Preparation of Soap Using Different Types of Oils and Exploring its Properties

Submitted by
Debesh Mishra
(109ch0476)
Department of Chemical Engineering
National Institute of Technology

Under the guidance of
Dr. Susmita Mishra

ACKNOWLEDGMENT

First and the foremost, I would like to offer my sincere gratitude to my supervisor, **Dr. Susmita Mishra** for her immense interest and enthusiasm on the project. She was always accessible and worked for hours with me. She was always kind and generous to me and lend me a helping at all stages of my project. She has been a constant source of encouragement for me.

I would also like to thank **Dr. RK Singh** and **Dr. HM Jena** for the wonderful support they gave to me. I would also like to appreciate the efforts put in by my friends during various stages of my thesis preparation. I would also like to thank all technical assistants in our department for their constant help.

Last but not the least, I wish to profoundly acknowledge my parents for their constant support.

Debesh Mishra

109ch0476

ABSTRACT

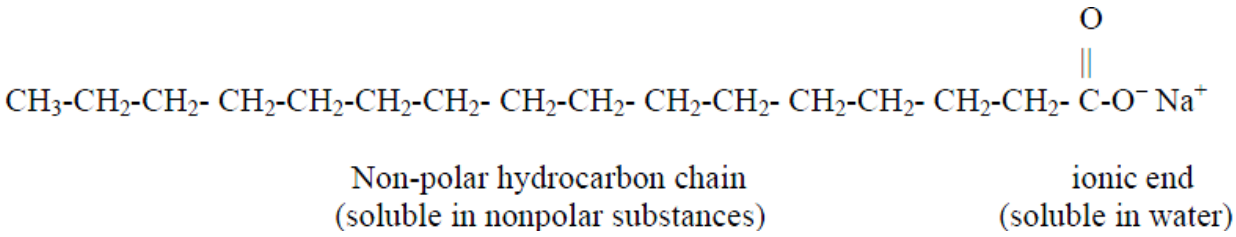
Soaps are the sodium salts or potassium salts of stearic acids or any other fatty acids. They are prepared by the saponification process, which is, reacting the oil which contain triglycerides with caustic soda (NaOH) to give the soap. However different oils have different composition of fatty acids which are responsible for different properties of soaps made out of them. In the present work 5 different types of oils are taken. They are blended in various ratios to prepare 14 different samples of soap. Different properties of these samples were analyzed to see which soap is the best one. The cleansing and lathering properties of all samples were compared. The blend of coconut oil and castor oil at 3:1 ratio is found out to be the best with 76.8% of TFM and 89.46% of yield. The best blend is analyzed for various properties and they were compared with that given in the literature. The saponification values, iodine values of coconut oil and castor oil were found out and these values were also found for the blend. It was found that the blend was having SAP value of 230.4 and iodine value of 40 which are higher than the individual values. Thus soap prepared using blend of both these oils has better properties than the soaps prepared by individual oils.

CONTENTS

List of Tables

1 Introduction:

A soap is a salt of a compound, known as a fatty acid. A soap molecule has a long hydrocarbon chain with a carboxylic acid group on one end, which has ionic bond with metal ion, usually sodium or potassium. The hydrocarbon end is non polar which is highly soluble in non polar substances and the ionic end is soluble in water. The structure of the soap molecule is represented below:



The cleaning action of soapsbecause of their ability to emulsify or disperse water-insoluble materials and hold them in the suspension of water. This ability is seen from the molecular structure of soaps. When soap is added to water that contains oil or other water-insoluble materials, the soap or detergent molecules surround the oil droplets. The oil is, dissolved in the alkyl groups of the soap molecules while the ionic end allows it to be dissolved in water. As a result, the oil droplets are to be dispersed throughout the water and can be washed away.

A number of things affect the soap-making process and the quality of this soap produced. The characteristics of this soap depend on the quality of oil, and the amounts of the caustic soda and water used to make it. The speed of the reaction between the oil and the caustic soda is influenced by free fatty acid content of the oil, the heat of the components before mixing, and how vigorously the mixing is to be done. Free fatty acid contents, vigorous mixing, and heat, speed up the given soap-making process.

Castile soap: a mild soap originally made in Spain with pure olive oil. Today many “castile” soaps are made with other vegetable oils. Castile is agood cleanser, producing a rich lather.

Cream soaps: soaps containing cold cream materials, and moisturizers. Cream soaps are good for dry and delicate skin

Deodorant soap are soaps to which antibacterial agents have been added to reduce odor-causing bacteria.

Floating soaps: soaps which have air bubbles incorporated have low density. This causes the bar to float.

Hypo-allergenic soaps: Mild formula soaps, low in potent irritants. They generally produce a poor lather.

Milled soaps: these are the most commonly used, mass produced soaps. Milling is referred to the mixing of colour and soap flakes.

Oatmeal soap: A rough textured soap to which oatmeal has been added as a mild abrasive and lather. Good for and normal skin.

A good soap is biodegradable when it does not contain chemicals that cannot be made to their natural elements. Neither does it contain chemicals that can be harmful to the environment or cause undue destruction to the environment.

- A good soap gets dissolved easily and remove stains from the clothes, human skin or any material being cleaned.
- It gets dissolved in water and produces enough suds.
- It gives a clear and sparkling kind of cleanliness.
- It gives a pleasant smell.
- A good soap does not leave sticky traces on the clothes or on the skin.
- It has a good color that is even and does not streak.
- It disinfects and kills germs.
- It does not damage the fibers or textiles.

2 LITERATURE REVIEW:

Oil used for analysis	Soap prepared and process followed	Reference
Neem oil	Toilet soap prepared using neem oil	E. E. Mak-Mensah et.al (2011)
Apricot kernel oil and palm stearin	Toilet soap prepared	Adel Y. Girgis et.al (1998)
Palm oil and castor oil	Soap prepared using different blends	ShogeMansuratOluwatoyin (2011)
Sheabutter oil and palm kernel oil	Soap prepared using different blends	Eke U. B. Dosumu et.al (2004)
Sheanut oil and groundnut oil	Soaps prepared using cold process synthesis	A.A. Warra et.al (2010)

Various attempts have been made to produce soap by first decomposing the fat or oil into fatty acids and glycerin, and then converting the acids into the soap by treatment with sodium or potassium carbonate. Three conventional methods of soap making are generally used:

