

PRODUCTION OF DETERGENT FROM CASTOR OIL.

BY

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**A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING,
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DEDICATION

This research work is dedicated first of all to the almighty Father and Creator Jehovah God and the whole of Iledu's family.

ACKNOWLEDGEMENT

I wholeheartedly glorify the almighty Father Jehovah for the gift and preservation of my life as well as his loving directions and blessings on me in my entire life and my academic pursuit. I deeply appreciate the love and support of my parents Mr and Mrs Aduma Iledu, my elder brother Bethel and my deeply loved Uncle, late Sunday O. Iledu.

My unreserved thanks go to my uncle Paul and Isaac Oregbe for their goodwill and great support.

I also thank my friends Femi, Emmyfresh, Wisdom, Bright, Job, Christian, Frank, Mayaki, Victor, Judah, and those that I cannot mention.

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Finally, my profuse thanks go to Engr. G. Isah, my project supervisor, for his reasonableness, kindness and guidance in this work.

May Jehovah bless you all.

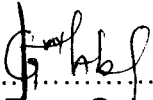
DECLARATION

I, **ADUMA OREGBE GIDEON**, hereby declare that the project "**PRODUCTION OF DETERGENT FROM CASTOR OIL**" originally done by me, to the best of my knowledge has not been presented either wholly or in part, for the award of degree or similar qualifications.

.....
Signature/Date

CERTIFICATION

This is to certify that this project titled "PRODUCTION OF DETERGENT FROM CASTOR OIL" was carried out originally by ADUMA OREGBE GIDEON of the Chemical Engineering Department, Federal University of Technology, Minna, Niger State.



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ABSTRACT

This research work was carried out with the objective of extraction of oil from castor seeds and its utilization to produce a synthetic detergent. Solvent extraction method was employed in extracting the oil and the total percent oil yield was found to be 23.8%. The experimentally determined saponification value of the oil was 183.7275mgKOH/g of oil. The detergent efficiency, determined as a measure of the foamability of the detergent was found to be 2.6cm. pH tests revealed mildly basic properties. The color, scent and efficiency of the detergent were improved with the addition of bleaching agent, perfume and foaming agents respectively.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF THE STUDY

Many of the activities of man, starting from the primitive farming techniques to today's high technology industrial activities have in small or large ways impacted negatively on man and his environment while the various products developed are highly desirable for the enhancement of the citizenry's well being and sustenance of nations' economy, the negative impacts precipitated by the introduction of its unwanted by-products into the ecological systems may be catastrophic if allowed to build up and uncontrolled.

Industrial revolution and evolution have been targeted principally at satisfying immediate changing demands rather than tailored towards a structured, wholesome and guided global program that will satisfy not only temporary human needs but environmentally safe. The result of this is an increasing ecological degradation that has severely polluted water, land and air. (Odigire, 1998)

The detergent and soap making industries are no exceptions to the above trends, for while they provide us with cleansing agents, their processing and by-products are also a cause of public nuisance. For instance, detergents, unlike soaps, have proved very effective cleansing agents in hard and cool water whereas soap is often wholly ineffective under such condition. It was observed, however that many of these detergents were neither soluble nor biodegradable, that is they were so stable that when they flow into the soil in laundry sewage water, they remain unchanged, resisting conversion into less complex and more soluble substances. They thus, create suds and foams in fresh tap water, naturally occurring ground and surface waters. (John, 1978).

To correct these odds require that environment issues be considered at the initial stages of conceptualization and development of synthetic detergents and other products as well. This will not only reduce pollution to the barest minimum but also save costs of treating these products. Adept knowledge and good manipulation of the process chemistry with a view to eliminating wastes is the most viable means of achieving this objective.

About 75 percent of detergents used in the early 1960s were for the most part made with an alkyl benzene, made from propylene tetramer coupled to benzene. Tetrapropylene is a very highly branched alkyl molecule. Research into the bacteria decomposition of alkyl based detergents showed that branched molecules are indigestible to the bacteria that must decompose the detergents when they reach the ground. To be biodegradable, a detergent should be based on straight chain alkyl molecule examples of straight chain alkyl molecules are the linear alkyl benzene sulphonates (LABS) and ricinoleic acid obtained from the castor bean. (Encarta online search)

This research project work is aimed at studying the replacement made to the non-biodegradable detergents. Production of environmentally friendly synthetic detergents (soft detergents) from castor oil has the dual advantage of using locally sourced raw material that can be grown and generating wastes that are appetizing to micro organisms (bacteria)

1.2 AIMS AND OBJECTIVES OF THE STUDY

- Extraction of oil from castor bean (seeds)
- Processing of the oil (refining)
- Production of detergent from the oil.

1.3 SCOPE OF WORK

The scope of this research work covers extraction of oil from castor beans, refining of the oil, determination of its saponification value and production of detergent from the oil.

1.4 RELEVANCE OF STUDY

Uses of castor oil as a detergent base is a step in the right direction, since petroleum and coal, the conventional detergent bases, are natural resources and will eventually be depleted. Castor plant can be grown and locally at that, its source is renewable. Hence, this work will complement or emphasize producing soft detergents from castor oil.

CHAPTER TWO

LITERATURE REVIEW

2.1.0 CASTOR – OIL PLANT

Castor – oil plant, common name for a plant of the spurge family and native to the eastern and north – eastern Africa where it grows to 12m (40ft) or more. Scientifically, the castor oil belongs to the family Euphorbiaceae. It is classified as *Ricinus Communis*.

The bean – shaped seeds, castor beans, contain the oil that is greatly valuable for manufacturing various useful products. The fruit is covered with soft, Orange – Brown spines. All parts of the plant are poisonous to humans and animals, the seeds are extremely poisonous, the injection of only 2 to 6 seeds would be a fatal dose. (Encarta Encyclopedia, 2005)

2.1.1 CASTOR OIL (RICINUS OIL)

Castor oil is a colorless or yellow to yellowish brown, thick oily liquid obtained from the seeds of castor oil plant. Although it has a disagreeable taste, castor oil is practically odorless. It is insoluble in water but soluble in organic solvents. The medicinal oil is prepared from the husked seeds, unhusked seeds; the source of industrial castor oil yield from 45-55% oil. The oil is extracted by several processes or a combination of processes which include cold pressing, continuous screw expellers and solvent extraction. Cold pressing yields the highest quality castor oil (www.Rheological.com)

2.1.2 CHEMICAL COMPOSITION OF CASTOR OIL PLANT

Castor oil is non-drying oil that owes its commercial value to being the available hydroxyl vegetable oil. The hydroxyl group is present on the 12 carbon atom of the fatty acid chain and there is a double bond between the number 9 and the number 10 carbon atoms as seen in figure 1.0 below (www.groshea.com)

Castor oil is the glyceryl tri-ester of ricinoleic acid which is also known as hydroxyoleic acid or 12-hydroxy-cis-9-octadecenoic acid (www.rheological.com). The oil is unique among all fats and oil in that:

- It is the only source of an 18 – carbon hydroxylated acid with one double bond.
- Ricinoleic acid comprises approximately 90% of the fatty acid composition.
- Product uniformity and consistency are relatively high for a naturally occurring material.
- It is non-toxic, biodegradable, renewable resource.

The remarkably constant composition of castor oil fatty acids is shown below:

Table 2.0 : - Composition of Castor oil.

COMPOSITION	PERCENTAGE
Ricinoleic acid	89.5%
Linoleic acid	4.2%
Oleic acid	3.0%
Stearic acid	1.0%
Palmitic acid	1.0%
Dihydroxy stearic acid	0.7%
Linolenic acid	0.3%
Eicosanoic acid	0.3%

The hydroxyl group in castor oil account for a unique combination of physical properties:

- Relatively high viscosity and specific gravity
- Solubility in alcohol in any proportions
- Limited solubility in aliphatic petroleum products

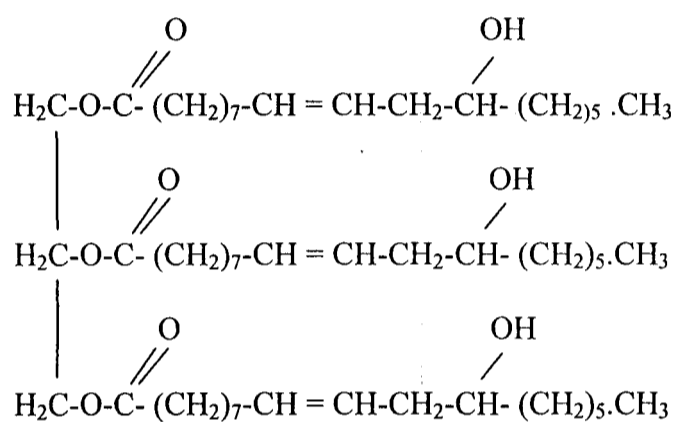
The uniformity and reliability of its physical properties are demonstrated by the long – term use of castor oil as an absolute standard for viscosity. Because of its higher polar hydroxyl groups, castor oil is not only compatible with but will plasticize a wide variety of synthetic resins, waxes, polymers and elastomers. Castor oil also has excellent emollient and lubricating properties as well as marked ability to wet and disperse dyes, pigments and fillers. In the form of its chemical derivative, castor oils application versatility is further enhanced.

