Addis Ababa University

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School of Chemical and Bio Engineering

Process Engineering Stream

Synthesis and Characterization of Frankincense Scented Castor Oil Body Soap

"Thesis Submitted to the School of Chemical and Bio engineering of Addis Ababa Institute of Technology, Department of chemical engineering in Partial Fulfilment of the Requirements for the attainment of the Degree of Master of Science in Chemical Engineering under Process Engineering Stream"

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ABSTRACT

Scented body soap is a type of soap that has been infused with fragrance or essential oils. This gives the soap a pleasant smell that can last for hours after bathing. Scented body soap is available in a wide variety of scents, so there is something to suit everyone's taste. In this thesis work the extraction of oil from castor seeds and infusion of frankincense and their utilization to produce soap has been conducted. The soap has been produced from castor seeds by using pre-treatment of castor seed, extraction of castor oil, neutralization and saponification methods. The extraction and refining of castor oil using stage wise equipment is carry out. In this project, the castor seed oil was extracted at a temperature of 80 and a time of 5 hr. by screw press. And the oil yield of 35% was obtained. Frankincense scented castor oil body soap was synthesized using the cold process method. The soap was characterized using Fourier transform infrared (FTIR) spectroscopy, which revealed the presence of functional groups indicative of fatty acids, esters, alcohols, methyl groups, and aromatic compounds. The soap was also evaluated for its lathering ability and moisturizing properties. The soap was found to be soft, have a good lathering ability, and be moisturizing. The saponification parameters used were temperature in the range of 85 to 90 ^{o}C, water to lye ratio in the range of 1.5 to 2 hr. and castor oil volume in the range of 5 to 10ml. The optimized conditions were at a temperature of 85 ^{o}C, water to lye ratio of 1.75 and a volume of castor 10ml.stable lather, fluffy lather, and moisturizing optimized. The FTIR spectrum of the soap showed peaks at 1745.6 cm−1, 2852.6 cm−1, and 2921.8 cm−1, 719.8 cm−1 which are indicative of the presence of carbonyl (C=O) groups, methyl (CH3) groups, and aromatic (C=C) groups, respectively. The presence of these functional groups is consistent with the expected composition of the soap, which is made with castor oil, frankincense essential oil, and sodium hydroxide. The optimized stable lather, fluffy lather and moisturizing, value found in the entire batch studied were 3.2, 9.7, 6.9, 9.99 respectively. The moisturizing properties of the soap were evaluated by measuring the skin moisture content of participants before and after using the soap. The results showed that the soap significantly increased the skin moisture content of the participants. Overall, the frankincense scented castor oil body soap was found to have good physical and chemical properties. It is a soft, lathering, and moisturizing soap. The soap is also made with natural ingredients, including frankincense essential oil, which is known for its many therapeutic benefits.

Keywords: Castor seed, Castor oil, frankincense, moisturizing

Table of Contents

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LIST OF TABLES

LIST OF FIGURES

Acronyms and Notations

RA	Ricinoleic acid
TFM	Total fatty matter
AIM	Alcohol insoluble matter
FCA	free caustic alkali
ANOVA	Analysis of Variance
WIJS	Bromine chloride method
RGB	Red green blue
S.N	Saponification number
I.V	iodine value
A.V	acid value
Kpa	kilo Pascal
FFA	free fatty acid
ASE	accelerated solvent extraction
SFE	super critical fluid extraction
MAE	microwave assisted extraction
IPN_S	interpenetrating polymer network
PU	polystyrene
FDA	food and drug administration

CHAPTER ONE

1. Introduction

1.1 Background

Throughout the course of history, individuals have been known to enhance their bathing experiences by adding various substances to the water, believed to have beneficial effects. Cleopatra, the renowned Egyptian queen, incorporated mare's milk, honey, and essential oils into her bathing rituals. Historical research has unveiled that soap was employed in ancient Egypt and Babylonia around 5000 years ago. (Gianfaldoni, 2017)

Soap production in ancient times involved a combination of animal fats and alkaline plant ash. It was believed that early civilizations used a mixture of wood ashes and water for washing, and then alleviated any resulting irritation by applying grease or oil. In the first century A.D., Pliny documented the use of a soap made from tallow and wood ashes by Germanic tribes to enhance the brightness of their hair. Historical records indicate that Babylonians began making soap as early as 2800 B.C., and the Phoenicians were familiar with soap by 600 B.C. These early references to soap and its production were primarily focused on its use in cleansing textile fibers like wool and cotton, in preparation for weaving into cloth. (Gianfaldoni, 2017)