- Semi boiling
- Full boiling
- Cold processed

Semi – boiling

The soft and hard oils or their blends are very suitable for this process in which the fat is first of all melted, followed by treatment with a weak 9-10% caustic soda solution followed by boiling of the mixture. The quantity of caustic soda required for the saponification of the oil is 14-15% of the weight of the oil. This weight of caustic soda is dissolved in ten times its weight of water to obtain a 9% solution. When the caustic solution is added into oil, then saponification starts when an emulsion is formed as the soap is stirred. More caustic solution is then added in to prevent the thickening of mass. After sufficient solution is added bit by bit to complete the saponification and the boiling of the mass continues until the soap was clear. During the boiling process moderate heat was maintained and each addition of caustic soda solution must be allowed to react with the oil before the next addition is made. A hurried addition in the initial stages of the process may retard the saponification, or at the final stages of the saponification may result in the drying of the soap, while judicious addition will keep the mass in a form of smooth homogeneous emulsion. If the soap shows any signs of separation and graining, further water is added to bring the mass to a homogeneous state. The ribbon test involves taking a small sample of the soap from the pan and cooling it. When a little quantity of this cooled soap is pressed in between the thumb and forefinger, the soap does not come out in the form of firm shiny ribbons with slight opaque ends and be clear when held against the light. If this cooled sample draws out in threads, there is excess water present in the soap, and more boiling is required to evaporate more water. If opaque ends appear and vanish, the soap is more oily and requires more caustic, while if the soap is graining, or turbid and white, it indicates a high level of presence of un reacted caustic, and requires more oil. A physical test - the taste test – is also done to determine the level of alkali. This test involves cooling a small quantity of the soap, and tasting it with the tip of the tongue. A sharp bite indicates too much caustic in the soap, while small bite indicates a high level of unsaponified fat or oil. A good soap gives a faint bite on the tongue.

After the completion of the boiling process, the fire is taken off, and the soap is allowed tocool with little stirring. At this point, perfume and colour can be added into the soap.

This process is not suitable for the production of toilet soap, can be used to produce laundry and all other types of soft and liquid soaps. The process does not permit the removal of waste alkali which contains the glycerine produced in the soap making process, and hence the glycerine, which tends to decrease the hardening property of the soap and improves the cosmetic property,

is retained in the finished soap. This method has some advantage over the other two since large quantities of good soap can be produced within a short time. The use of this method also allows a high percentage of fillers to be added in soaps, thus it increases the soap bulk.

2.1 Full Boiling:

The process consists of 4 stages:

- Saponification of the oil with alkali
- Graining out of the soap
- Boiling on strength
- Filling.

Saponification of the oil with alkali:

The process is started by putting the melted oil into the boiling tank and running a weak caustic soda solution into the oil. The mixture is then slowly boiled to start the saponification. The beginning of is denoted by the formation of emulsion. When saponification has started caustic soda of higher strength was frequently added in small quantities with continued boiling.Rapid addition of caustic alkali in the initial stages can also entirely delay saponification and in this case water should be added and the boiling continued till the excess alkali is taken up for the saponification to proceed. The end of saponification is determined by the 'ribbon' and 'taste' tests. When saponification is completed, the soap becomes very firm and dry with a permanent faint caustic like flavour on the tongue when cooled. The soap, which now consists of this imperfect soap together with water in which is dissolved glycerine and any slight excess of caustic soda, is then ready for graining out.

Graining out of the soap:

The objective of this is to separate the waste lye which is a mixture of glycerine produced during the soap boiling process and excess caustic soda solution from the soap. This is brought about by small use of common salt in dry form or as brine.

The term 'graining' is used here because after the introduction of the salt, the homogeneous soap gives the appearance of grains. The graining is complete when the soap is practically free from foam and floats as clean soap on the lye. At this stage, this sample of soap taken from the tank consists of distinct grains of soap and a liquid portion which is easily separated.

2.2 Cold Process

This process involves the treatment of fat or oil with a definite amount of alkali and no separation of waste lye. Although it is possible with lot care to produce neutral soap by this process the soap is very liable to contain both free alkali and unsaponified fat. The process is usually based on the fact that the glycerols of certain low fatty acids oils (nut oils like coconut and palm kernel oils) readily combines with strong caustic soda solutions at low temperatures, and generate little heat to complete the saponification reaction.

In this process, it is absolutelynecessary to use high grade raw materials. Oils and fats should be freed from excess acidity because caustic soda rapidly neutralizes the free fatty acids forming granules of soap which grain out in the presence of strong caustic solution, and since the grainy soap is very difficult to remove without heat increase, the soap tends to become thick and gritty and sometimes discolors. The caustic soda being used should also be pure,it must contain as little carbonate as possible, and the water must be soft and all other materials carefully freed from all particles of dirt.

The process involves stirring into the milled fat in a tank, half of its weight of caustic soda solution of at thetemperature of 24°C for coconut and 38°C to 49°C for the blend. The pushing of the caustic solution into the oil must be done not only slowly and continuously.

When the solution is being run into the oil, the mixture must bestirred in only one direction. When all the caustic soda solution had been run into the oil and the mixture stirred for 30 to 45 minutes, chemical reaction takes place with lot of generation of heat, finally resulting in the saponification of the oil. The content of the tank looks thin, but after some few hours it becomes a solid mass. The edges of the soap becomes more transparent as the process advances further, and when the transparency has extended to the full mass, the soap is ready, after perfuming to be poured into moulding boxes for hardening, cutting and stamping.

A little caustic potash solutionis used to blend the caustic soda solution which greatly improves the appearance of the given soap, making it smoother and milder.

Toilet soaps can be classified according to the method of manufacture into the following classes:

a) cold processed soap,

b) milled ,

c) remelted

The process consists of melting the fat in a pan and sieving out all impurities in it, after settling.

The oil is then run into the pan and cooled to 35°C. The right quantities of dye and perfume are then stirred intothe oil. Dyestuff was dissolved in a small quantity of water and filtered to avoid specks of color in the soap. For carbolic varieties, the cresylic acid is not added till after the saponification of the given oil. After adding the dye and the perfume to oil, the required quantity and strength of caustic soda solution is to be run into it in a thin stream with the constant stirring until the oil is completely saponified and the mass begins to become thick. Finally the thickened mass is drained out into soap moulding boxes and then allowed to harden slowly.

Different Types of Soaps:

Milled Toilet Soap making

Almost all the high class soaps used in the market passthrough the milling process which generally consists briefly of thegiven operations: drying of soap, mixing of perfume, milling, compressing, cutting and stamping.

After the solidifying in soap frames, this soap contains 28-30% of water, and this quantity is reduced by half before any satisfactory milling is done. Drying is best done by chipping the given soap into smaller sizes and exposing the chips in trays to a current of hot air at 35-40°C. There are several forms of drying chambers in which the chips in the trays are placed upon a series of racks, one above the other and warm air circulated through.

It is very important that the correct amount of moisture should be left in the soap, not too much or little - the exact point can be determined only by judgement and experience, and depends on the nature of the soap to be made and the quantity of perfume to be added. A range of ll-14% moisture is preferred. Below this range, the soap will crumble during the milling process and the finished soap will have the tendency to crack, while above the range, the soap will stick to the rollers of the milling machine, and mill only with difficulty.