2.1.3 CHEMISTRY AND USES OF CASTOR OIL

Castor oil has carved out a niche for itself as the starting point for the earliest synthetic surfactants first produce in 1875. Sulphonated castor oil has a good wetting action which permits dyes to absorb easily on to fabrics. In the nineteenth century castor oil was found to be particularly effective in promoting dyeing by Turkey Red, a brand of commercial dye widely used in that century, and which later came to be known as Turkey Red Oil (S.T Bajah, 1995)

The ester linkages, double bonds and hydroxyl groups in castor oil provide reaction sites for the production of many useful derivatives.

The diagram below shows the fundamental structure of castor oil and its functions in these reactions



Chemical reactions commercially used to produce a variety of derivatives are as follows:

Table 2.1: Chemical reactions used to produce a variety of products from Castor oil

(www.groshea.com).

	Nature of Reactants	Added Reactants	Type of Products
Ester Linkage	Hydrolysis	Acid, enzyme or Twitchell reagent catalyst.	Fatty acids, glycerol
	Esterification	Monohydric alcohols	Esters
	Alcoholysis	Glycerol, glycols, Pentaerythritol etc	Mono-acid diglycerides, monoglycols etc.
	Saponification	Alkalies, alkalis plus metallic salts.	Soluble soaps, insoluble soaps
	Reduction	Na Reduction	Alcohols.
	Amidation	Alkylamines, alkanamines etc	Amine salts, amides.
	Halogenation	SOCL ₂	Fatty Acid halogens.
Double Bond	Oxidation, Polymerization	Heat, oxygen, cross link agent	Polymerized Oils
	Hydrogenation	Hydrogen (moderate pressure)	Hydroxystearates
	Epoxidation	Hydrogen Peroxide	Epoxidized oils
	Halogenation	Cl ₂ , Br ₂ , I ₂	Halogenated oils
	Addition reaction	S, Maleic acid	Polymerized oils
	Sulfonation	H ₂ SO ₄	Sulphonated oils
Hydroxyl Group			
	Dehydration, Hydrolysis, Distillation	Catalyst (plus heat)	Dehydrated Castor oil, octadecadienoic acid.
	Caustic fusion	NaOH	Sebacic acid, Caprylalcohol
	Pyrolysis	High heat	Undecyclic acid, heptaldehyde
	Halogenation	PCl ₅ , POCl ₅	Halogenated Castor oils.
	Alkoxylation	Ethylene and/or propylene oxide.	Alkoxyated Castor oils.
	Esterification	Acetic, phosphoric, maleic, Pthalic anhydrides	Alkyl and alkylaryl esters, phosphate esters.
	Sulfation	H ₂ SO ₄	Sulforated Castor oil (Turkey red)
	Urethane reactions	Isocyanates	Urethane polymers.

2.2 DETERGENCY

2.2.1 DEFINITION OF DETERGENT

Many definitions of synthetic detergent have been proposed, all of which are very wide. The Comiti international de Dirivis Tensio Actifs has after several years of deliberation agreed on the following definition:

- Detergent: Product the formulation of which is especially devised to promote the development of detergency. Note: A detergent is a formulation comprising essential constituents (surface active agents) and subsidiary constituents (builders, boosters, fillers and auxiliaries) Detergents have also been defined as surface active agents, substances that lower the surface tension of water.
- Surface – Active Agent: Chemical compound which when dissolved or dispensed in a liquid is preferentially absorbed at an interface. Giving rise to a number of physico – chemical or chemical properties of practical interest. The molecules of the compound includes at least one group with an affinity for markedly polar surfaces, ensuring in most cases solubilization in water and a group which has little affinity for water. Note: Compositions in general are usually mixtures of such compounds.
- Amphiphilic product: Product comprising in its molecule at the same time one or more hydrophilic (water loving) groups and one or more hydrophobic (water fearing) groups. Surface active agents are amphiphilic products. (Berna, 1990)

2.2.2 DEVELOPMENT OF THE DETERGENT INDUSTRY

Although the start of the synthetic detergent industry is not shrouded in the veils of history as were the beginnings of the soap industry, it is nevertheless not easy to pinpoint exactly when the detergent, as such came into being.

The term “synthetic detergent” is used for materials, the solution of which aid in the removal of dirt or other foreign matter from contaminated surfaces, but in this definition, soap is not included. Even so, this is still a wide definition because, of course, it can refer to the active ingredient, or the solid, liquid, paste or powder compounded from this active matter. However this should not lead to confusion, as the industry itself as yet makes no distinction in terminology between the basic material and the ready-for use product.

Therefore synthetic detergents which fall into our definition of the term seem to have been developed by the Germans in the first world war period to allow fats to be utilized for

other purposes. The detergents were of short-chain alkyl naphthalene sulphonate type, made by coupling propyl or butyl alcohols with naphthalene and subsequent sulphonation, and appeared under the general name of Nekal.

In the late 1920s and early 1930s, long-chain alcohols were sulphonated and sold as the neutralized sodium salts without any further addition except for sodium sulphate as an extender.

In the early 1930s, long-chain alkyl aryl sulphonates with benzene as the aromatic nucleus, and the alkyl portion made from a kerosene fraction appeared on the market in the USA. Also during World War II, the shortage of fats, from which soap is made, spurred the development of soapless or synthetic detergents in the United States. After the war, the need for new types of detergents for automatic washing machines accelerated the trend.

Detergents made by treating an aromatic, or benzene type compound with sulphuric acid, followed by neutralization with alkali to convert the product to its sodium salt, came into wide use in the late 1940s and early 1950s and proved to be very effective in hard water and cool or cold water, whereas, soap is often wholly ineffective under both conditions. These detergents, however, became a public nuisance because, unlike soaps, they were neither soluble nor biodegradable, that is once put into water, and they tended to remain there, resisting conversion into less complex and more soluble substances. Replacing the aromatic compound with a so-called linear alkyl-type compound in the process described above led to a more desirable product. It was as effective as the former kind in its detergent action but was more biodegradable and soluble. The new linear alkylate sulfonate is changed to harmless products by microorganisms in cesspools, sewage – treatment plants and ordinary soils. Thus, between 1963 and 1965, manufacturers of detergents in the U.S have changed their process to produce only biodegradable or soft detergents.

Compact detergents were brought onto the market for the first time in 1987 in Japan where they almost completely displaced the conventional powders within a few years. The compact detergents have a high concentration of the ingredients and a high specific density, compared to the conventional detergents.

The above mentioned, among others are the major developments in the detergent industry. The factors that accounted for the development are

- Availability of raw materials
- Ease of manufacture
- Cost of raw materials
- Cost of manufacture
- Suitability of finished product.

The suitability of the finished product has been purposely placed last, as it is only too true that not always is the best material made available. The associated problem of non – biodegradability, over supply of phosphate to water ways, algae growth among others remain as very serious environmental issues that the detergent industry must contend with (Encarta Online Search)

2.2.3 USE OF ADDITIVES IN DETERGENT INDUSTRY

Additives are substances added to improve the quality and / or yield of a product (detergent). Examples of such include builders, stabilizers, enzymes, optical brighteners, bleaching agents etc.

For instances, the requirement compact detergents are expected to meet, namely increased economy per unit weight, influenced the nature and content of the active ingredients as seen in the table below.

Table 2.2 : Active ingredients for making detergent.

