolysis on storage (Teweri, 2004).

Samples ST9, SHS9, SHT9 and D with %MC values of 10.19±0.07, 10.32±0.056, 10.92±0.014 and 10.03±0.07 respectively are all in close agreement with each other. These values are all greater than the rest soap samples which is as a result of the high percentage of *Butyrospernum parkii* fruit oil soap. All these samples are greater than 100:400 (ratio) composition of *butyrospermum parkii* oil to palm-karnel oil (PKO) soap with %MC value 9.60 (Eke *et al*., 2004) as well as both controls C1(3.9±0.014), C2(6.4±0.07) and also that of the standard palm oil soap with %MC value 7.0±0.07. Sample B has %MC value of 11.32±0.014 which is the highest among all the soap samples including the two controls C1 (3.9±0.014) and C2(6.4±0.07) as well as the standard palm oil soap (7.00±0.07); which is as a result of 100% *Butyrospermun parkii* oil used for the production of the soap sample. The value is greater than 9.53 for black – soap produced from coconut oil (Ogunsuyi and Akinnawo, 2012). Moisture content shows how hard a soap is. The higher the percent moisture content, the less hard the soap would be. Also, the lower the percent moisture content, the harder the soap. Soap samples with high percent of tallow were observed to have low percent moisture content thereby resulting in a very hard bar. The recommended percent moisture content is 5-15% (EICA, 2007) which implies these soup sample are suitable and ok for use.

Table 4.12 contains values for the free caustic alkali present in all the soap samples. Just few of the soap samples have free caustic alkali. This free caustic alkali is the amount of alkali free to prevent soap from becoming oily (Mak – Mensah and Firempong, 2011). Excess free caustic alkali causes skin itching and clothes wear – out (Mak – Mensah and Firempong, 2011).

Samples A,B,D,E,F,G, control 1, control 2, standard palm oil soap sample, ST4, ST5, ST6, ST7, ST8, ST9, SHS1, SHS2, SHS3, SHS4, SHS5, ShS6, SHS7, ShS8, ShS9, SHT6, SHS7, SHT8 and SHT9 all shows absent of free caustic alkaline in them which implies these soap samples are not corrosive to the skin. But samples C, ST1, ST2, ST3, SHT1, SHT2, SHT3, SHT4 and SHT5 all have free caustic alkali values of 0.35±0.02, 0.210±0.03, 0.137±0.02, 0.087±0.01, 0.312±0.02, 0.29±0.014, 0.273±0.03, 0.157±0.025 and 0.097±0.013 respectively with sample C having the highest value of free caustic alkali of 0.35±0.02 which might be as a result of incomplete saponification. All these values are greater than those of neem oil soap with value 0.06±0.002 (Mak – Mensah and Firempong, 2011).

Table 4.10 show the values of the foam ability of all the soap samples. Foam ability has little to do with cleansing ability (Mainkar and Jolly., 2000). It is of interesting importance to the consumer and is therefore considered a parameter in evaluating soaps and detergents (Warra *et al.*, 2015). From the Table (4.10); samples F, ST1, ST2, ST4, ST7, SHS7, SHS8 and SHT5 all have the same foam height as measured to be 50.00±1.42cm^3. This value is less than those of the controls C1 (110±1.14 cm^3) and C2 90±3.50 cm^3). The value is also less than that obtained for canary melon oil soap with value 75.13±0.15cm^3 (Warra *et al.*, 2015).

Also, samples D, SHS6, SHS9, ST8, SHT7 and ST6 all have the same foam height to be 40±2.83 cm^3 which is also less than that of the two controls. This value 40±2.83cm^3 is also less than 75.13±0.15 cm^3 for onion oil soap reported (Warra and Fatima., 2015).

Samples B, SHS1, SHS2, SHS3, SHS4, SHT5, SHT6, SHT7, SHT8 SHT9, SHS5 all have the same foam ability (cm^3) to be 25±2.83 cm^3 which is the lowest among all the soap samples produced which is as a result of the high percentage of *Butyrospermum parkii* oil in the oil sample (blends) used for the production of the various soap samples.

Sample SHT4 has a value of 30±2.12cm^3 which is less than that of the two controls C1 (110±1.14 cm^3) and C2 (90±3.50 cm^3). From Table 2.60, samples ST1, SHT, SHT2 and ShT3 all have the same foaming ability to be 75.00±1.41 cm^3. This value is slightly high compared to those which have a high percentage composition of *Butyrospermum parkii* oil in the blends used for the production of the soap sample. This value 75.00±1.41 cm^3 is less than those of the two controls C1 and C2 as well as the palm oil soap with values 110±1.14 cm^3, 90±3.50 cm^3 and 100±1.32 cm^3 respectively. Samples C and ST9 with foam height 65.5±0.71cm^3 and 64±1.41cm^3 respectively are in close agreement with each other but are less than that of C1 (110±1.14 cm^3) and C2 (90±3.50 cm^3). This value is also less than that of canary melon seed oil soap 75.13±0.15 (Warra *et al*., 2015). While sample ST3 and ST5 both have foam ability of 55±2.83 cm^3.

Sample E has the highest foam ability of 91.5±0.71 cm^3 among all the soap samples produced followed by sample G with value 80.00±2.83cm^3. These values are all less than that of the control C1 (110±1.14 cm^3) and C2 (90±3.50 cm^3) as well as that of standard palm oil (100±1.32cm^3) but greater than those of onion oil soap with values 75.13±0.15 cm^3(Warra and Sheshi., 2015). These differences in the foam ability between the controls might be as a result of the fact that foaming agent additives were not added during the saponification process. Foam ability has little to do with cleansing ability (Mainkar and Jolly, 2000). It is of interesting importance to the consumer and therefore considered a parameter in evaluating soaps and detergents (Warra *et al*., 2015).

CHAPTER SIX

6.0 CONCLUSION AND RECOMMENDATION

6.1 Conclusion

The Saponification value, Iodine value, Acid value and Specific gravity were determined in the various oil blends before the Saponification was done. The major oil / fat used which are palm oil (Standard), *butyrospermum parkii* fruit oil, *Sesamum Indicum* seed oil and animal fat (Tallow) had Saponification values of 204.61±2.27, 172.50±3.17, 187.92±2.01 and 196.50±3.50 respectively, acid values of 9.45±0.13, 11.53±0.18, 42.07±1.60 and 10.69±0.12 respectively, Iodine value of 44.66±2.35, 60.28±2.69, 102.99±5.66 and 42.20±1.35 respectively and lastly specific gravity values of 0.940, 0.900, 0.902 and 0.954 respectively. From the different blends obtained, Sample G with blend ratio SH:T:S – 60:20:20 has the highest saponification value of 215.29±2.98 mgKOH/g followed by Sample SHS1 (SH:10, S:90) with value 201.00±2.33 and blend ST7 has the least Saponification value of 172.50±3.23. Also, from the acid values obtained, SHS1 (SH:10; S:90) has the highest acid value 43.30±1.23mgKOH/g followed by ST9 (S:90; T:10) with acid value 40.03±2.70.