Mixing of Perfume and Dye

When the soap chips have already been dried to attain the required water content, they are put into the amalgamator which is the mixing machine, and the required amount of preferred perfume and dye are added to mix thoroughly at room temperature.

The quantity of the perfume to be added varies considerably with the perfume type used. For cheap grade soaps are used, while for costly soaps is sometimes used.

Milling

From the amalgamator, the soap is put into the milling machine for the chips have to be milled into homogeneous thinsoap ribbons. Milling does not improve the quality ofthe soap but only gives a semi-transparent appearance to it.

2.4 Different types of soap making oils

Fats and oils are esters of different fatty acids and glycerol.Fats and oils are divided into three classes, fixed oils, mineral oils and essential oils. Fixed oils form the main raw materials for soap making as they decompose into fatty acids and glycerol when strongly heated, and can be easily saponified by alkali. Fixed oils, which include both animal and vegetable fats and oils, are further classified according to its physical properties as follows [13]:

a) Nut oils: These oils are characterized to be having large proportion of fatty acids with low molecular weight, especially lauric and stearic acid. Examples of these oils are coconut oil. These oils, when used in toilet soaps are the chief foam-producing ingredients.

They usually saponify easily with strong alkali solution. Once these oils have begun to saponify, the process proceeds rapidly with the evolution of heat. They require very large quantities of strong brine (1648”Be) to grain their soaps, and the grained soaps tend to carry more salt than other soaps. These oils are more suitable for the making of cold process soaps.

b) Hard fats: The hard fats contain appreciable quantities of palmatic and stearic acids. Examples of these oils are palm oil, animal tallow and hydrogenated fats. These oils produce slow-lathering soaps but the lather produced is more resistant over long periods of time than the nut oils. In soap making, they are first saponified with weak alkali, and in the final stages with stronger alkali solutions.

c) Soft Oils: These oils have substantial amounts of unsaturated acids, namely oleic, linoleic and linoleneic acids. The soap making properties of these oils vary with their fatty acid composition, and their physical and chemical properties of the acids. Examples of these kind oils are groundnut, cotton seed, fish oil and olive oil. These oils cannot produce a very hard soap when

used alone for soap making. They are generally blended with nut oils. Their soaps lather freely and have very good detergent properties.

Soap making involves a definite chemical decomposition of fats and oils into their constituent parts, like fatty acids and glycerol. The fatty acids combine with little caustic soda, potash or other base forming soap, and glycerol remains free.

All fats and oils used in soap making consist of a mixture of compounds of glycerol with fatty acid which occur in nature in the form of triglycerides. The most important of these acids from the soap maker's point of view are stearin, palmitin, olein and laurin. The presence of stearin and palmitin, which are generally solids at room temperature, gives firmness. The greater the percentage present the harder the oil, and the higher its melting point. Where olein is liquid at ordinary temperature, is the chief constituent, the oil is soft.

The soap making properties of fats and oils can be determined by the molecular weights of their fatty acids. With increasing the molecular weight in the case of naturally occurring saturated fatty acids in fat or oil, the following properties are found [13]:

.

The properties of their corresponding sodium soaps vary as follows with increasing molecular weight:

- The solubility increases,
- The lathering properties improve up to lauric acid and deteriorate from lauric acid upwards,
- The stability of the lather increases,
- The detergent action decreases,
- The soaps have milder skin action as the series progresses,
- The property of holding filling solutions such as sodium silicate decreases.

This explains the reason why nut oil (such as coconut oil) soaps lather readily and profusely but not stably. They also have a firm texture and are hard but dissolve more readily in water than do soaps from the hard oils. They can also retain a good amount of water, and take up large quantities of fillers like sodium carbonate.

2.5 Castor Oil:

The oil is obtained from extracting or expressing the seed of a plant which has the botanical name *Ricinuscommunis* [7]. The oil is not only a naturally-occurring resource, it is inexpensive and environmentally friendly. Castor oil is viscous, yellow, non-volatile and non-drying oil with a bland taste and is sometimes used as a purgative. It has slightest characteristic odour while the crude oil tastes slightly acrid with a nauseating after-taste. Relative to other given vegetable oils, it has a good shelf life and it does not turn rancid unless subjected to excessive heat. India is the world's largest exporter of castor oil.

The extraction of oil from castor seed is by one or a combination of mechanical pressing and solvent extraction. In this process of mechanical pressing, the seeds are crushed and then adjusted to low moisture content by warming in a steam-jacketed vessel. Thereafter, the crushable seeds are loaded into hydraulic presses and they are pressed by mechanical means to extract oil. The oil coming from mechanical pressing has light colour and low free fatty acids [7]. However, mechanical pressing will only remove about 45% of the oil present and the remaining oil in the cake can be recovered only by solvent extraction. In the solvent extraction method, the crushed seeds are extracted with a solvent in a Soxhlet extractor or commercial extractor. Solvents generally used for extraction include heptane, hexane and petroleum ethers.

As in other vegetable oils, it is usual to refine the crude oil obtained from either mechanical pressing or solvent extraction. The main aim of this refining is to remove impurities (e.g., colloidal matter, free fatty acid, coloring matter) and other undesirable constituents, thus making the oil more resistant to deterioration during storage. Castor oil, like all other vegetable oils, have different physical and chemical properties that vary with the method of extraction. Cold-pressed castor oil has low a acid value, low iodine value and a little higher saponification value than the solvent-extracted oil, and it is little lighter in colour.

Castor oil-based synthetic detergents are very less prone to foaming and the disposal of the detergent is hastened since microbiological breakdown is simplified.

Fatty acid composition of this kind of castor oil is:

- Ricinoleic 90%
- Linoleic 3-4%
- Oleic 3-4%

Castor in soaps contributes to fluffy, stable lather, conditioning, moisturizing, quicker trace, softer soap. It is often used to superfat soaps. Castor oil should be used at low percentages to avoid overly soft soaps. Also often used in balms, soaps, shampoos, hair oils, and other thick emulsions for the skin and hair.

2.6 Olive Oil:

Olive oil is one of the most common base oils used in soap making today. 100% olive oil soap, or "Castile" soap has been made for centuries – and today, soap makers of all types usually include at least some olive oil in their blends.

Olives are a type of fruit called a drupe which is basically a type of fleshy fruit that has one hard seed at the center. First, the olives are generally crushed and ground into a paste. Then, the oil is to be separated from the paste by various methods. The first oil that has come from the very first crush is the "virgin" olive oil. The paste that is left behind after the first extraction is called "pomace." Fatty acid composition of Olive oil:

- Oleic 63-81%
- Palmitic 7-14%
- Linoleic 5-15%
- Stearic 3-5%

Olive oil contributes to soap hardness, stable lather, slippery feel, conditioning, moisturizing. Olive Oil attracts external moisture to your skin, helping to keep skin soft and supple.