Ingredient	Conventional	Compact
Anionic and non-ionic Surfactants	10 – 15	10 – 25
Builders	25 – 50	25 – 40
Co – builders	3 – 5	3 – 8
Bleaching agents	10 – 25	10 – 20
Bleach activator	1 – 3	3 – 8
Redeposition inhibitors	0 – 1	0 – 1
Corrosion inhibitors	2 – 6	2 – 6
Stabilizers	0 – 1	0 – 1
Enzymes	0.3 – 0.8	0.5 – 2.0
Optical brighteners	0.1 – 0.3	0.1 – 0.3
Soil Repellents	+ / -	+ / -
Fillers / Auxiliaries	5.30	-
Water, others	Balance	Balance
Bulk density (g/l)	500 – 650	600 – 900

The biggest single revolutionary trend in the detergent industry in later years has been the use of enzyme additives. Enzymes as aids to washing are not new to the industry. Proteolytic enzymes had been tried as additives to washing powders in Germany in the 1920s with only moderate success and again in Switzerland in 1930s. Enzymes, which can be called organic catalysts, tend to hasten reactions and the proteolytic enzymes ‘break down’ proteins wholly or partially into amino acids. The action is rather slow and the production costs high. However, with improved methods of production and purification, strains of enzymes, usually in addition to mixture with a proportion of amylase which breaks down starches, were developed, which were relatively fast acting.

These were added initially to ‘pre-soak’ and found immediate acceptance in European countries where washing habits were such that materials were soaked for a period prior to the wash proper.

2.2.4 CHEMISTRY OF DETERGENCY

Detergents are surface – active agents, substances that lower the surface tension of water. Water alone is a poor cleansing agent. Its molecules are so polar that they stick to each other (via hydrogen bonds) rather than penetrate into non-polar region such as a film of grease on soiled hands or clothing. The detergents have molecules with strongly hydrophilic “heads” and hydrophobic “tails”, such as illustrated in figure 1.1.

Ordinary soap consists of a mixture of sodium (or potassium) salts of long – chain fatty acids obtained from the saponification of neutral fats. Because the negative ions of soap from precipitates with metallic ions in hard water (Ca^{2+} , Mg^{2+} or $\text{Fe}^{2+}/\text{Fe}^{3+}$), synthetic detergents (“synderts”) were developed that were fully soluble in such water.

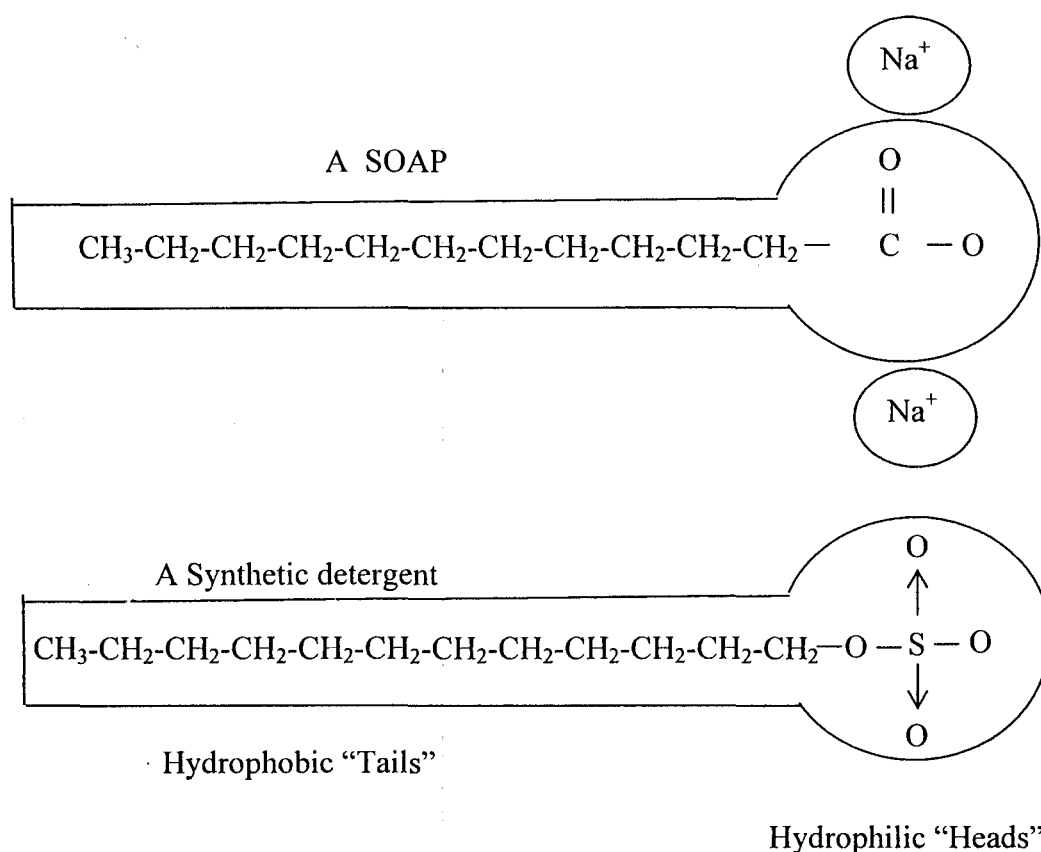
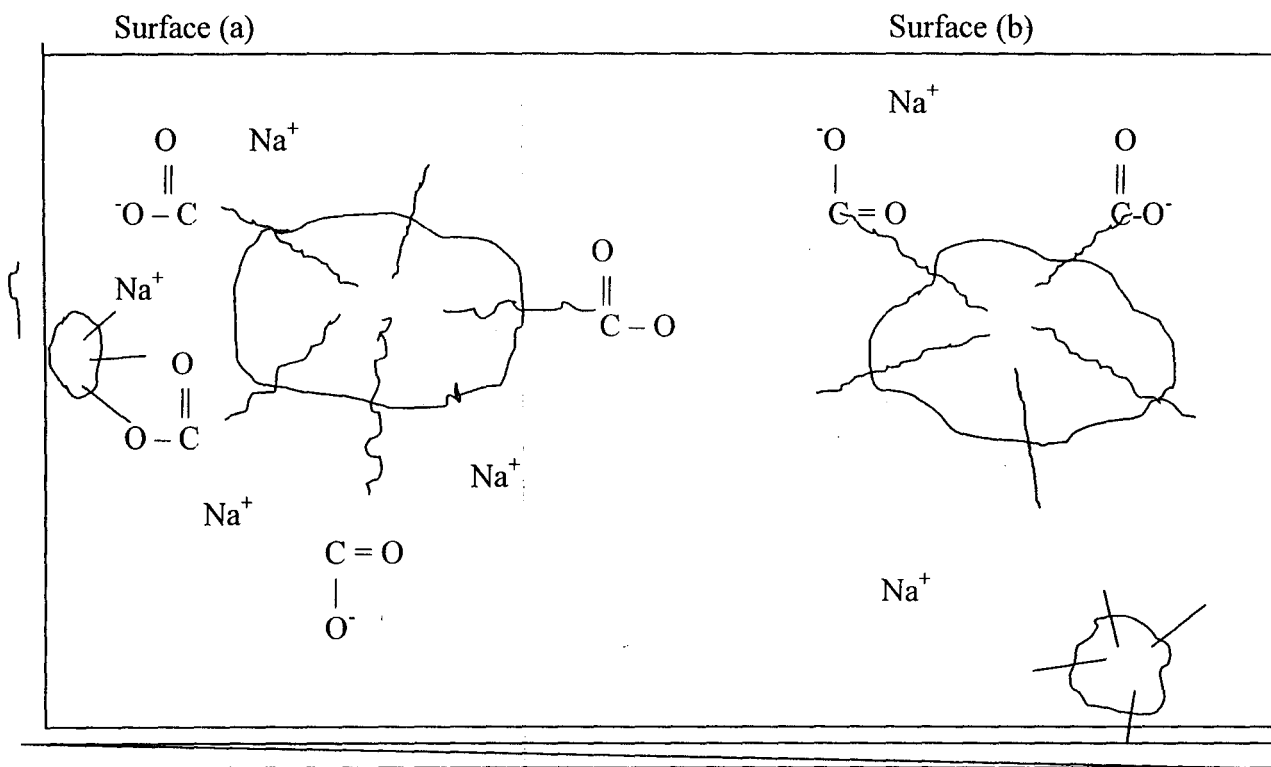


Fig 2.0 Two organic salts with detergent properties

How detergents work to loosen greasy films is illustrated in figure 1.2.