The soap samples SHS8 (Shea butter 80% - sesame 20%), SHS9 (Shea butter 90% - sesame 10%) SHT9 (Shea butter 90% - Tallow 10%) and G (Shea butter 60% - sesame 20% - Tallow 20%) with total fatty matter (75.60±0.35), (75.10±0.35), (75.80±0.28) and (75.30±0.28) respectively, gave the best soap judging by % Total Fatty Matter (TFM) while blends E (Shea butter 20% - sesame 60% - Tallow 20%) and F (Shea butter 20% - sesame 60% - Tallow 20%) with foam height (cm^3) (91.50±0.71) and (80.00±2.83) respectively gave a better soap in terms of foaming ability. The pH values observed for blended samples at 10% soap solutions were ranged from 10.50±0.14 to 11.20±0.14, while pH values for the commercial soaps used as standard at 10% soap solutions were 10.90±0.14 to 10.95±0.07. Soap sample C (100%Tallow) had the highest pH value of 12.00±0.30 which makes it harsh for the body. Thus, from the results obtained in the present study it can be concluded that due to the favorable physicochemical properties (high %TFM, high pH and high Foam height) of soap samples, some should be recommended for bathing (G, SHS8, SHS9 and SHT9) and some for laundry (D, ST8, ST7, ST9, SHS1, SHS2 and SHS3) judging by % TFM and pH values as well as from the comparison with the commercial soaps used as control. The results were compared with the data in the literature. It can be concluded that the values determined are within the limits set by standards (SON and ASTM).

6.2 RECOMMENDATION

From the analysis carried out on all the soap samples produced;

I. It is hereby recommended that oil blends of Tallow, Sesame oil and Shea butter oil with high percent of Tallow be used to produce soaps if foam ability (height) is taken into consideration. But since %Total fatty matter is considered as the determining parameter when producing soap especially in this research work, oil blends with high percent of *Sesamum Indicum* seed oil be used to produce laundry soap as it will produce soaps of low %TFM.

II. Also, oil blends with high percent of *butyrospermum parkii* oil be used to produce toilet soaps as it leads to the production of soaps with relatively high %TFM. High value of %TFM implies that there is high content of fatty matter which would give the softness that is expected out of soap.

III. Blends of Tallow with other fruit and seed oil is recommended as it has a wonderful property of hardening properly compared to *Sesamum Indicum* seed oil and *butyrospermum parkii* fruit oil.

IV. Since this research covered only three selected oils/fats, other oils/fats as well as blend options or ratios should be investigated. For instance coconut oil is a good oil for soap making, so if blended with either tallow or any seed oil, a desirable soap with good quality would be produced.

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Appendix A

SAMPLE A (*Sesamum indicum* seed oil)

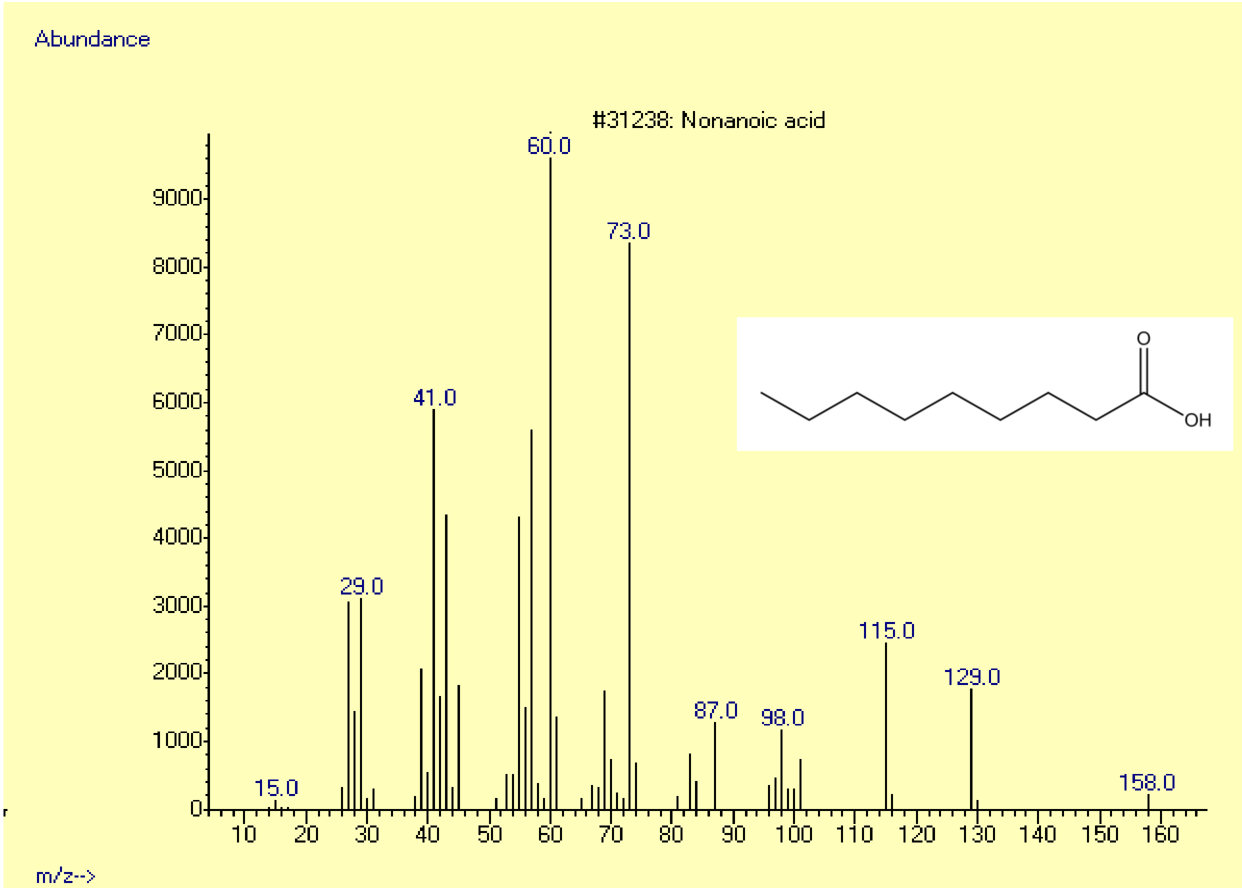

Figure A1: Mass spectra of Nonanoic acid

Name of fatty acid: Nonanoic Acid.