The soap industry is a worldwide market that includes a diverse range of products and manufacturers. Soap is an essential item used by people and households worldwide for personal hygiene, cleaning, and various other purposes. Over the years, the global soap industry has seen consistent growth due to factors like population increase, higher disposable incomes, urbanization, and increased awareness of hygiene and cleanliness. Within the industry, there are both large multinational corporations and small-scale local manufacturers, each serving different market segments and meeting the preferences of consumers. The two main categories in the soap industry are bar soap and liquid soap. Bar soap remains the more traditional and commonly used form, while liquid soap has become popular due to its convenience and ease of use. Furthermore, specialty soaps like organic, herbal, antibacterial, and moisturizing soaps have gained popularity as consumers seek specific benefits or preferences. (Gamag, 2017)

The soap industry extends beyond personal care products and encompasses industrial soaps used in various sectors such as hospitality, healthcare, and manufacturing. These specialized industrial soaps are designed for specific applications like laundry, dishwashing, and commercial cleaning. Major soap-producing countries, including the United States, China, Germany, India, and Indonesia, play a significant role in the global soap market. These countries host multinational soap manufacturers as well as local producers. With increasing consumer awareness of sustainability and environmental impact, there is a growing demand for natural and eco-friendly soap products. This has led to the emergence of niche markets for handmade, organic, and cruelty-free soaps, catering to environmentally conscious consumers.

The soap industry is a dynamic and thriving sector on a global scale, driven by the essential need for cleanliness and personal hygiene. It continues to evolve to meet changing consumer preferences, embrace technological advancements, and prioritize sustainability. (Gamag, 2017)

Soap has long been a vital component of personal hygiene in Ethiopia. The introduction of soap in the country has been instrumental in enhancing sanitation practices and promoting general cleanliness. The utilization of soap in Ethiopia has undergone a transformation throughout the years. Initially, traditional techniques involving the use of natural components like plant extracts and animal fats were employed in soap-making. These methods have been handed down through generations and are still observed in certain rural regions. . (Alemayehu, 2022)

In Ethiopia, there has been a notable rise in the availability of commercially manufactured soaps in recent times. Both local and international soap producers have introduced a diverse array of soaps that cater to various needs and preferences. This includes bar soaps, liquid soaps, and specialty soaps with different fragrances and additional ingredients. The introduction of soap has played a significant role in promoting personal hygiene, particularly in urban areas where access to clean water and sanitation facilities is more accessible. Soap aids in the removal of dirt, germs, and bacteria from the skin, thereby reducing the risk of infections and diseases. (Alemayehu, 2022)

Moreover, soap has become an essential component of public health campaigns in Ethiopia. These initiatives aim to increase awareness about the significance of proper hand hygiene, particularly during crucial moments such as before meals and after using the restroom. The introduction of soap in Ethiopia has had a substantial influence on personal hygiene and public health. It has played a crucial role in enhancing cleanliness practices, minimizing the transmission of diseases, and fostering overall well-being in communities throughout the country.

Apart from the underdeveloped traditional Ethiopian economy, the industrial sector, specifically manufacturing, contributes a relatively small portion to the country's overall GDP, placing it among the lowest in the world. In terms of per capita manufacturing value added, Ethiopian Industrial Manufacturing growth has remained stagnant compared to population growth. The future prospects rely heavily on the successful execution of reform measures, including managerial reforms and the establishment of a policy environment that encourages private investment. If these reforms are effectively implemented and accessibility to investment opportunities is improved, the rate of industrialization will be closely tied to the overall economic performance of the country. (Alemayehu, 2022)

According to a recent investigation conducted by the Ministry of Trade, it has been found that more than half of the soap available in the market does not meet the required standards. Iyasu Simon, the director of imports and exports at the Ministry, stated that a total of 1044.92 metric tons of soap samples were examined. The inspection covered various types of detergents and soaps, including liquid and powder detergents as well as solid soaps, that are being sold in shops. The results revealed that over 50% of these products failed to meet the required standards. The import and export director highlighted that most of the soap and detergent products are not in compliance with the standards, citing issues such as inadequate total fatty content, total active matter, and foam testing parameters. Lack of awareness regarding national standards, mislabelling, and limitations in the testing laboratory's capacity are some of the reasons behind this non-compliance. Additionally, the use of unknown and untraceable raw materials poses significant challenges in ensuring product quality. (Astatike, 2019)

Castor seeds and castor oil hold great importance in Ethiopia's agricultural and industrial sectors, presenting economic prospects, traditional remedies, and export potential that contribute to the country's development. Within Ethiopia's rich history, castor soap, also known as Ethiopian black soap, holds a special place. This traditional soap has been utilized by Ethiopian communities for centuries, serving a range of purposes. Crafted from natural components, including castor oil, it is widely recognized for its cleansing and moisturizing attributes.