Surface (C)

Fig 2.2 How detergents work

- a) Hydrophobic tails of detergent molecules work into the hydrophobic environment of the greasy layer.
- b) Hydrophilic heads of the detergent molecules, remaining in contact with the water phase; help work the greasy layer loose. This process goes faster if the water phase is hot and mechanical agitation used (as in washing machines).
- c) Globules of greasy material form, pin-cushioned by detergent molecules. The head of the pins all bear electrical charges, which help keep the Globules from rejoining and also keeps them in solution (John, 1978)

2.2.5 TYPES OF DETERGENTS

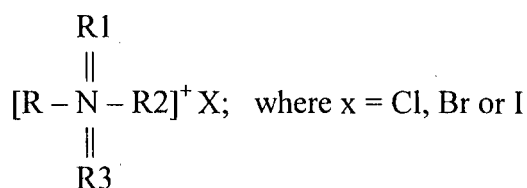
Four different classes of synthetic surfactants are known. There are anionic surfactants, cationic surfactants, non-ionic surfactants and amphoteric surfactants (Bajah, 1995).

- Anionic surfactants: Anionic surfactants resemble soap because like soaps the hydrophilic head is an anion. Examples include the alkyl sulphates and alkyl sulphonates whose structures are compared with that of soap as follows:

Table 2.3: Examples of anionic surfactant

Type	Structure	Example
Soap	$R.CO.O.Na^+$	$C_{15}H_{31}COONa$ (sodium palmitate)
Alkyl Sulphate	$\begin{array}{c} O \\ \\ [R-O-S-O]Na^+ \\ \\ O \end{array}$	$C_{12}H_{25}OSO_3Na$
Alkyl Sulphonate	$\begin{array}{c} O \\ \\ [R-S-O]Na^+ \\ \\ O \end{array}$	$C_{11}H_{23}CH_2-SO_3Na$

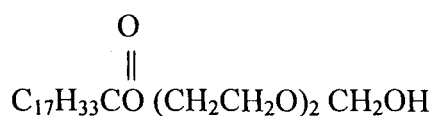
- Cationic surfactants: In this class of surfactants the hydrophilic head is positively-charged. Many are substituted ammonium halides; with the general formula



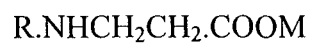
They are thus, quaternary ammonium salts and for the case where the alkyl groups contains $C_6 - C_{12}$ chain the detergents have antibacterial activity. Hence, in addition to being surfactants they are also disinfectants.

An example of cationic surfactant is benzalkonium chloride.

- Non-ionic surfactant: This class of surfactant is characterized by the fact that they do not ionize in water i.e. they are neutral emulsions. An example is the oleic ester of polyethylene glycol.



- Amphoteric surfactants: This class of surfactants contains both acidic and basic functions. They have the following structure.



In this structure, R is the fatty acid and M is the cation or hydrogen. Depending on the pH, amphoteric surfactants can exist in three forms of cationic, amphoteric and anionic, as seen in the table below.

Table 2.4: - pH range of detergent

Acidic pH range	Neutral pH range	Basic pH range
$RNH_2CH_2CH_2COOH$ (cationic)	$RNH_2CH_2CH_2COO^-$ (amphoteric)	$RNHCH_2CH_2COO^-$ (anionic)

With this class of surfactants, detergency falls to a minimum at the isoelectric point and hence can be controlled. They find use in hair shampoos where it is desired to clean the oil sebum but not to wash off all the oil on the skin or hair.

2.2.6 DETERGENT FOAMABILITY

The efficiency of a washing powder is assessed through the amount of foam it is capable of producing. This assessment is however subjective. The presence of persistent foam usually exemplifies a good detergent. Foam stabilizers are designed to maintain persistent foam. Economic foam boosters are coconut fatty acid diethanolamides made from coconut fatty acids and diethanolamine.

2.3 BIODEGRADABILITY (BIODEGRADATION)

2.3.1 DEFINITION OF BIODEGRADABILITY

The term biodegrade is often associated with environmental friendly products. It is defined as the act of being broken down by naturally processes, into more basic components. Products are usually broken down by bacteria, fungi or other simpler organisms. By this definition, most chemicals are biodegradable. The only thing differing would be the amount of time it takes to breakdown.

Rate of breakdown may not be as important as what the product breaks down into. The ideal final product of any complex product of carbon, hydrogen and oxygen would be carbon dioxide (CO₂) and water (H₂O). A majority of products are made mostly of these three elements.

In detergents, though other organic ingredients such as enzymes any also be biodegradable, the term is most often applied to the surface active agents- surfactants contained in the product. When detergents first came out into the general use in the 1950's, there were problems of foaming on rivers. In this first generation of "synthetic" detergents washing powders, branched chain alkyl benzene sulphonate (ABS) was extensively used as the surfactant.

Because even traces of detergents can produce suds, we do not want them in the drinking Water supply. The negative ions in sod nary soap in destroyed by bacteria in the soil or in sewage treatment plants. These bacteria metabolize such ions. We say that soap in biodegradable because biological organism degrade. The structure of one given in table 1.3 has several branches. Bacteria evidently cannot work their way around these branches. The most biodegradable detergents have straight – chain hydrophobic "tails".

Table 2.5: Biodegradability of Some Synthetic Detergents

Types of synthetic detergents	Examples to illustrate structural Features	Biodegradable?
Sodium alkyl sulphate	$\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2\text{OSO}_2\text{O} - \text{Na}^+$	Yes
Sodium alkyl benzene sulphonate, tetrapropylene – based	$\begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \quad \text{CH}_3 \quad \text{CH}_3 \\ \quad \quad \quad \\ \text{CH}_3\text{CHCH}_2\text{CHCH}_2\text{CHCH}_2\text{CH} - \text{C}_6\text{H}_4 - \text{S} - \text{O} - \text{Na}^+ \\ \\ \text{O} \end{array}$	NO
Sodium alkyl benzene sulphonate, n-paraffin or ∞ - olefin based	$\begin{array}{c} \text{CH}_3 \quad \text{O} \\ \quad \\ \text{CH}_3(\text{CH}_2)_n\text{CH} - \text{C}_6\text{H}_4 - \text{S} - \text{O} - \text{Na}^+ \\ \\ \text{O} \end{array}$	Largely so
Sodium alkane sulphonate	$\text{C}_n\text{H}_{2n+1}\text{SO}_3 - \text{Na}^+$ (chain is largely straight, n is 15 to 18)	Almost completely

Detergents are for the most part made with tetra propylene (table 1.3), a very highly branched alkyl molecule. Branched molecules are indigestible to the bacteria that must

Decompose the detergents when they reach the ground. To be biodegradable, a detergent should be based on a straight – chain alkyl molecules. The straight- chain linear alkyl benzene sulphate (LABS) solved the problem (John 1978).

In West Germany, where the detergent – disposal problem is very severe a research firm developed a series of entirely new detergents that are claimed to be 100% biodegradable. These are made from sugar – esterifies with a fatty acid, preferably one having more than one hydroxyl group, for example ricinoleic acid, obtained from Castor oil (table 1.1) (Encarta online research). Castor oil is the glyceril-ester of ricinoleic acid which is also known as hydroxyoleic acid. The structure of the glycerides of castor oil has been ascertained with approximately 68 percent tri-ricinoleic, 28 percent di-ricinoleic, 3 percent mono-ricinoleic and one percent ricinoleic acid. Hence, detergents synthesized from oil will satisfy the biodegradability requirement.

Moreover, Ricinoleic acid comprises approximately 90% of the fatty acid composition.

The move to using biodegradable surfactants came in the early 1960s and was, in the UK, a voluntary action by detergent industry. The first legal requirements were not laid down until 1973 when a European council directive was issued on surfactant biodegradability. In these days of growing consumers awareness of green issues however, “biodegradable” is a word often used as an indication of a product’s environmental friendliness. It is though, only one aspect of the environmental profile of a product. For example, though it is an important factor influencing the fate of the components after their disposal to the environment, other processes such as photo degradation, chemical degradation and absorption may also have an influence.

Biodegradation of detergent starts as soon as dirty laundry water is washed down the drain. Micro organisms (or bacteria) thrive naturally in sewage treatment of plants, waterways and soil. These bacteria break down surfactant, using them as food for energy and growth. Since there are very large numbers of bacteria at the sewage treatment plants, biodegradation takes place there very rapidly and substantially complete biodegradation is usually achieved within a few hours.