Pomace olive oil contains a larger proportion of unsaponifiable ingredients. This slightly affects its SAP value and imparts a greenish color to the oil and to soaps made with it. Pomace oil is preferred to grade A olive oil for soapmaking.

2.7 Neem Oil:

Neem oil is obtained from the seeds of the neem tree. the oil is greenish yellow, non-drying with an acrid and bitter taste, and an unpleasant garlic odour. The oil is extensively used to blend other oils in the making of both laundry and toilet soaps in India. The oil saponifies readily and gives a hard-grained soap with good and very stable lather. When used alone for the making of

soap it is very necessary to grain the soap as this helps to remove most of the disagreeable odour and colour. On the other hand, if it is used to make soap with other oils, it is advisable to first make neem oil soap. After the soap has been grained, the other oils are stirred into the soap and the required amount of caustic soda solution added to start the saponification again. Neem oil soap is used for both laundry and antiseptic purposes. Neem oil has been used in the manufacture of natural cosmetics, soap, toothpaste, hair and skin products, emulsions, liquors, ointments and medicinal cosmetics [5]. However neem oil can be produced mechanically (hot or cold press) or chemically (solvent extraction) from dried neem seeds. The best quality neem oil with a majority of phytoconstituents intact is obtained through cold press. In cold press the oil is lighter in colour and has a milder odour [6]. Moreover potential residual solvents in chemical extracted oil that may pose health hazards to consumers are eliminated since solvents are not used in the pressing techniques.

Neem oil is rich in essential fatty acids (EFAs), triglycerides, vitamin E and calcium. Because of its EFAs and vitamin D, neem oil penetrates deep within the skin to heal the minute cracks brought on by severe dryness. Fatty acids present in neem kernel oil are

- oleic acid (52.8%),
- linoleic acid (2.1%),
- palmitic acid (12.6%) and
- stearic acid (21.4%) and
- other lower fatty acids (2.3%) [8] and
- linolenic acid (1%) [17].

Acid value of neem oil is <20.0, [7]. Neem also stimulates collagen production, good for aging skin. Vitamin E acts as a free radical scavenger, by hindering the oxidizing processes in the skin. It promotes soft and supple skin, helps in reducing old scars and promotes healing. The neem soap will also be slightly antimicrobial. The neem soap will be acceptable to people suffering from skin diseases such as psoriasis and eczema who are allergic to soaps containing Diethanolamine, Isopropyl alcohol, Butylated toluene and Triclosan additives [8].

Neem Oil contributes to stable lather , conditioning. It is said to have the ability to treat a variety of skin disorders such as dandruff.

2.8 Coconut Oil

On an industrial scale, the dry process is the traditional method of extracting oil from the coconut [9]. This is done by crushing copra in an expeller, the trade name of the machine patented by V. D. Anderson. The meal may be further treated with solvents to extract residual oil. The dry process involves mechanical extraction of oil in crushers or expellers with copra as feedstock.

The wet-process feedstock is fresh kernel instead of copra. The extracted oil does not have to be refined, unlike the oil from copra. The co products of oil from the wet process are edible. In addition to oil, other edible co products are recovered from the kernel, namely, coconut flour, protein, carbohydrates, and vitamins. To encourage wider commercial application of this process, the following advantages are emphasized: superior quality of the oil product and the recovery of nutrient co products that would otherwise be lost in copra.

Refining:

Refining of crude fats and oils involves a series of steps for the removal of impurities from the glycerides to make the product suitable for human consumption and improve product shelf life. The impurities are generally fatty acids, phosphatides, metal ions, color bodies, oxidation products, solid particles, and volatiles that include objectionableodour. Crude coconut oil is refined by any of the following methods: (1) chemicalrefining (batch or continuous) and (2) physical refining.

Physical Refining

Coconut oil refiners have gained interest in the physical refining system as a substitute

for chemical refining for the following reasons: (1) physical refining has lower oil losses vis-a´-vis chemical refining; (2) pollution problems associated with soap stock acidulation is precluded; (3) lower installation cost; (4) lower steam, water, and power consumption; and (5) distilled fatty acids are of a higher grade than the acid oil from chemical refining

Coconut oil composition

Coconut oil belongs to unique group of vegetable oils called lauric oils. The most abundant fatty acid in this group is lauric acid, $CH_3(CH_2)_{10}COOH$. Other sources of lauric oils are palm kernel, cohune, and cuphea.

Table1. Composition of coconut oil[10]

Fatty Acid	Range Weight (Mean)	%
Caproic C6	0.4–0.6	(0.5)
Caprylic C8	6.9–9.4	(7.8)
Capric C10	6.2–7.8	(6.7)
Lauric C12	45.9–50.3	(47.5)
Myristic C14	16.8–19.2	(18.1)
Palmitic C16	7.7–9.7	(8.8)
Stearic C18	2.3–3.2	(2.6)
Oleic C18 : 1	5.4–7.4	(6.2)
Linoleic C18 : 2	1.3–2.1	(1.6)
C20	*t*–0.2	(0.1)
C20 : 1	*t*–0.2	(*t*)

More than 90% of the fatty acids of coconut oil are saturated. This accounts for its low iodine

value ranging from 7 to 12. The saturated character of the oil imparts a strong resistance to oxidative rancidity. Assessment

of the oil by active oxygen method (AOM) yielded results between 30 h and 250 h [10]. Although oxidative stability is reduced in some RBD oils, due to losses in the natural antioxidants of crude coconut oils, the addition of citric acid at the end of deodorization as the oil is cooled to 100^0C was effective in regaining considerable oxidative stability in the oil [11].

3 Materials and Method:

All the five varieties of soap making oils were bought from the local market.