Molecular formular: $C_9H_{18}O_2$

Retention Time: 12.476

Ref N0.: 31238

CAS NO.: 112-05-0

Library: D:\MassHunter\Library\NIST14.L

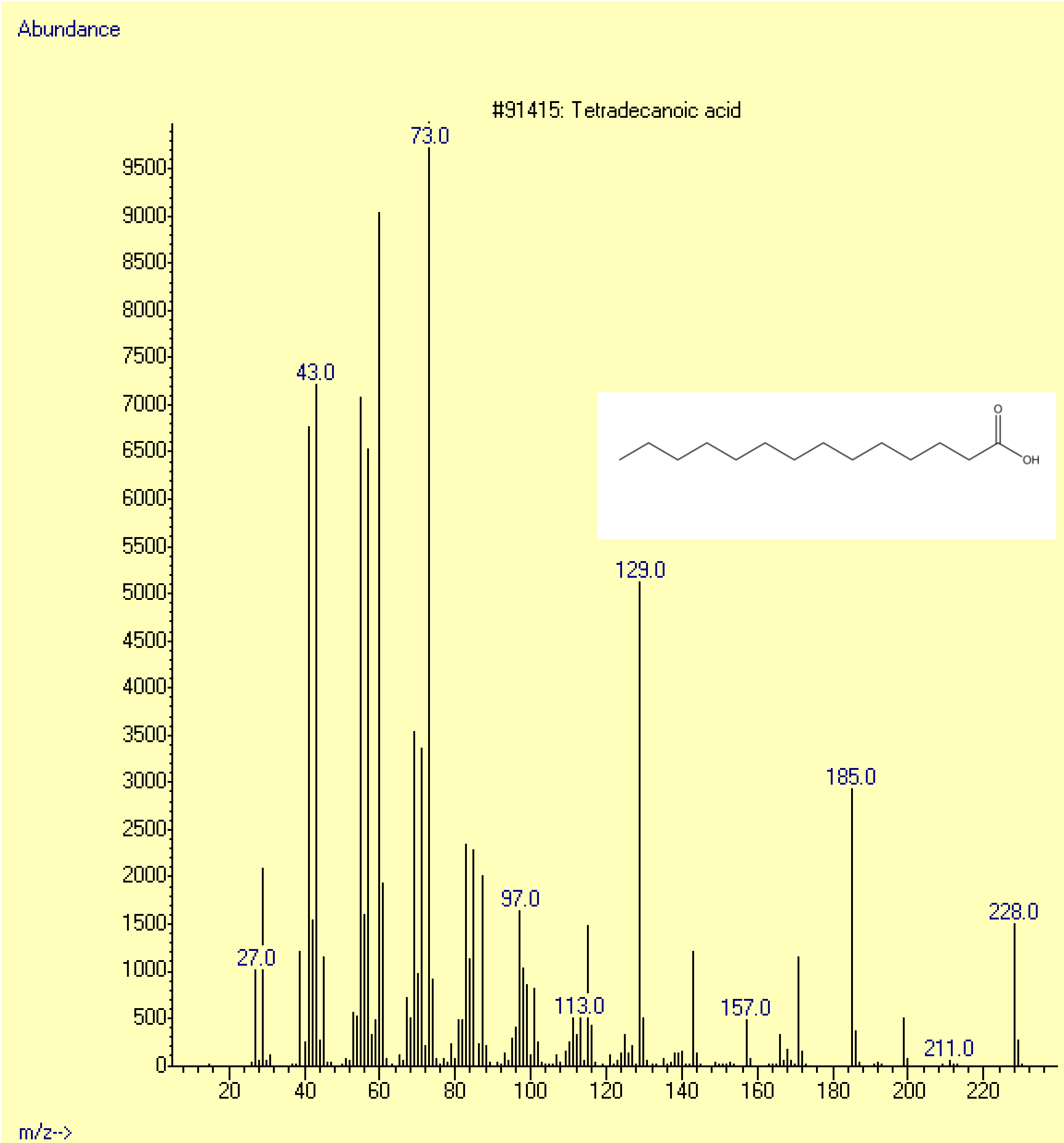

Figure A2: Mass spectral of Myristic Acid.

Name of fatty acid: Myristic Acid.