The introduction of castor soap in Ethiopia has had a significant impact on the country's skincare practices. This soap is renowned for its effectiveness in treating various skin conditions, including acne, eczema, and dryness. Ethiopian black soap is also highly regarded for its exfoliating properties, which help in removing dead skin cells and leaving the skin feeling revitalized and smooth. The production of castor soap in Ethiopia often involves community-based efforts, with local artisans and women's cooperatives playing a crucial role in the process. These skilled individuals meticulously blend castor oil with other natural ingredients such as Shea butter, coconut oil, and various herbs to create the soap's unique formula.

The introduction of castor soap not only encourages the adoption of natural and organic skincare products but also contributes to the support of local communities and the preservation of traditional knowledge. Through the embrace of castor soap, Ethiopians are reconnecting with their cultural heritage and safeguarding traditional practices.

In conclusion, the introduction of castor soap in Ethiopia has brought about positive changes in skincare routines, advocating for the use of natural ingredients and providing support to local artisans. As the awareness of the benefits of this traditional Ethiopian soap increases, its popularity continues to grow.

For centuries, frankincense, also known as olibanum, has held immense value. It is obtained from the resin of the Boswellia tree, predominantly found in Ethiopia and other areas of the Horn of Africa. Ethiopia boasts a rich heritage of frankincense production, with a history spanning thousands of years. The resin is carefully extracted by making precise incisions in the tree's bark, after which it solidifies into resin tears. Frankincense has been utilized in diverse cultural, religious, and medicinal applications, renowned for its aromatic qualities and perceived spiritual benefits. (Kokoszko, 2021)

In Ethiopia, castor oil is derived from the seeds of the castor bean plant, scientifically known as Ricinus communis. This plant is native to East Africa, including Ethiopia. For centuries, castor oil has been renowned for its healing properties, especially in the realm of skincare. Its moisturizing, calming, and nourishing attributes have made it a valuable component in various skincare products. (Kokoszko, 2021)

By blending the captivating fragrance of frankincense with the nourishing advantages of castor oil, the Frankincense Scented Castor Soap harmoniously merges the finest qualities of these two elements. This exquisite soap not only purifies your skin but also offers a sensory journey that transports you to the timeless landscapes of Ethiopia. It stands as a splendid homage to Ethiopia's abundant cultural legacy and the cherished natural treasures passed down through generations.

1.2 Statement of the problem

The growing demand for sustainable and eco-conscious personal care products has spurred research into natural ingredients like frankincense and castor oil for soap production. Frankincense, an aromatic resin native to Ethiopia, has been traditionally used for its medicinal and cosmetic properties, while castor oil, derived from the castor bean plant, is known for its moisturizing and lathering qualities. Several studies have explored the synthesis and characterization of frankincense-scented castor oil soap, demonstrating its potential as a natural alternative to commercial soaps.

This research endeavors to bridge this gap by undertaking a comprehensive investigation into the formulation and production process of Frankincense-scented castor oil soap in Ethiopia. The study aims to develop a soap formulation that incorporates Frankincense as a fragrant and culturally significant ingredient while harnessing the unique properties of castor oil to enhance the soap's quality. Furthermore, the research will involve the rigorous assessment of the physical and chemical properties of the soap, including its cleansing efficacy, scent retention, and overall performance. By doing so, this study seeks to determine the feasibility and potential of Frankincense-scented castor oil soap as a sustainable and culturally relevant personal care product in Ethiopia. The findings of this research will not only contribute to the advancement of soap production methods using locally available resources but also promote sustainability and cultural preservation in the personal care product industry.

1.3. Objective

General objective

The general objective of this study is to Extract and characterize oil from castor seeds to produce frankincense scented castor soap.

Specific objective

- To extract castor oil by screw press and characterize oil from castor seed (viscosity, PH value, free fatty acid value and specific gravity).
- To optimize the saponification process parameters on soap production from castor seed oil (water to lye ratio, mixing time and castor oil concentration).
- To characterize the produced soap from castor seed oil and frankincense (viscosity and foam ability, volatile matter, moisture content, total fatty matter content (TMF), alkali content and pH in soap) and compare the values or standards with commercial products.
- To Enhance the acceptability of frankincense-scented castor oil soap

1.4 Significance of the study

Frankincense scented castor oil soap lies in its cultural, skincare, aromatherapy, economic, and sustainability implications. It offers an opportunity to create a product that reach Ethiopia's heritage, provides exceptional skincare benefits, enhances well-being, and supports sustainable practices. It offers a distinctive sensory experience, combining the captivating aroma of frankincense with the moisturizing properties of castor oil. Castor oil is known for its moisturizing, soothing, and nourishing properties, making it beneficial for various skin types. Frankincense is associated with potential skincare benefits such as anti-aging and anti-inflammatory properties. Studying the synthesis of this soap allows for exploring the potential synergistic effects of these ingredients, leading to the development of a soap that not only cleanses but also promotes healthier skin. Understanding the formulation and characteristics of this soap can enhance user satisfaction and contribute to the development of unique and luxurious skincare products. The study can shed light on sustainable practices for frankincense resin extraction and castor oil production. By promoting responsible sourcing and environmentally friendly techniques, it can contribute to the conservation of the

Boswellia tree species and the protection of natural resources, ensuring their availability for future generations.