The first steps in the biodegradation process will often produce an intermediate product or metabolic and this stage is referred to as primary biodegradation. Subsequent action by bacteria may then result in further decomposition of the metabolites to produce simple inorganic compounds such as carbon dioxide, water and mineral salts. This complete breakdown of the compounds is known as ultimate biodegradation.

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Primary biodegradation of a surfactant usually results in the loss of environmentally undesirable properties such as foaming ability. It is therefore very important that this Stage of biodegradation takes place rapidly if undesirable effects are to be avoided. Thus, the European directives on surfactant biodegradability specify the primary biodegradability of anionic or nonionic surfactants (i.e. those which have a negative charge or more) to exceed 80 percent in a specific test with a residence of only 3 hours. This does not mean that the remaining 20 percent will not degrade, but rather that this may not always be achieved in the standard duration of the laboratory test.

In 1995, UK pilot for a European monitoring study measured how effectively detergent surfactants were removed by sewage treatment works. The results showed a removal rate of 99.9 percent. Similar studies carried out in Spain, Germany, Italy and the Netherlands resulted in the average surfactant removal rate of 99.2 percent overall.

Any residual material of metabolites released to waterways before ultimate biodegradation is complete will continue to biodegrade (Feijtel 1995)

2.3.2 BIODEGRADATION AND THE LAW

Experience with foaming problems caused by the use of poorly biodegradable alkyl benzene sulphonate led to legislation controlling their biodegradability in a number of countries. The European council for instance, harmonized this legislation and the biodegradability of surfactants in detergents is the subject of a number of council Directives.

In 1973 directive (73/404), all surfactants should have 90 percents average biodegradability. No specific test methods were laid down. (73/404) of the directive (second) was specifically concerned with anionic surfactants used in detergents and specifically concerned with anionic surfactants used in detergents and specified laboratory test methods.

In 1982, the list of acceptable test for biodegradability was modified (directive 82/243). Similar test methods and criteria for non-ionic surfactants were laid out (Feijtel 1995).

2.3.3 PHOSPHATE - CONTAINING DETERGENTS

When Phosphate detergents are used, disposal of the wash waste water is an environmental issue. The breakdown of the phosphorus complexes in phosphates creates

biologically available phosphates. In waterways, these can contribute to oversupply of phosphate. Low concentration of plants nutrients, such as nitrogen and phosphorus, can limit plant and algae growth. When phosphates from the source mentioned above are introduced to waterways any phosphorus limitation may be removed and may cause excessive algae growth which in turns, can lead to:

- Increased Numbers of insects, crustaceans and fish in the water way. When these die and decompose, oxygen in the water is reduced to such low levels that other aquatic organisms are killed.
- Cyano-bacteria (blue- green algae) release toxins that kill other organisms and make the water unsuitable for humans, livestock's and wildlife.

Thus, phosphate detergents waste waters should not be allowed to wash into storm drain but rather should be directed to sewage treatment plant.

2.3.4 PHOSPHATE - FREE DETERGENTS

Disposal of phosphate-free detergent water is also an environmental issue. Phosphate is primarily used in detergents as “builders”, which remove calcium ions and allow the surfactants to work more effectively. In phosphate-free detergents, the phosphates replaced by such alternatives builders as:

-Zeolites

- Increased concentration of surfactants

Because the surfactants are among the most toxic compounds in detergents and have implications of decreasing the ability of aquatic organisms tom breed, the waste waters must be treated in sewage treatment plants. Unfortunately, even after treatment, the effect of some alternative builders remains.

2.4 EXTRACTION OF OILS

2.4.1 PRINCIPLES OF EXTRACTION

Leaching is concerned with the extraction of soluble material or constituents from a solid by means of a solvent. The process may be used either for the production of a concentrated

solution of a valuable solid material, or in order to remove an insoluble solid, such as a pigment from a soluble material with which it is contaminated. The method used for the extraction is determined by the proportion of the soluble constituent present, its distribution throughout the solid, the nature of the solid and the particle size. If the solute is uniformly dispersed in the solid, the material near the surface will be dissolved first, leaving a porous structure in the solid residue. The solvent will then have to penetrate the outer layer before it can reach further solute and the process will become progressively difficult and the extraction rate will fall. If the solute forms a very small portion of the solid, the porous structure may break down almost immediately to give a fine deposit of insoluble residue, and access of solvent to the solute will not be impeded.

In some cases, the soluble material will be distributed in small isolated pockets in a material which is impermeable to the solvents such as gold dispersed in rock, for example. In such cases, the material is crushed so that all the soluble material is exposed to the solvent. If the solute has a cellular structure, the extraction rate will generally be comparatively low because the cell walls provide an additional resistance, which walls can also impede the extraction of undesirable constituents.

2.4.2 METHODS OF EXTRACTION

Processing of seeds into oils is a very common practice for obtaining economically useful oils and protein meals. Several processes or combination of processes are employed in the extraction of oils e.g. castor oil and these include rendering, cold processing, continuous strew expellers and solvents extraction.

- **RENDERING:**

This method is used for seeds or fruits bearing large amount of fatty materials. It involves the heaping the piles, exposing them to the sun and collecting the exudates (oil).

- **COLD PRESSING:**

This is a mechanical process where the oil bearing fruit or seed is crushed and then rolled or pressed under pressure to liberate the oil. Commonly used are hydraulic or screw pressing. Cold pressing yields the highest quality castor oil.

- **SOLVENT EXTRACTION:**

Dissolution of soluble matter (oil) from its mixture with an insoluble solid, using a suitable solvent is the most suitable method for the extraction of oil from oil – bearing seeds where oil content is less than about 5%. Examples of solvents (organic) include n-hexane, benzene etc. The process can be carried out as a cold process (maceration) or hot process. The hot process is faster and less tedious compared to the cold. The oil is obtained finally after the solvent has been recovered from the leached solution.

Solvent extraction was used in this process work for the extraction of the castor oil of relatively high molecular weight, as in the extraction of sugar from beet- in the extraction of oil from seeds, the solute is itself liquid.

2.4.3 SOLVENT EXTRACTION OF CASTOR OIL

Generally, the process of solvent extraction of oil from seeds (castor seed) can be considered in these parts.

- Firstly, the change of phase of the solute as it dissolves in the solvent.
- Secondly, the diffusion of the solute (oil) through the solvents in the pores of the solid to the outside of the particle.
- Thirdly, the transfer of the solute from the solution in contact with the particles to the main bulk of the solution.

Anyone of the above mentioned process may be responsible for limiting the extraction rate , though the first process usually occur so rapidly that it has negligible effect on the over all rate.

2.4.4 FACTORS INFLUENCING THE RATE OF EXTRACTION

The selection of equipment fro an extraction process is influenced by the factors which are responsible for limiting the extraction rate. Thus, if diffusing of the solute through the porous structure of the residual solid is the controlling factors; the material should be of small size so that the distance the solute has to travel is small. On that other hand, if diffusion of the solute fro

the surface of the particles to the solution is the controlling factor, a high degree of agitation of the liquid is required.

There are four important factors to be considered:

- **PARTICLE SIZE:**

The smaller the size, the greater is the interfacial area between the solid-liquid (solvent), and therefore, the higher is the rate of transfer of material and the smaller is the distance the solute must diffuse within the solid as already indicated. However very finely divided material may impede the circulation of the solvent and separation of the oil from the solid becomes more difficult.

- **SOLVENT:**

The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely, though this will reduce as the process progresses.

- **TEMPERATURE:**

In most cases, solute solubility will increase with temperature to give a higher rate of extraction. Further, the diffusion coefficient will be expected to increase in temperature and this will also improve the rate of extraction.

- **AGITATION OF THE FLUID:** Agitation of the solvent is important because this increases the eddy diffusion and therefore the transfer of material from the surface of the particles to the bulk of the solution, as discussed earlier. Further, agitation of suspension of fine particles prevents sedimentation and more effective use is made of the interfacial surface.

2.4.5 MASS TRANSFER IN LEACHING (SOLVENT EXTRACTION)

OPERATION

Mass transfer rates within the porous residue are difficult to access because it is impossible to define the shape of the channels through which transfer must take place, it is possible however, to obtain an approximate indication of the rate of transfer from the particles to the bulk of the liquid. Using the concept of a thin film as providing the resistance to the transfer, the equation of mass transfer may be written as:

$$Dm/dt = K'A (C_s - C)/b$$

where A is the area of the solid liquid interface b is the effective thickness of the liquid film surrounding the particles C is the concentration of the solute in the bulk of the solution at time t C_s is the concentration of the saturated solution in contact with the particle M is the mass of solute transferred in time t and K' is the diffusion coefficient (COULSON 1991)

2.4.6 SOLVENT SELECTION

For optimum extraction rate and product (oil) quality, several factors should be considered in making the choice of solvent in extraction operations and these include the following.