Molecular formular: $C_{14}H_{28}O_2$

Retention Time: 19.041

Ref N0.: 91415

CAS NO.: 544-63-8

Library: D:\MassHunter\Library\NIST14.L

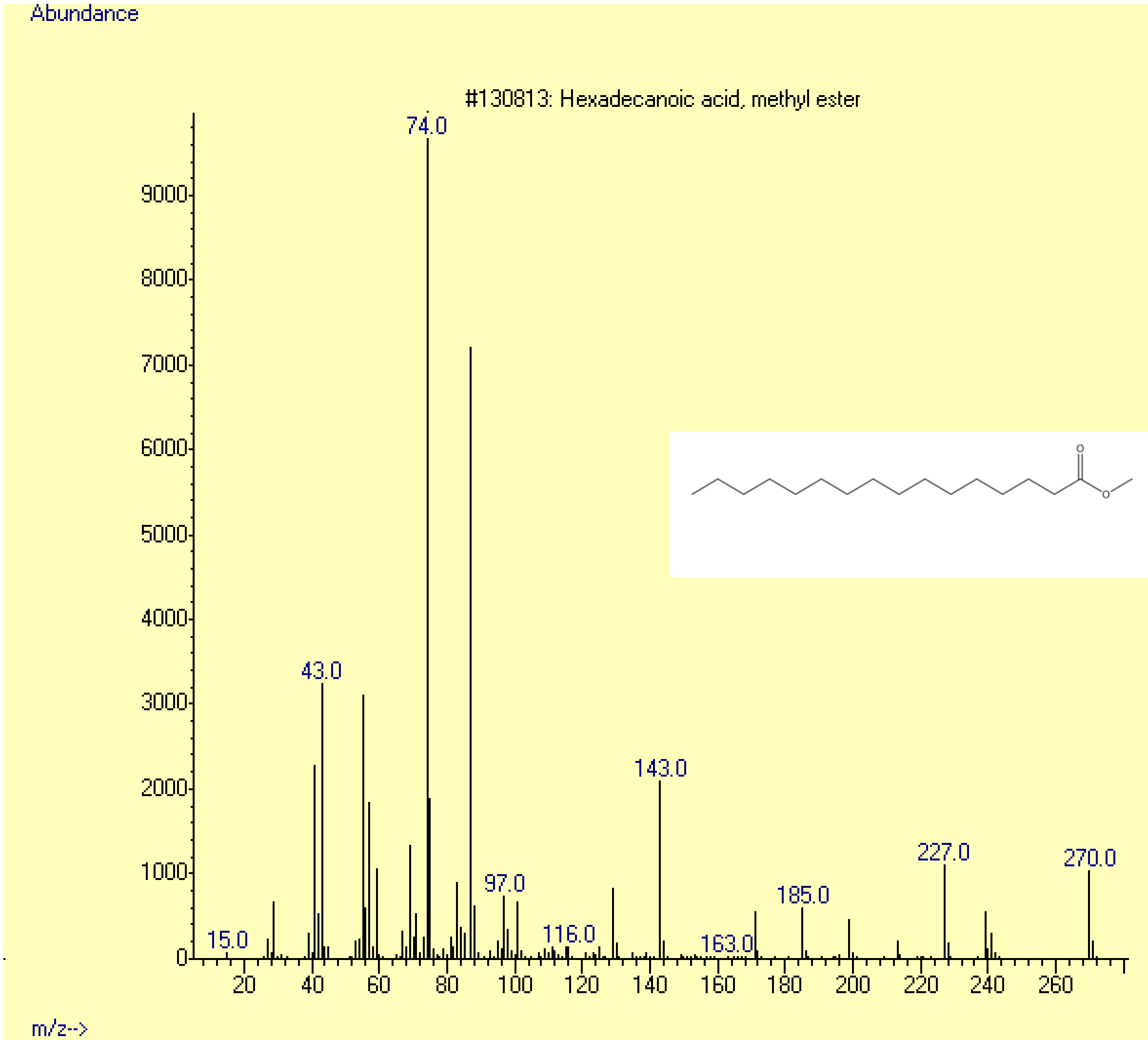

Figure A3: Mass spectral of Hexadecanoic acid, methyl ester

Name of fatty acid: Hexadecanoic acid, methyl ester

Molecular formular: $C_{17}H_{34}O_2$

Retention Time: 20.839

Ref N0.: 130813

CAS NO.: 112-38-0

Library: D:\MassHunter\Library\NIST14.L

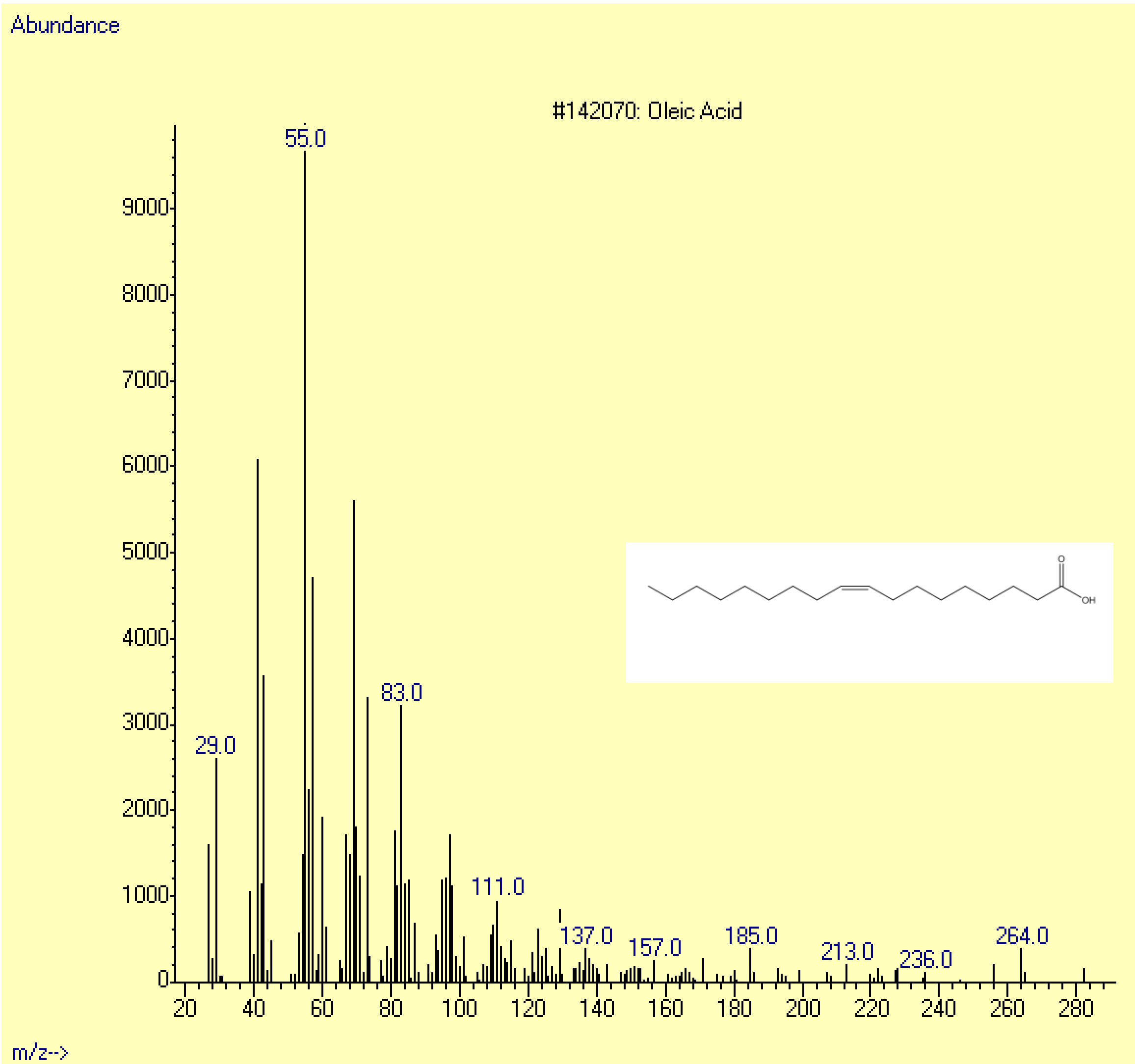

Figure A4: Mass spectra of Oleic

Name of fatty acid: Oleic Acid.

Molecular formular: $C_9H_{18}O_2$

Retention Time: 21.100

Ref N0.: 142070

CAS NO.: 112-80-1

Library: D:\MassHunter\Library\NIST14.L

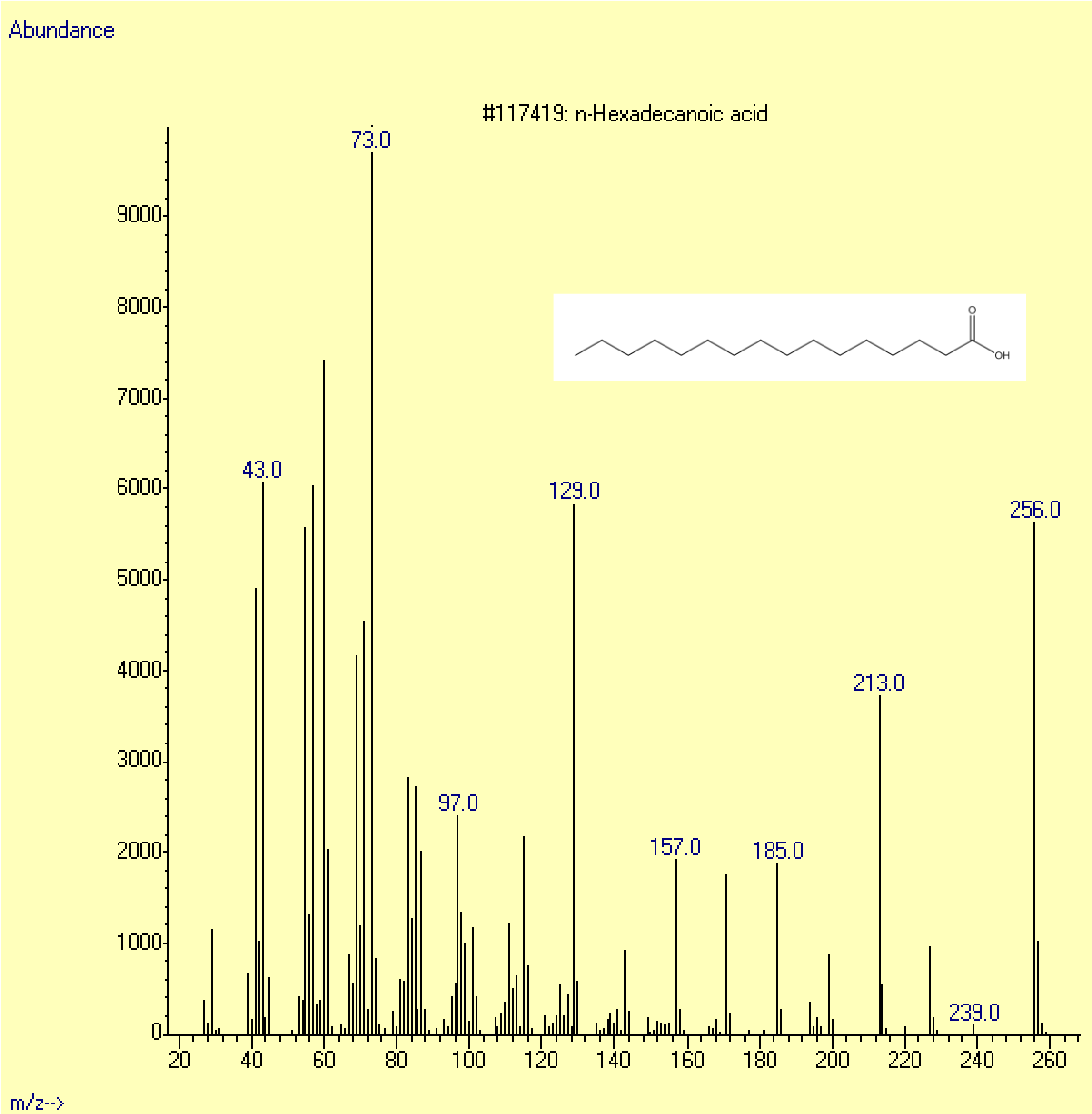

Figure A5: Mass spectra of Palmitic Acid.