Type of oil	Brand name	Cost Rupees/100ml
Coconut oil	Shalimar	35
Olive oil	Leonardo	95
Neem oil	Plasma	45
Karanjia oil	Local	30
Castor oil	Dabur	40

Glasswares required for the experiments were acquired from local suppliers:

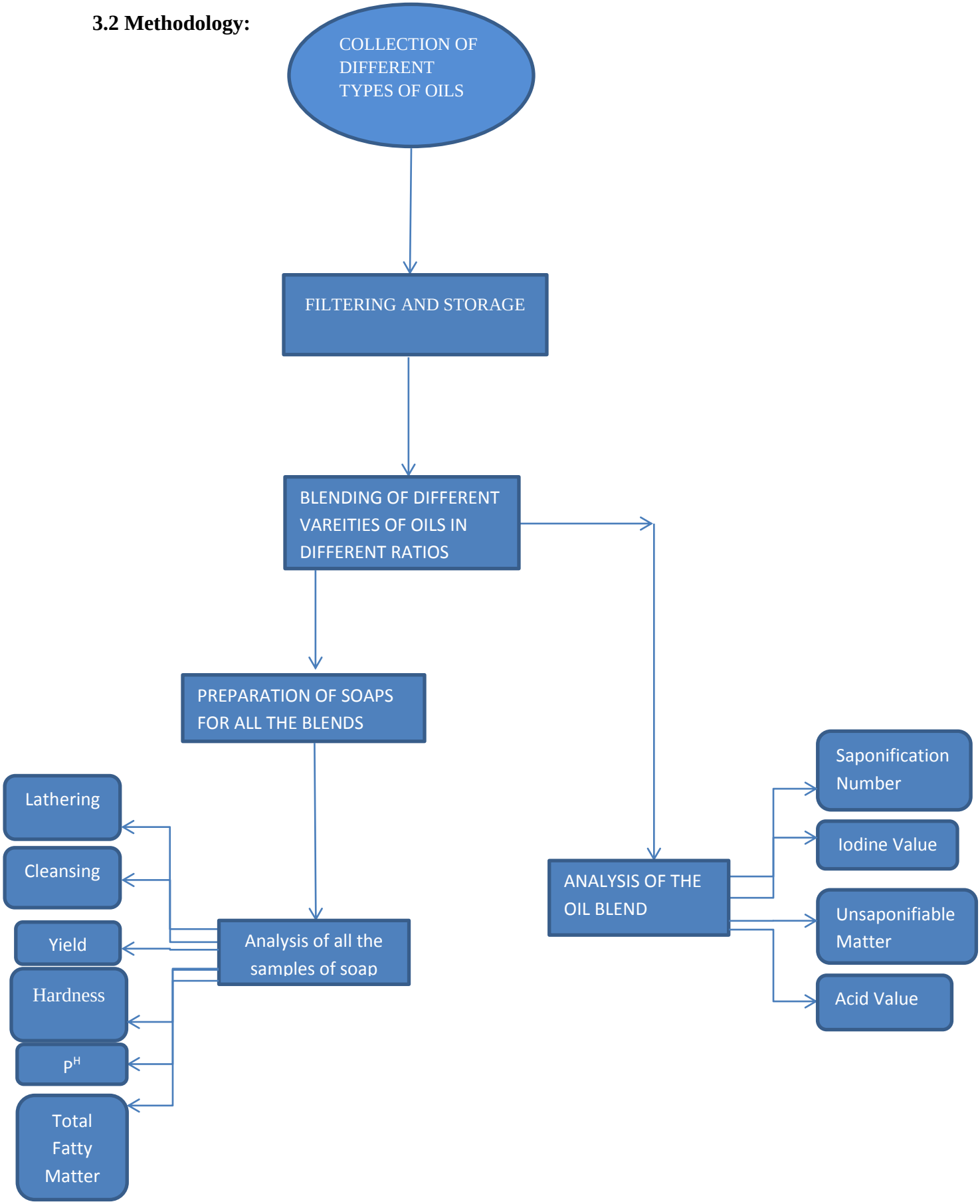
Type of glassware	Brand
Beaker	Borosil
Round bottomed flask	Borosil
Conical flask	Borosil
Pipette	Borosil
Burette	Borosil
Stirrer	Borosil

Test tubes	Borosil
Reflux condenser	Borosil

Name of all the reagents used:

- Caustic soda
- Potassium Iodide
- Sodium chloride
- Methanol
- Sodium sulphate
- Ethanol
- Sodium carbonate
- Starch
- Sodium silicate
- Chloroform
- Nitric acid
- Phenolpthalein
- Sulphuric acid
- Diethyl Ether
- Calcium carbonate
- Sodium Thiosulphate
- Barium chloride
- Potassium Hydroxide
- Iodine monochloride

3.2 Methodology:



3.2.1 PREPARATION OF SOAP:

10 g of the oil was measured into a plastic container. It was warmed in order to quicken the reaction between the alkali and the fat. A calculated amount of NaOHwas weighed and a fixed amount of distilled water was added to it to prepare a 0.2 N NaOH solution. The caustic soda was stirred well using a stirring rod until it blends with the fat. The caustic soda was poured very gradually into it and stirred gently in one direction to enhance thorough mixing of the solution. The plastic container was insulated with pieces of cloths to prevent the fat from hardening before the soap mix properly. Small amount of sodium carbonate, sodium sulphate and sodium silicate was added into the soap mixture and it was stirred properly until it blends. The heating is done to 110^0 C in a heating tub.Sodium sulphate is added during the soap is clarified but in the molten stage. It helps in the binding of the soap chemicals and it induces the foaming ability of the soap. It is equally a binder and an extender.

Sodium silicate is added when the soap is being cooled down. Sodium silicate hardens the soap. It eases removal of dirt and preventsre deposition of dirt particles. To check whether the saponification is completed or no 'ribbon test' was performed. In this test a small sample of the soap from the beaker was taken and cooled. When a little quantity of the cooled soap is pressed between the thumb and forefinger, the soap should come out in the form of firm shiny ribbons with slight opaque ends and be clear when held against the light. If the cooled sample draws out in threads, there is excess water present in the soap, and further boiling is required to evaporate more water.

If the opaque ends appear and vanish, the soap is oily and requires more caustic, while if the soap is grainy, or turbid and somewhat white, it indicates a high level of unreacted caustic, and requires more oil. A physical test - the taste test – was also done to determine the level of caustic. After this the soap was mold into a proper shape and kept in a filter paper. This soap was taken in air oven to dry up the moisture. Before tken in the oven the weight of soap was taken. Soap is kept for 12 hrs in the oven maintained at 300-350^0C. The weight of the soap is again taken.

The above method is followed to prepare soap using all five different types of oils.

Calculation of Yield:

For all the soap samples prepared using different soap making oils, weight is taken after they are taken out of the air oven. Yield is calculated by dividing the weight of the soap by the weight of the oil taken multiplied by 100. It is calculated for all the samples.