- a) Selectivity: The solvent should be capable of selectively extracting a desired solute at the expense of others.
- b) High Relative Volatility : The term volatility refers to the ability of a liquid to change to vapor state. The volatility of a chosen solvent should be very high for easy recoverability after extraction since the solvent is always recovered by distillation.
- c) Stability: The chosen solvent should be thermally stable to prevent decomposition and any side reaction during heating.
- d) Low Viscosity: This is necessary so that the solvent can flow freely
- e) Toxicity and Availability: Oils extracted are mostly used directly or indirectly by man or livestock's. Hence, a solvent should be non-toxic and should be readily available at affordable cost.
- f) Polarity: for solubility purposes be considered. For example, organic compounds are insoluble in inorganic solvents.

- g) Inflammability: To avoid the risks of fire and explosion, solvents with flash points below 3.22 should not be used for extraction.

The solvent extraction method was selected here as it has the advantage for oil extraction when the oil content is too low for rendering and pressing to be employed. Moreover, the process is faster and less tedious. However, it has the disadvantages of difficulties of solvent solute separation after the extraction and possible explosion poor extraction if a wrong choice of solvent is made. *hazardous or highly flammable solvents*

2.4.7 PROPERTIES OF n - HEXANE

The properties of n - hexane, that makes it desirable and hence the choice solvent for the extraction of castor oil, as reported in the dictionary of organic compounds (1982) 5th edition are given below.

- (1) Boiling point: 68.95°C
- (2) Flash point: - 23 °C
- (3) Freezing point: - 93.5 °C
- (4) Refractive index: 1.3723
- (5) Moderately toxic vapor: TLV of 360

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS

The castor seeds (or beans) were gotten from the Kabba local government area of Kogi state. Other materials used are shown in the table 3.1 below:

Table 3.1 List of materials

S/No	Materials	Make
1	Mortar and Pestle	Locally made
2	Electric oven	Genlab widnes, England
3	Digital weighing balance	Ohaus Corporation, N J USA
4	Soxhlet apparatus	Genlab widnes, England
5	Stand and clamp	Ohaus Corporation, N J USA
6	Water bath	Genlab widnes England
7	Rotary evaporator	Genlab widnes England

Table 3.2 List of Chemicals and Reagents

1	N – hexane	100%
2	Distilled water	...
3	Ethanoic potassium hydroxide	...
4	HCl	0.5M
5	Phenolphthalin indicator	0.1M

3.2 METHODS

The operations involved in extraction of castor oil can be divided into two:

- (i) Pre-treatment of the castor seeds
- (ii) Solvent extraction of the castor oil

3.2.1 PRE-TREATMENT OF CASTOR SEEDS

The pre-treatment involves the preliminary preparations of the seeds in the following:

- (a) **Shelling:** This involves the removal of the shells to obtain the seeds. It was done manually.
- (b) **Clearing:** This involves the removal of foreign matter introduced during the sun drying and any unshelled seed.
- (c) **Drying:** The moisture content of the seed was reduced using the electric oven. The oven was operated at 80°C for about three hours.
- (d) **Crushing:** Mortar and pestle were used to reduce the sizes of the dry seeds so as to increase the interfacial area between the solvent and the seeds.

3.2.2 SOLVENT EXTRACTION OF CASTOR OIL

The soxhlet apparatus (figure 3.10) was used in extracting the oil.

3.2.3 OPERATION OF SOXHLET APPARATUS

100ml of n-hexane was poured into the round bottom flask. 10g of castor beans was placed in the thimble and inserted in the center of the soxhlet extractor as seen in the figure. The extractor was heated at 70°C when the solvent was boiling, the vapor rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the center which contains the solid sample to be extracted. The extract sips through the pores of the thimble and fills the siphon tube where it flows back down into the round bottom flask. This was allowed for 30mins after which the sample was removed from the tube, dried in the oven, cooled in the desiccators and weighed to determine the amount of oil extracted.

The experiment was repeated by placing 5g of the sample into the thimble again, and after every 30mins, the samples were withdrawn for drying and weighing.

The miscella (extracted oil mixed with solvent) was heated at the end of the extraction to recover the solvent from the oil. The solvent free oil was then refined for further use.

3.2.4 REFINING OF CASTOR OIL

The refining was needed to remove gum from the extracted oil.

Boiling water was added to the oil and the mixture stirred for 2Mins and allowed to stand in the separating funnel. The aqueous layer was then removed. The procedure was repeated to ensure removal of most gums. The degummed oil was collected and stored for use.

3.2.5 DETERMINATION OF SAPONIFICATION VALUE

2g of the oil was placed in a conical flask to which 25ml of ethanoic potassium hydroxide (0.1M) was added and the mixture allowed to boil gently for about 60Mins with shaking at regular intervals of 5mins.

Few drops of phenolphthalin indicator, as specified by International Standards Organization (ISO 3657, 1988) was added to the warm solution and then titrated with 0.5M HCl. The end point was reached when the pink colour of the indicator just disappeared. The same procedure was followed for the blank.

The saponification value (s v) is given by

$$s v = \frac{56.1N(V_0 - V_i)}{m} \quad \text{-----} \quad 3.0$$

where V_0 = volume of HCl solution used for the blank test

V_i = volume of HCl solution for the determination

N = actual molarity of HCl used

m = mass of sample

3.3 PRODUCTION OF DETERGENT

3.3.0 MATERIALS

The following were the equipments used in preparing the detergents.

Table 3.3.0: List of Equipments

S/No	Materials	Make
1	Stainless steel plate	Tower extrusions, Lagos
2	Syringe	SOLOGARD
3	Glass rod	Genlab widnes, England
4	Measuring cylinder	Genlab widnes, England
5	Beakers	Genlab widnes, England
6	Hot plate	Ohaus Coporation, N J USA
7	Digital weighing balance	Genlab widnes, England

Table 3.3.1 Chemicals/Reagents

S/No	Reagents	Function
1	Castor Oil	Base reagent
2	Palm Kernel oil	Base reagent
3	Caustic soda (0.1M)	Neutralization
4	Hydrogen peroxide	Bleaching agent
5	Sulphuric acid (18M)	Surfactant/foaming agent
6	Perfume	Scent

3.3.1 PROCEDURE

0.1M sodium hydroxide solution was prepared by weighing 40g of NaOH Pellets into a beaker containing 100ml of water and shaking vigorously.

The electric hot plate was switched on, 30ml of palm kernel oil in a stainless steel plate was placed on it and heated at 35oC for about 2Mins. Caustic soda (0.1M) was added and the mixture stirred with a glass rod. 18M sulphuric acid was then added with constant stirring, and the reaction allowed to completion after which hydrogen peroxide (Bleaching agent) was introduced into the reaction mixture. When the foaming had subsided, the heating was continued to allow for more vaporization before putting off the heating system. Finally, perfume was added and the system was allowed to cool.

The powdered detergent formed was subjected to foamability test to ensure the effectiveness of the process.

The same steps were then followed, but this time castor oil was used as the base material. The resulting powdered detergent formed was again collected and tested as above. The results are as shown in the result section of this project work.

The quantities of the reagents used are shown in the tables below:

Table 3.3.2: Quantity of reagents used

Material/Reagent	Amount
Castor oil	30ml
Caustic soda	18ml
Sulphuric acid	3ml
Hydrogen peroxide	3ml
Perfume	To taste

Material/Reagent	Amount
Palm Kernel Oil	30ml
Caustic soda	18ml
Sulphuric acid	3ml
Hydrogen peroxide	2.0ml
Perfume	To taste

3.4 FOAMABILITY TESTS ON DETERGENTS PRODUCED

3.4.0 MATERIALS

500ml measuring cylinder

Weighing balance

Distilled water

Meter rule

3.4.1 PROCEDURE

About 2.0g of the palm kernel based detergent was added to a 500ml measuring cylinder containing 100ml of distilled water and the mixture shaken vigorously so as to generate foams. After shaken for about 2mins, the cylinder was allowed to stand for about 10mins. The height of the foam in the solution was measured and recorded.