Name of fatty acid: Palmitic Acid.

Molecular formular: $C_{16}H_{32}O_2$

Retention Time: 21.363

Ref N0.: 117419

CAS NO.: 57-10-3

Library: D:\MassHunter\Library\NIST14.L

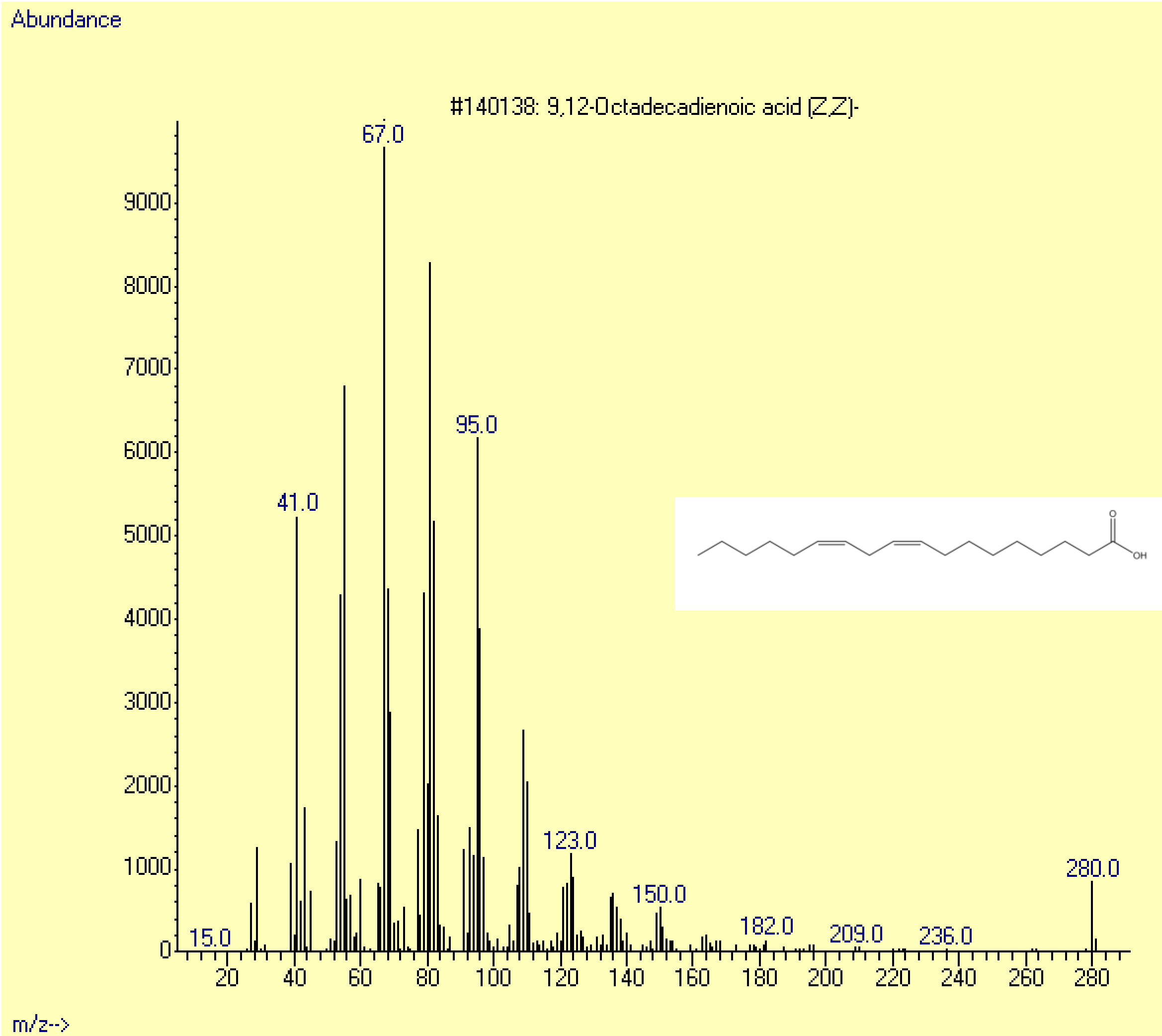

Figure A6: Mass spectra of Linoleic Acid.

Name of fatty acid: Linoleic Acid.