3.2.2 Comparision of properties soaps and detergents:

Alkalinity:

- 1% of soap solutioncis prepared by dissolving about 0.5 g of the soap in 50 mL
- of deionized water. It may help to heat the water to get the soap to dissolve completely.
- Using a pH meter the alkalinity of the soap solution is determined. The electrode of the pH meter is dipped inside the soap solution. The pH value of the solution is recorded. This is carried out for all the cases.
- 5 mL of local detergent solution was added to a beaker or test tube. this solution was compared with the soap solution for the remaining tests.
- alkalinity of the detergent solution was calculated using a pH meter

Lathering Power

- 2 mL of deionized water was added to four large test tubes.
- An equal amount of soap solution was added to one test tube of water and shaken vigorously by placing a stopper in the tube. This should give a permanent lather that lasts for at least 30sec. If the lather doesn't last, add another 10 drops of soap solution was added and shaken vigorously.
- An equal amount of detergent solution was added to another test tube of water and shaken vigorously. This should give a permanent lather. If not, add another 10 drops and shake vigorously.
- 2 mL of 5% calcium chloride solution to each of the two remaining test tubes of water was added.
- An equal amount of soap solution to one of the tubes containing calcium ion was added and shaken vigorously. It was observed whether this solution forms a permanent lather and it was noted whether there is any floculent precipitate in the tube. Does the precipitate float or sink?

- An equal amount of detergent solution to the was added to the other tube containing Ca2+ ion and shaken vigorously to get a permanent lather. Does the lathering power of the detergent differ from the soap when Ca2+ is present? Is there any precipitate in this solution?

Cleansing Power:

- A drop of used brake oil, was placed on four separate thin strips of filter paper. It is made sure that the strips of filter paper will fit in the test tubes used in the previous step.
- One filter paper with oil spot in the tube containing soap in water. Another is placed in the tube containing detergent and water. a third strip is placed in the tube containing soap in calcium solution. the fourth strip of oily paper was placed in the tube containing detergent and calcium solution. Each one is shaken well and made sure that the filter paper is immersed in the solution.
- After 2 min the filter paper was removed and rinsed with tap water. Did the oil get washed out of the filter paper strip? the solutions were thrown in the sink. the paper strips were thrown in the trash can. The cleaning power of soap versus detergent was compared.

This reaction was carried out for all the five samples prepared.

Moisture content:

A sample of the 10g scrapped soap was put into a petri dish and placed in an oven for 1 hour at 110°C. It was allowed to cool down and then weighed. The moisture content in percentage was calculated.

Hardness:

The hand felt hardness was determined relatively to each other for all the soap samples.

The properties of the soap were tabulated and compared with each other. But the primary objective of this work is to blend different varieties of oils in different ratios and prepare soap samples and compare their properties.

3.2.3 Blending:

All the five varieties of oils were taken and different blends were prepared taking 2 oils at a time. The blended oil samples used for saponification, were prepared by varying the weight of any two of soap making oils taken. Combined weight of the mixture was kept constant at 10 g.

The same procedure was followed as mentioned in section to prepare 14 different samples of soap. For all samples of soap the properties like

(a) Yield,

(b) P^H,

(c) Lathering power,

(d) Cleansing power,

(e) Total Fatty Matter

(f) Free Alkali Content

(g) Moisture

(h) Hardness

were calculated to determine the best blend of oils for soap malking.

3.2.4 Total Fatty Matter:

- Accurately weighed 5 gm of soap and transferred into 250 ml beaker.
- To completely dissolve the soap 100 ml hot water was added.40 ml of 0.5 N HNO3 was added to the mixture until contents were slightly acidic.
- The mixture was heated over water bath until the fatty acids were floating as a layer above the solution.
- Then the mixture is cooled suddenly in ice water in order to solidify the fatty acids and separate them.
- 50 ml of chloroform was added to the remaining solution and transferred into a separating funnel.
- The solution is shaken and allowed theto separate into 2 layers and the bottom layer was drained out.
- 50 ml of chloroform was added to the remaining solution in the separating funnel.
- The fatty acid dissolved chloroform is again separated as in the previous case and it is transferred to the collected fatty matter.

- The fatty matter was weighed in a previously weighed porcelain dish.
- From the difference in weight, the % of fatty matter was calculated in the given soap sample.

3.2.5 Free Alkali Content:

- A sample of the scrapped soap *(10* g) was placed in a conical flask and
- *100* em3of neutralized alcohol was added.
- The flask and the content therein was placed on a water bath and heated until the soap dissolved.
- The 10 cm3 of 10% Barium chloride solution was added
- 2 to 3 drops of phenolphthalein indicator were added.
- The whole content was titrated against *O.1N* H2SO4until the solution became colourless.
- The free alkali as Na20 was then' calculated.

After finding out all these parameters for all the 14 samples, and Total Fatty Matter content for the best blends. Finding out the best blend out of all, the important properties of the oils affecting its soap making characteristics were thoroughly analyzed.

3.2.6 The properties analyzed for the best soap making blend are:

- Saponification Number
- Iodine Value
- INS Value
- Acid Value
- Unsaponifiable Matter

3.2.7 Saponification Number:

- 1g of fat is weighed in a tared beaker and dissolved in about 3ml of ethanol.
- Three times the contents of the beaker were washed with 7 ml of the solvent and transferred to a round bottomed flask.
- 25ml of 0.5N alcoholic KOH was added and mixed well. The 0.5N alcoholic KOH was prepared by adding 27 g of KOH in as much methanol as required to dissolve it and rest of 500ml was compensated with distilled water

- The round bottomed flask was attached to the reflux condenser.
- Another reflux condenser is setup with the blank with all other reagents present except the oil.
- Both the flasks were placed on a boiling water bath for 30 minutes.
- The flasks were cooled to room temperature.
- Phenolphthalein indicator was added to both the flasks and titrated with 0.5N HCl.
- The endpoint of blank and test flasks were noted down.
- The difference between the blank and test reading gives the number of millilitres of 0.5N KOH required to saponify 1g of fat.
- The saponification value using the formula :

  Saponification value or number of fat = mg of KOH consumed by 1g of fat.

  - Weight of KOH = Normality of KOH * Equivalent weight* volume of KOH in litres
  - Volume of KOH consumed by 1g fat = [Blank – test]ml

3.2.8 Iodine Value:

- 10ml of oil sample dissolved in chloroform pipette out to an iodination flask labeled as “TEST".
- 20ml of Iodine Mono chloride reagent was added in to the flask. the contents in the flask were thoroughly mixed.
- Then the flask was allowed to stand for half hour incubation in dark.
- A BLANK was set up in another iodination flask by adding 10ml Chloroform to the flask.
- 20ml of Iodine Mono chloride reagent added to the BLANK and the contents were mixed in the flask thoroughly.
- The BLANK was incubated in dark for 30 minutes.
- TEST was taken out from incubation after 30 minutes and 10 ml of potassium iodide solution was added into the flask.
- The stopper and the sides of the flask were rinsed using 50 ml of distilled water.