The same steps were followed using the detergent produced with the castor oil so that the foamability of the two could be compared. The results obtained are as recorded in the result section of this work.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.0 RESULTS

The results obtained for the percent volume of oil extracted, determination of saponification value and the production as well as the foamability tests of the detergent produced are as seen below:

Table 4.0: Percent volume of oil extracted

Weight of raw sample W_1 (g)	Weight of raffinate W_2 (g)	Weight of extract (oil) (g)	Percent oil extracted W (g)
25.00	16.45	8.55	34.20
20.00	13.20	6.80	34.00
30.00	22.00	8.00	26.67
35.00	26.80	8.20	23.43
40.00	29.10	10.90	27.25

Table 4.1: Height of foam formed by the detergents produced

Detergent base material	Height of foam formed in water (cm)	
	Sample 1 (2.0g)	Sample 2 (2.0g)
Castor oil	2.57	2.60
Palm kernel oil	2.10	2.00

The saponification value obtained for castor oil as calculated from equation 3.0 is 183.7275mgKOH/g of oil.

4.1 DISCUSSION OF RESULTS

The results obtained for the extraction showed a total percent oil extracted to be 28.3%. This value is low relative to similar works done with a total percent oil yield of 33.2% oil (Abdulahi, 2002). The low yield could be attributed to the nature of the seeds, unequal particle size and low temperature of extraction (70°C) compared to the boiling point of n – hexane (68.95°C). The oil quality was very desirable as demonstrated from the saponification value of 183.7275 mg KOH/g of oil, which compares very favorably with that in literature (180.00 mg KOH/g of oil).

Moreover, the sulphation and neutralization reactions gave a powdered detergent of high enough efficiency as seen from the result of the foamability tests. Usually, the efficiency of a washing powder is assessed through the amount of foam it is capable of producing. The presence of persistent foam exemplifies a good detergent (Bajar, 1995). The foam height of 2.6cm persisted for about 10 minutes and is higher than that formed by the palm kernel based detergent. The detergent formed was the result of the esterifications of the castor oil.

When ricinoleic acid (castor oil) is treated with concentrated H₂SO₄, it gives a complex mixture consisting of hydrogen sulphate (OSO₃H) of ricinoleic acid in which the hydroxyl group is esterified and a compound in which the H₂SO₄ has added to the double bond. Esterification and addition do not occur together in the same molecule of ricinoleic.

The product which is known as Turkey red oil (sulphated castor oil) has good wetting properties. Neutralization of this with aqueous NaOH gave a detergent plus water. The reaction proceeded at temperatures between 35 – 40%. The water was vaporized by further heating and a solid (powdered) detergent was the result. The bleaching agent (H₂O₂) added helped to bleach the color of the castor oil so that a milk colored detergent was produced. pH tests showed that the detergent exhibited basic property. The detergent can thus be described as amphoteric (i.e. containing both basic and acidic functions). This classification is characteristic of the intrinsic property of castor oil. This pH range is preferable to that of acidic as it is non – corrosive to the skin and cloths.

From the structure of castor oil (Ricinoleic acid) as shown under literature review, the sulphation reaction occurred at the hydroxyl group while the esterification reaction occurred at

the ester linkages and this can be used to produce both soluble and insoluble soaps. Hence the detergents produced was the result of the esterification of the ricinoleic acid.

The equations of the reactions are as seen in Appendix D

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.0 CONCLUSION

This research project have been undertaken to extract castor oil from its seed, refine this and use the same to produce a synthetic detergent.

The extraction was done using n-hexane as the solvent. The oil was refined by degumming to remove most gums. Sulphation and neutralization of the refined oil gave a synthetic detergent. Other operations like bleaching, perfuming and drying were done to improve color, scent and texture of the detergent. Although many other additives such as optical brighteners, extenders, re-deposition inhibitors, enzymes, etc. were not added, the active ingredients (sulfactants) were used and as such the detergent efficiency was high.

The production of synthetics detergent from castor oil was successively done. The many desirable intrinsic qualities of castor oil makes it very useful in the detergent industry, thus castor oil can serve as a good substitute to petroleum and coal, the conventional detergent bases.

5.1 RECOMMENDATION

Literature reviews showed that castor oil has a linear structure and as such a detergent based on it should be digestible to soil micro organisms. It would therefore be a step in the right direction if this biodegradable property of the produced detergent is tested. Moreover, castor oil finds wider application in other areas such as pharmaceuticals and lubricating fluids, synthetic polymers e.t.c.

It is therefore recommended that more practical work be done to modify the oil for such wider applications

APPENDIX A

Percentage of Oil Extracted

$$\begin{aligned}\text{Sample 1: } W &= \frac{W_1 - W_2}{W_1} \times 100 \\ &= \frac{(25 - 16.45)}{25.0} \times 100 \\ &= 34.2\%\end{aligned}$$

$$\begin{aligned}\text{Sample 2: } W &= \frac{(20 - 13.20)}{20.0} \times 100 \\ &= 34.0\text{g}\end{aligned}$$

$$\begin{aligned}\text{Sample 3: } W &= \frac{(30 - 22.0)}{30.0} \times 100 \\ &= 26.67\%\end{aligned}$$

$$\begin{aligned}\text{Sample 4: } W &= \frac{(35 - 26.8)}{35.0} \times 100 \\ &= 23.43\%\end{aligned}$$

$$\begin{aligned}\text{Sample 5: } W &= \frac{(40 - 29.1)}{40.0} \times 100 \\ &= 27.25\%\end{aligned}$$

$$\begin{aligned}\text{Total Percent oil extracted: } &= \frac{W_{1\text{total}} - W_{2\text{total}}}{W_{1\text{total}}} \times 100 \\ &= \frac{150 - 107.55}{150} \times 100 \\ &= 28.3\%\end{aligned}$$