Molecular formular: $C_{18}H_{32}O_2$

Retention Time: 23.348

Ref N0.: 140138

CAS NO.: 60-33-3

Library: D:\MassHunter\Library\NIST14.L

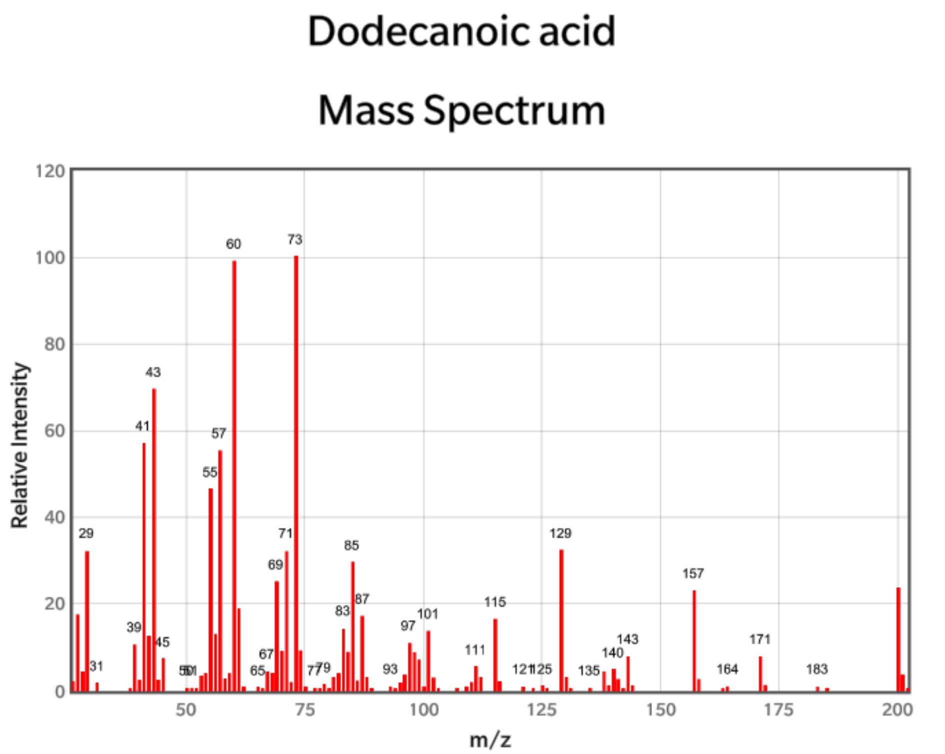

Figure A7: Mass spectra of dodecanoic acid

Name of fatty acid: dodecanoic acid

Molecular formular: $C_{12}H_{24}O_2$

Retention Time: 25.717

Ref N0.: 207455

CAS NO. 112-39-0

Library: D:\MassHunter\Library\NIST14.L

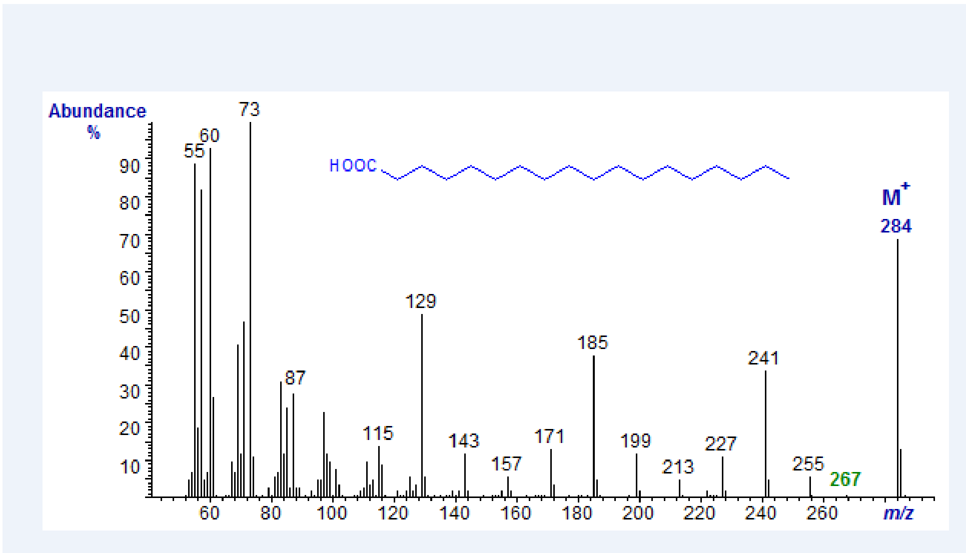

Figure A8: Mass spectra of Stearic Acid.

Name of fatty acid: Stearic Acid.

Molecular formular: $C_{18}H_{36}O_2$

Retention Time: 21.552

Ref N0.: 144271

CAS NO. 57-11-7

Library: D:\MassHunter\Library\NIST14.L

SAMPLE 2 (*Butyrospermum parkii* fruit oil)

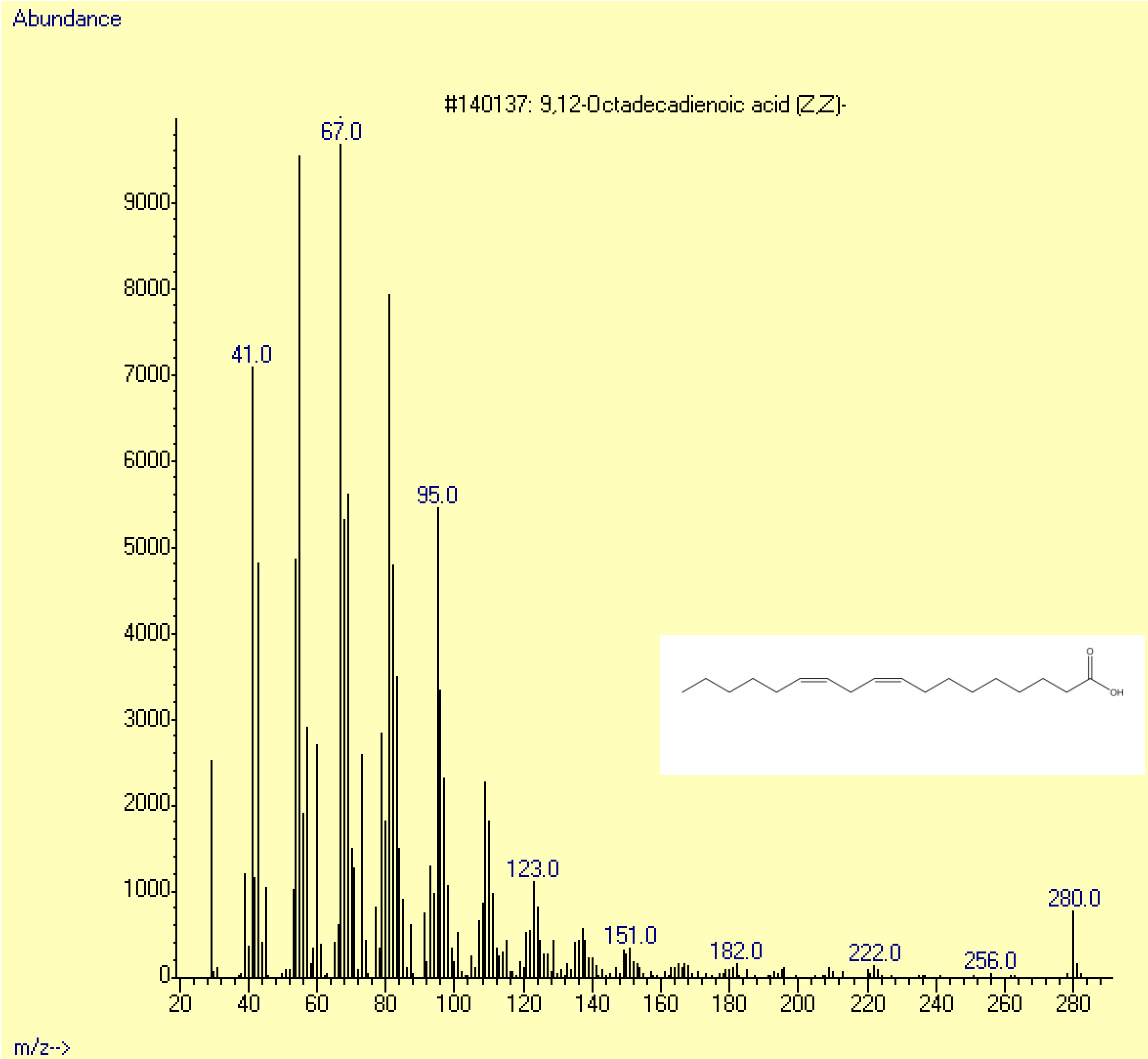

Figure A9: Mass spectra of Linoleic Acid.

Name of fatty acid: Linoleic Acid.