- "TEST" was titrated against standardized sodium thio sulphate solution until a pale straw colour is observed.
- 1ml starch indicator was added into the contents in the flask, a purple color was observed.
- The titration was continued until the color of the solution in the flask turns colorless.
- The disappearance of the blue colour was recorded as the end point of the titration.
- The procedure is repeated for the flask labeled 'Blank'.
- The endpoint values of the BLANK were recorded.
- Calculate the iodine number using the equation below:
  Volume of Sodium thiosulphate used= [Blank- Test] ml

$$\text{Iodine Number} = \frac{\text{Equivalent weight of Iodine} * \text{Volume of } Na_2S_2O_3 * \text{Normality of } Na_2S_2O_3}{\text{Weight of Oil taken for analysis} * 10}$$

3.2.9 Unsaponified Matter:

- A sample of the oil (5 g) was refluxed with 50 cm3 of 0.1N alcoholic potassium hydroxide' solution on a water bath for about an hour. This 0.1N alcoholic KOH was prepared in a similar way as mentioned above.
- When saponitication was completed, the content of the flask was transferred to a separating funnel and the flask was washed with 50 cm3 of distilled water into the separating funnel.
- Then 50 cm3 diethyl ether was used to extract the water insoluble matter (unsaponified matter).
- This extraction was repeated and the average was calculated.

3.2.10 Acid value:

- 50 cm3 of alcohol was boiled on water bath for few minutes.
- 2 cm3 of phenolphthalein and 0.1N NaOH solution were added to produce a permanent pale pink colour. Then 5 g of oil was added to 50 cm3 of this neutralized solution and the mixture boiled on a water bath.
- While still hot the solution was titrated against 0.25N NaOH until the pink color returns.

- Acid value = (Volume of KOH x Normality of KOH x Eq. wt x 1000) / Weight of Oil sample.

4 Results and Discussions:

4.1 Comparison between soap and detergent:

Table2. Yield of soap using individual oils

Type of soap making oil used	Weight of soap before drying	Weight of soap after drying	Yield (%)
Coconut oil	9.8 g	9.2 g	92.0
Olive oil	9.4 g	8.77g	87.7
Neem oil	9.1 g	8.23 g	82.3
Karanjia oil	8.9 g	8.47 g	84.7
Castor oil	8.8 g	8.13 g	81.3

The yield of soap depends on the soap making oil used. This also depends on the particular carboxylic acid and base that make up the soap. Higher the yield is more economical is the process of soap making.

Table3. Properties of soap versus detergent

Type of soap making oil used	pH of the solutions prepared	Lathering power	Cleansing power
Coconut oil	9.5	High	High
Olive oil	8.75	Good	High
Neem oil	9.75	Good	Good
Karanjia oil	10.2	Poor	Poor
Castor oil	9.2	Good	Good
Detergent	11.8	High	High

The cleansing power and lather produced by different soap can be explained based on the fatty acids composition of oil used in soap formulation. It has been found out that, lauric acid and myristicacid, which are all saturated fatty acids produces soap with fluffy lather and high cleansing power [4]. However, the observed difference in the cleansing power and nature of lather formed in the soap formulation may be due to the method use in the soap preparation in addition the nature of fatty acid composition of the fat or oil. Here the castor oil, coconut oil, olive are found to have both high cleansing and lathering power.

Table4: Moisture and Hardness of soap samples from individual oils

Type of soap making oil used	Moisture content (%)	Hardness
Coconut oil	3.43	Very hard
Olive oil	4.5	Soft
Neem oil	2.8	Hard
Karanjia oil	3.2	Hard

Castor oil	3.8	Soft

4.2 Analysis of soap samples prepared by blending of different varieties of oils in different ratios:

Table5: Yield and pH values of soap samples from blending of oils

Blend of soap making oil used	Ratio of oils in the blend	Yield(In percentage)	pH values
Coconut oil+ Karanjia oil	4:1	89.21	9.73
	3:1	87.56	9.92
	2:1	89.34	9.95
Coconut oil+ Castor oil	3:1	89.46	9.46
	3:2	89.38	9.70
	1:1	88.23	9.52
Olive oil + Neem oil	3:2	86.66	9.30
	1:2	87.34	9.30
	1:1	85.32	9.54
Olive oil + Castor oil	3:1	89.36	9.46
	3:2	88.38	9.48
	1:1	88.13	9.61
Coconut oil + Olive oil	2:1	85.46	9.13
	3:1	89.67	9.38

Table6: Lathering and cleansing power of soap samples from blending of oils

Blend of soap making oil used	Ratio of oils in the blend	Lathering power	Cleansing power
Coconut oil+ Karanjia oil	4:1	Good	Good
	3:1	Good	Good
	2:1	Poor	Poor
Coconut oil+ Castor oil	3:1	High	High
	3:2	High	High
	1:1	Good	High
Olive oil + Neem oil	3:2	Good	Good
	1:2	Poor	Good
	1:1	Poor	Good
Olive oil + Castor oil	3:1	Good	High
	3:2	Good	Good
	1:1	Good	Good
Coconut oil + Olive oil	2:1	Good	High
	3:1	Good	High

Table7: Moisture and Hardness of soap samples from blending of oils

Blend of soap making oil used	Ratio of oils in the blend	Moisture (%)	Hardness
Coconut oil+	4:1	4.20	Hard

Karanjia oil	3:1	3.9	Hard
	2:1	4.25	Hard
Coconut oil+ Castor oil	3:1	4.3	Hard
	3:2	4.2	Hard
	1:1	4.3	Hard
Olive oil + Neem oil	3:2	3.9	Soft
	1:2	3.8	Not very hard
	1:1	4.0	Hard
Olive oil + Castor oil	3:1	4.1	Soft
	3:2	4.0	Soft
	1:1	4.2	Hard
Coconut oil + Olive oil	2:1	3.4	Did not bind
	3:1	4.3	Hard

This table shows the hardness and moisture content of all the blends. Blend of castor oil which is a soft oil and coconut oil which a nut oil produces a very hard soap. This is the benefit of blending of which brings in the characteristics of both oils enhancing the property of the soap produced from the blend of both the oils. The soap produced from other oil blends like that of Olive oil and Castor oil, or Olive oil and Neem oil form soft soaps. The moisture affects the lathering and cleansing property of the soaps. However this moisture is reduced with passage of time.

4.3 Table8: **Total fatty matter of best blends of oils**

Oil blend selected	Mass of soap taken	Mass of fatty matter in g	Total Fatty Matter (TFM) in %
Coconut + Karanjia	5 g	3.32g	66.4
Coconut + Castor	5 g	3.84g	76.8
Olive + Neem	5 g	3.6g	72
Olive + Castor	5 g	3.5g	70

Soaps are graded in terms of total fatty matter or TFM. TFM or total fatty matter is a measure for identifying the amount of fatty matter present in soaps. The TFM measures the quality of soap and the accepted percentage value for toilet soap is between 76-77% while that of laundry soap is between 45-50%. The best blend is selected mostly on the basis of TFM. For oil blend of coconut oil + castor oil (3:2) the TFM is the highest at 76.8 which falls in the range of TFM required for toilet soap. TFM is what lends soap its soapy feel and it is the TFM and the insoluble matter in the soap that largely distinguishes soap from the others. Other soap blends also have appreciable TFM content with Olive oil and Neem oil with 72% and also can used for toilet soaps. The worst is of Coconut oil and Karanjia oil with 66.4% which makes it fit for laundry soaps .