APPENDIX B

Calculation of saponification value of castor oil

From equation 3.0,

$$s.v = \frac{56.1N(V_0 - V_i)}{m}$$

where N is the actual molarity of HCl = 0.5M

V_i is the average volume of HCl required for the determination = 2.90ml

V_0 is the blank solution volume of HCl required = 16.00ml

m is the mass of oil sample used = 2g

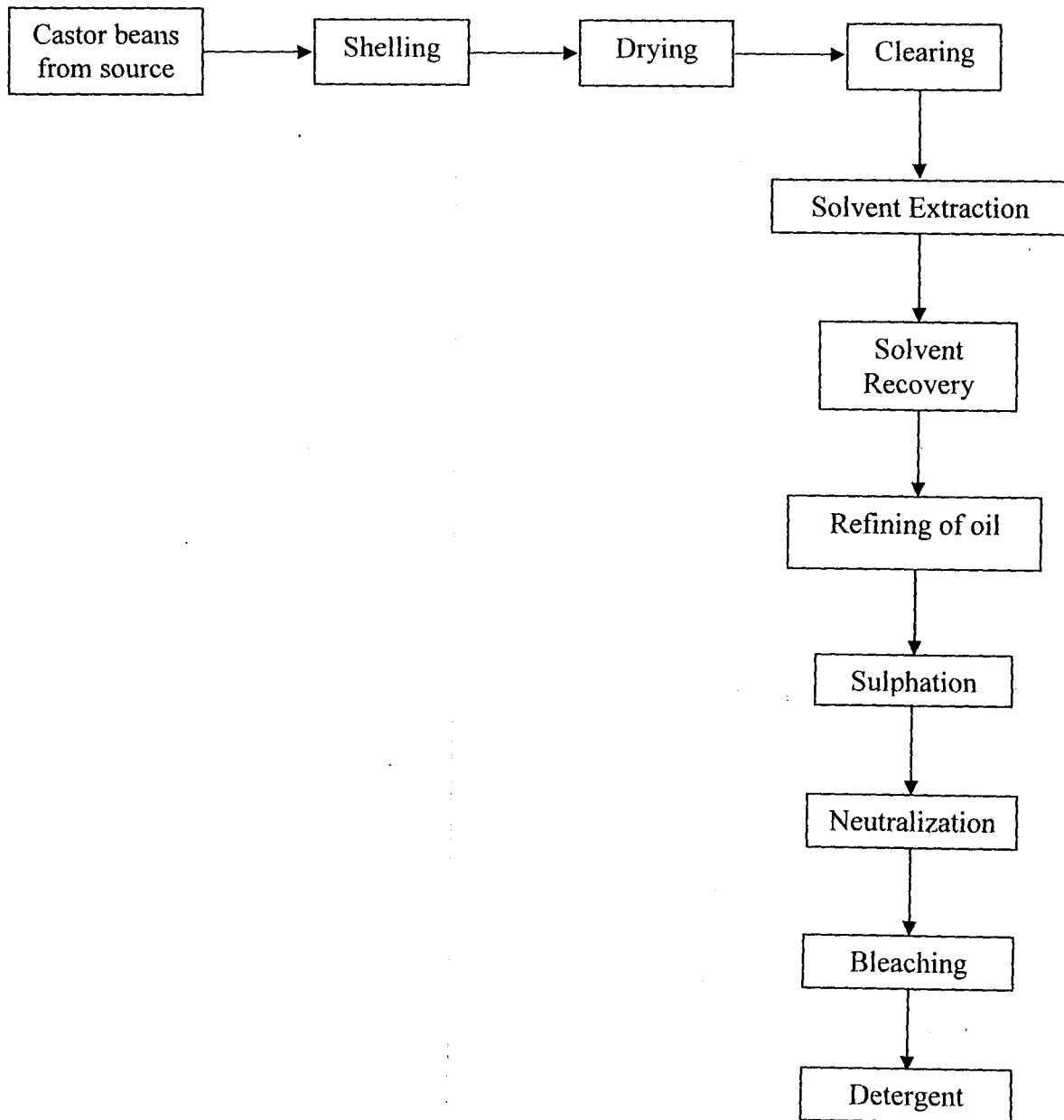
Thus,

$$\begin{aligned} s.v &= \frac{56.1 \times 0.5(16 - 2.90)}{2} \\ &= 183.7275 \text{mgKOH/g of oil} \end{aligned}$$

APPENDIX C

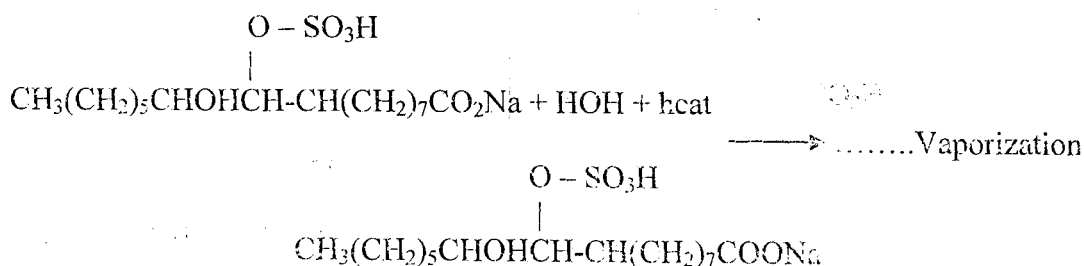
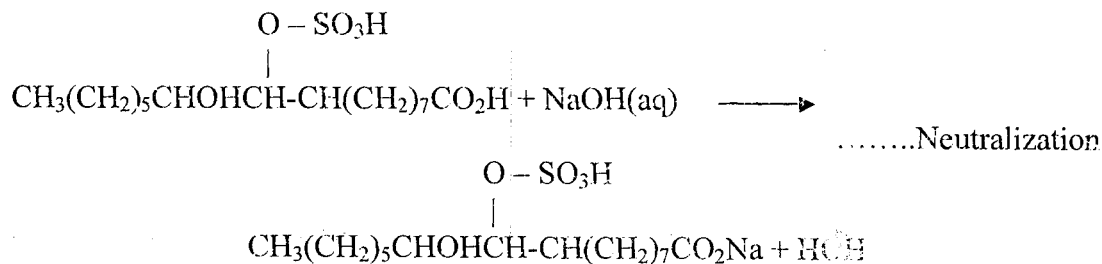
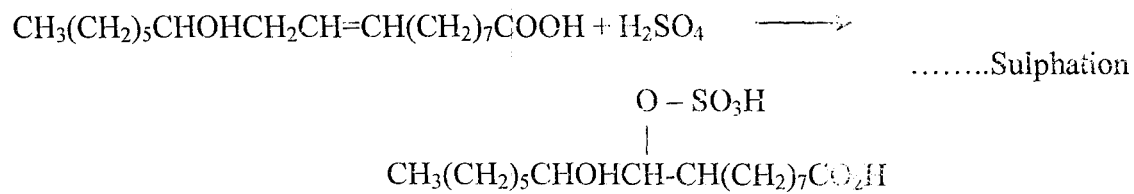
Flow Diagram for the extraction of castor oil and production of synthetic detergent.

The operation performed on the castor beans from the first stage of collection of the beans through extraction and the manufacture of the detergent is represented by the flow diagram below:



APPENDIX D

The equations of the reactions are:



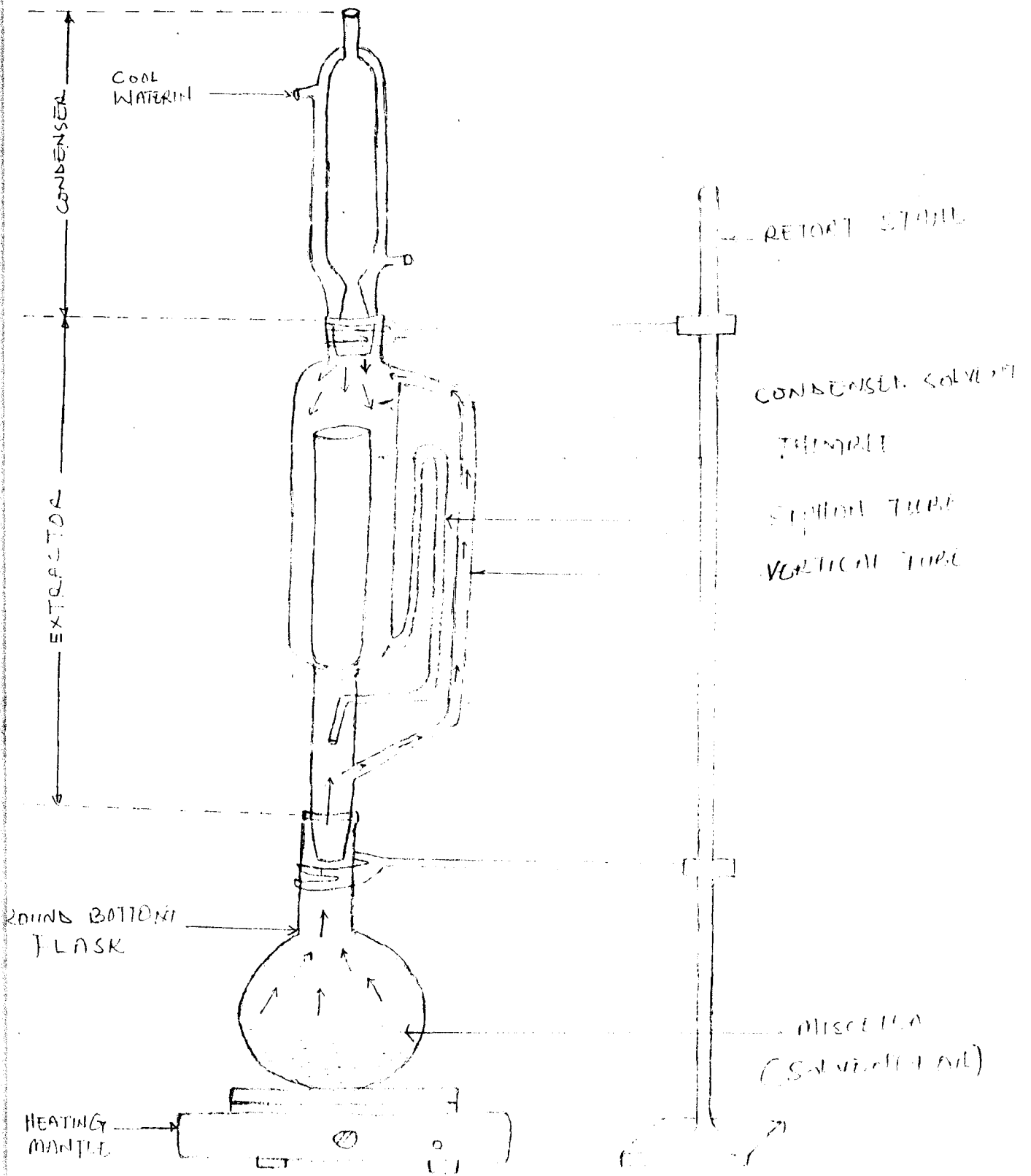


Fig. 3.0 : Soxhlet Apparatus.