Molecular formular: $C_{18}H_{32}O_2$

Retention Time: 16.269

Ref N0.: 140138

CAS NO.: 60-33-3

Library: D:\MassHunter\Library\NIST14.L

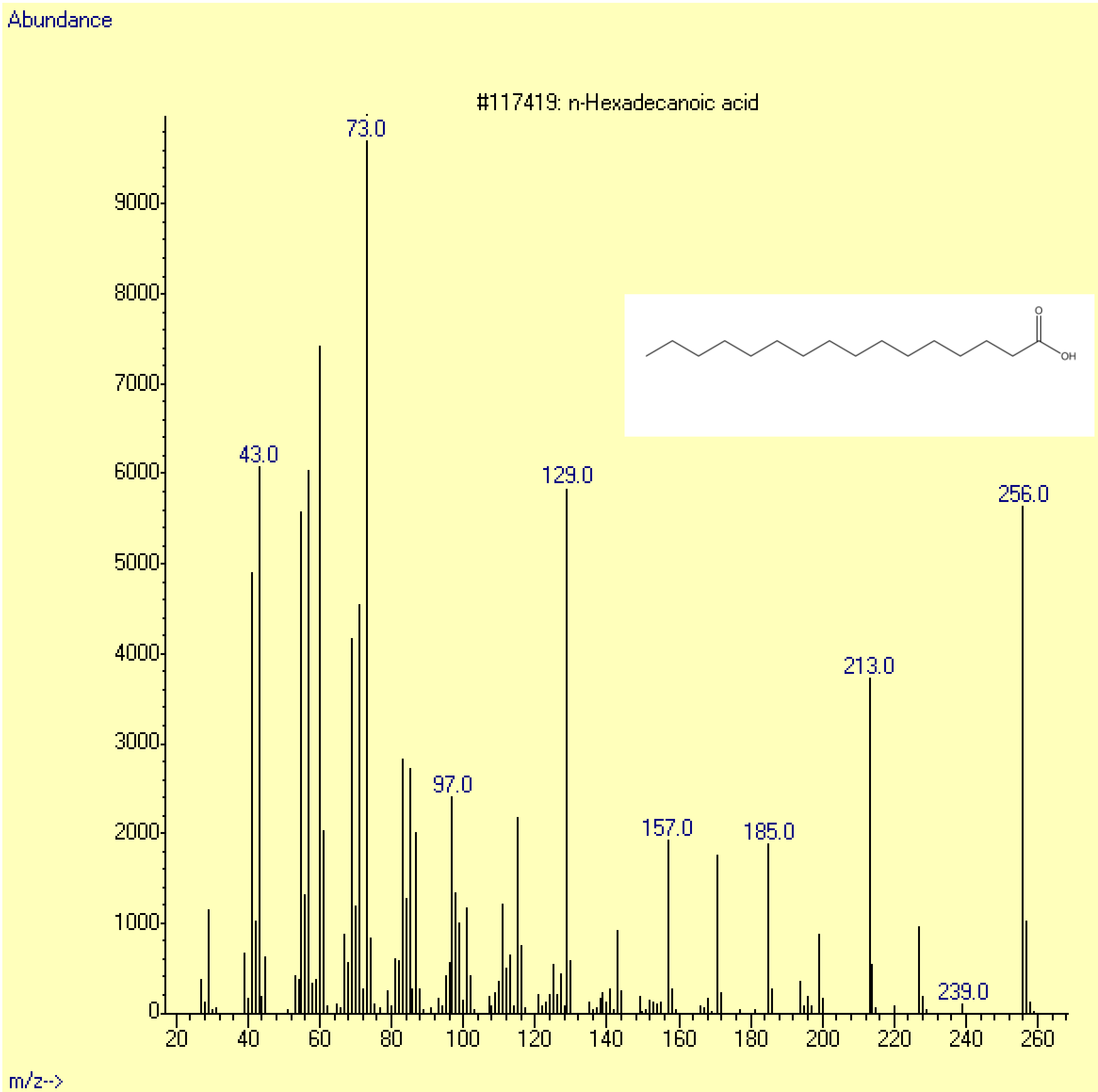

Figure A10: mass spectra for Palmitic Acid

Name of fatty acid: Palmitic Acid

Molecular formular: $C_{16}H_{32}O_2$

Retention Time: 21.283

Ref N0.: 117419

CAS NO.: 57-10-3

Library: D:\MassHunter\Library\NIST14.L

SAMPLE 3 (Tallow)

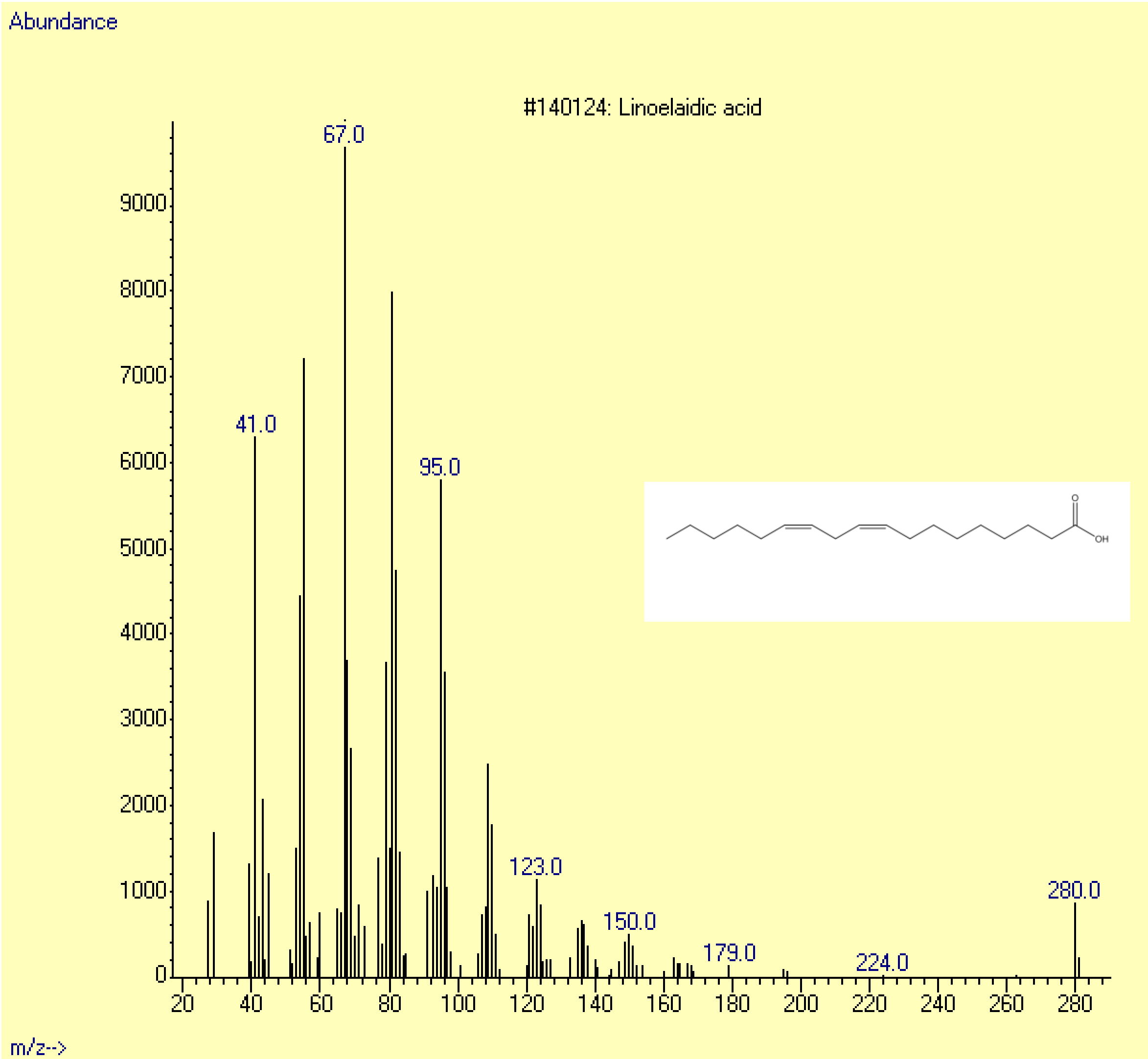

Figure A11: Mass spectra for Linoeliedic Acid.

Name of fatty acid: Linoeliedic Acid.