4.5 Analysis of the best blend of oil

After analysis of all the blends of the oils and soaps produced from the blends it was affirmed that blend of coconut oil and castor oil in the ratio of 3:1 is the best one. Thus we explore into the properties of this blend of oil which affect its soap making characteristics and see whether it is agreeable.

4.6 Saponification Value:

Table9: saponification value of the oil blend

Oil or blend	Saponification number
Coconut oil	268
Castor oil	180.3
Coconut+ Castor(3:1)	230.4

It gives information concerning the character of the fatty acids of the fat- the longer the carbon chain, the less acid is liberated per gram of fat hydrolysed. It is also considered as a measure of the average molecular weight (or chain length) of all the fatty acids present. The long chain fatty acids found in fats have low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat and therefore high molecular weight. Oils with high saponificaton values such as coconut oil (257.0) and palm oil (199.1) are better used in soap making [1]. Soap manufacturers blend their oils with coconut oil because of its high saponification value. When it is blended with castor oil then saponification number of the blend is higher than castor oil which certify the results in the above tables of better quality soaps.

4.7 Iodine Value:

Iodine value or number is the number of grams of iodine consumed by 100g of fat. A higher iodine value indicates a higher degree of unsaturation.

Oil or blend	Iodine value

Coconut oil	10
Castor oil	68
Coconut+ Castor(3:1)	40

Table10: Iodine Values of the oil blend

High iodine value justifies utilization of the oil in soap and shampoo productions [2] Castor oil is an example of nondrying oils whose iodine numbers are less than 100 [3] they have the advantage of not undergoing oxidation to form a film, hence are useful in the manufacture of soaps [3]. The coconut oil has a very low iodine value because of the saturated fatty acids present. The blend has a moderately high iodine value which makes it suitable for soap making but does not make a soft soap because of the presence of coconut oil.The higher the number for an oil, the greater the percentage of these acids, and thus the softer the soap produced from the oil. The soft oils have high iodine numbers and are readily oxidized. The iodine number thus indicates the hardness of the soap, the lower the number, the harder the soap produced.The variation in colours is due to the degree of unsaturation of the fatty acids. Increase in double bonds causes increase
in intensity of colour

4.8 Unsaponifiable Matter:

Table11: Unsaponifiable matter of the oil blend

Oil or blend	Unsaponifiablematter(%)

Coconut oil	0.2
Castor oil	0.7
Coconut+ Castor(3:1)	1.3

Here the unsaponifiable matter of both castor oil, coconut oil and there is very low.
These oils can be used for saponification without refining, although. phytosterol, cinnamic acid, karitene that are responsible for high values ofunsaponified matter can be removed by boiling in water and ethanol.

4.9 Acid Value:

Acid value indicates the proportion of free fatty acid present in an oil or fat and may be defined as the number of milligrams of caustic potash required to neutralize the acid in 1 g of the sample.A high acid value indicates a stale oil or fat stored under improper conditions.

Table12: Acid value of the oil blend

Oil or blend	Acid Value
Coconut oil	1.68
Castor oil	2.42
Coconut+ Castor(3:1)	1.92

Acid value of both the individual oils and the blend confirms for the minimum purity to get yield of better quality soaps.

Free alkali content in the soap is found to be 4.5% because this in its very crude form and has not undergone any refining procedure.

5 CONCLUSION:

Soap was prepared using all varieties of oils including that of all the various blends of oils.

The soap was tested for various properties and compared with detergents. One soap making oil in itself does not have all the properties. Therefore blends of oils are prepared taking 2 oils together because it would be easier for analysis. All necessary properties like Lathering power, Cleansing power, pH, Hardness, Total Fatty Matter, Moisture, Yield were all studied to select the best blend out of all the blends. The best blend was found out to be coconut oil and castor oil (3:1). Its TFM value was found to be 76.8% which lies in the range of toilet soaps. It had the maximum yield out of all soaps with 89.46%. It had excellent lathering as well as cleansing power. As coconut oil is a nut oil, therefore soap prepared with this blend was very hard. Analysis of this blend confirms to that given in the literature and the results of the above analysis. Saponification number of this blend of oil was found to be 230.4 and the iodine number was found to be 40. Both high saponification number and iodine number indicates this blend to be highly preferred for soap making. The soap prepared is not affected by high iodine number as the soap prepared is very hard. The acid value found that is 1.3, was also acceptable according to the literature. The unsaponifiable matter is also within the limits and oil blend can be used without being refined.

6. References:

1. Abayeh,O.J,Aina,E.A and Okuonghae*Oil content and oil quality characteristics of some Nigerian oil seeds*. Journal of pureand Applied sciences; 17- 23(1998).

2 .Hassan, L.G, Osoba, O, Umar, K.J and Inuwa M*Physico chemical Analysis of Cyperusesculentus oil*.Yauri Journal ofArts and Science 1-4 (2007).

3. Kochhar,S.L*Economic Botany in the Tropics* 2nd edition. Macmillan India Ltd (2007).

4. Phansteil O.N, Dueno, E, Xianghong Wang, Q*Synthesis of Exotic Soaps in theChemistry Laboratory*Journal of chemicalEducation; 612-614 (1998)

5. Chatterjee A, Pakrashi S, *The Treatise on Indian Medicinal Plants. Publications and Directorate*, New Delhi, 73 (1998).

6. Ramakrishna G, Prasad NBL, Azeemoddin G, *Cold processing neem seed*, JNTU, Oil Technological Research Institute, Proceedings of the World Neem Conference, Bangalore, India. 24 – 28 (2004).
7. Saxena RC, Neem: *a tree for solving global problems*. Report of an ad hoc panel of the Board on Science and Technology for International Development, National research council, Washington, DC, USA: National Academy press,pp 141; (1991).
8.*http://www.naturalconnection.co.za*
9. *Oil Milling Manual, Coconut Industry Investment Fund Oil Milling Group, Metro Manila, Philippines, personal communication*, 1993.
10. F.V.K.Young, 60(2), 376 (1983).
11. M. H.Gordon and I.A.Rahman, 68(8), 574–576 (1991).
12. N. Gopalakrishnan et al, 64(4), 539 (1987).
13. Donkor, Peter. *Small Scale Soap making*, TCC and Intermediate Technology Publications, 1986
14. Kirk, Othmer" *Encyclopedia of Chemical Technology*", vol. 5John Wiley & Sons, New York (1979)

.