Molecular formular: $C_{18}H_{32}O_2$

Retention Time: 18.194

Ref N0.: 140138

CAS NO.: 60-33-3

Library: D:\MassHunter\Library\NIST14.L

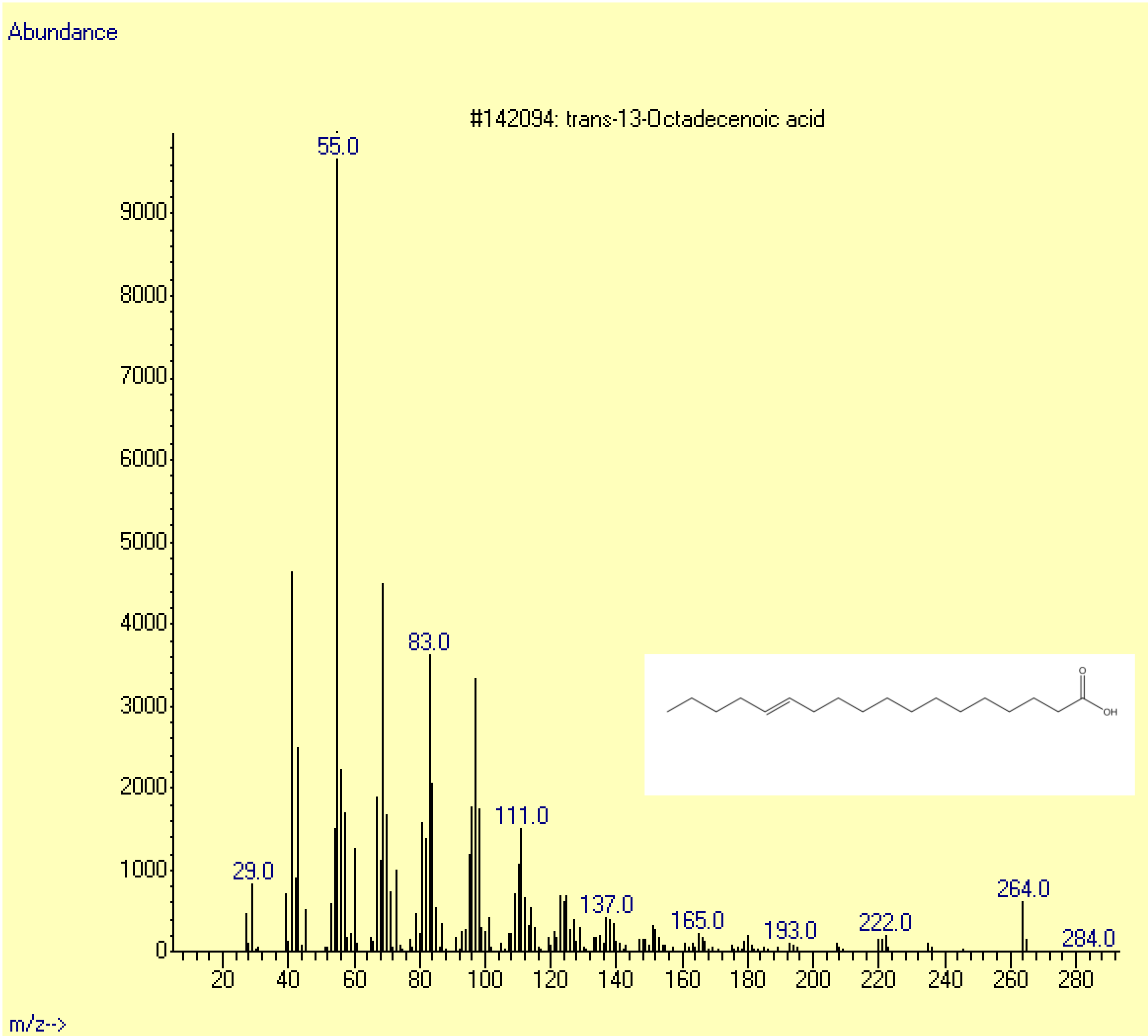

Figure A12: Mass spectra for trans-13-octadecenoic Acid.

Name of fatty acid: trans-13-octadecenoic Acid.

Molecular formular: $C_{18}H_{34}O_2$

Retention Time: 18.270

Ref N0.: 142094

CAS NO.: 693-71-0

Library: D:\MassHunter\Library\NIST14.L

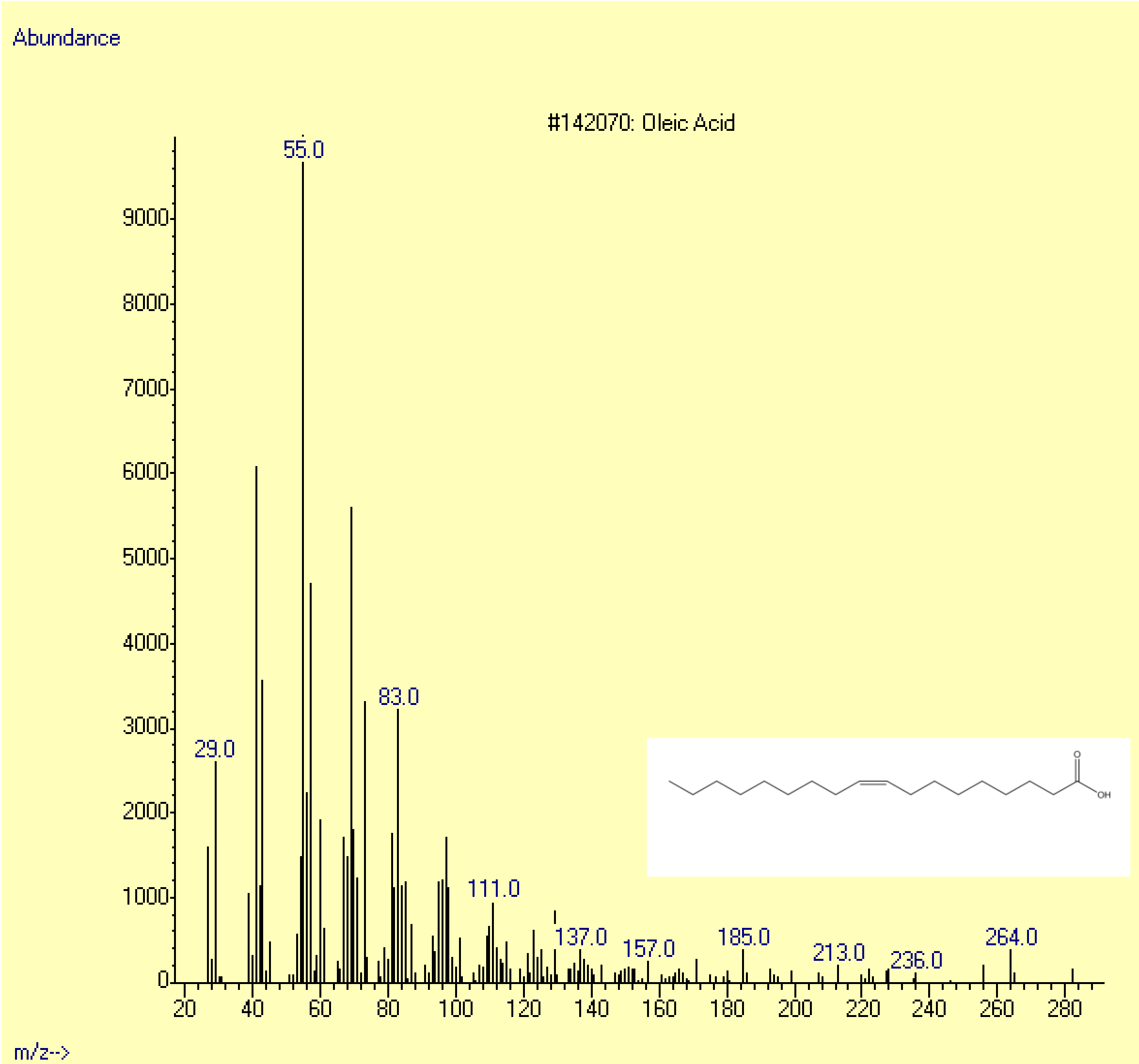

Figure A13: Mass spectra of Oleic Acid.

Name of fatty acid: Oleic Acid.

Molecular formular: $C_{18}H_{24}O_2$

Retention Time: 21.261

Ref N0.: 1402070

CAS NO.112-80-1

Library: D:\MassHunter\Library\NIST14.L