1.5 Scope of the study

Investigating different extraction methods for frankincense resin and castor oil to determine the most efficient and sustainable approaches. This may involve exploring traditional extraction techniques used in Ethiopia and evaluating modern extraction methods for optimal yield and quality. Developing soap formulations that incorporate frankincense and castor oil, considering factors such as ingredient ratios, compatibility, stability, and scent retention. This includes studying the impact of different formulation variables on the sensory properties, lathering ability, and overall quality of the soap. Conducting comprehensive characterization of frankincense resin, castor oil, and the resulting soap. This involves analysing the chemical composition, physical properties, and potential bioactive components of the ingredients. It may also include assessing the fragrance stability, pH, moisture content, and other relevant parameters of the soap.

CHAPTER TWO

2. Literature review

2.1. Introduction

2.1.1 Evaluation and Determination of different type of oils for soap production

In soap making, all fats and oils are composed of a combination of glycerol and fatty acid compounds, which naturally exist as triglycerides. From the perspective of soap makers, the most significant fatty acids are stearin, palmitin, olein, and laurin. Firmness in soap is attributed to the presence of stearin and palmitin, which are typically solids at room temperature. The higher the percentage of these compounds, the harder the oil becomes and the higher its melting point. On the other hand, if the oil contains a significant amount of olein, which is liquid at normal temperatures, it will result in a softer consistency. The soap-making qualities of fats and oils can be determined by the molecular weights of their fatty acids. In the case of naturally occurring saturated fatty acids in fats or oils, as the molecular weight increases, certain properties change. (Donkor, 1986)

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.

a) Nut oils: such as coconut oil, are characterized by a high proportion of fatty acids with low molecular weight, particularly lauric and stearic acid. These oils play a crucial role in generating foam when used in toilet soaps. They readily undergo saponification with strong alkaline solutions. Once the saponification process begins, it progresses quickly and generates heat. To form soap grains, these oils require a significant amount of concentrated brine solution (1648"Be), and the resulting soaps tend to contain more salt compared to other types of soaps. Nut oils are well-suited for making cold process soaps.

b) Hard fats: such as palm oil, animal tallow, and hydrogenated fats, contains significant amounts of palmatic and stearic acids. These oils yield soaps that lather slowly, but the resulting lather is more long-lasting compared to soaps made from nut oils. In the soap-making process, these fats are first saponified with weak alkaline solutions, and in the final stages, stronger alkali solutions are used.

C) Soft oils: such as groundnut oil, cottonseed oil, fish oil, and olive oil, contain significant amounts of unsaturated acids, including oleic, linoleic, and linoleic acids. The soap-making properties of these oils depend on their fatty acid composition, as well as the physical and chemical properties of the acids. When used alone in soap making, these oils do not yield very hard soaps. They are often blended with nut oils. Soaps made from these oils produce abundant lather and exhibit excellent detergent properties. Soap making involves a specific chemical breakdown of fats and oils into their constituent parts, namely fatty acids and glycerol. The fatty acids react with a small amount of caustic soda, potash, or other bases to form soap, while glycerol remains unreacted. (Donkor, 1986)

2.1.2. Oil- Seeds and Nuts

Oil-rich seeds and nuts are abundant in various parts of certain tropical and sub-tropical plants, including their roots, stems, fruits, and leaves. These plants are typically cultivated as annual crops and serve as the primary source of vegetable oil for both domestic and industrial purposes. Groundnut, coconut, Shea nut, castor, sunflower, sesame, and oil palm are among the most widely grown oil seeds and nuts in tropical, subtropical, and temperate regions. (Onwualu, 2005).

Groundnut (Arachis hypogaea L.)

Groundnut, also known as peanut, is a widely cultivated oil nut that is grown annually on approximately 19 million hectares in tropical, subtropical, and warmer temperate regions. It is primarily cultivated for its edible oil and protein-rich kernels or seeds. Groundnut thrives best in sandy loam soil and is considered the second most important source of vegetable oil globally. In 1985, the worldwide output of groundnut resulted in around 14.4 million tons of groundnut kernels, equivalent to the production of approximately 21 million tons in their shells (assuming a shelling percentage of 70%), with approximately 7.2 million tons of oil extracted.

The yield of groundnut kernels typically ranges from 0.5 to 4.0 tons per hectare. In developing countries, which account for 80% of the crop production, the average yield is approximately 1 ton per hectare. India, China, USA, Brazil, Senegal, and Nigeria are among the major producers of groundnut worldwide.

Coconut (Cocos nucifer L.)

Coconut cultivation is widespread across the globe, primarily in the humid tropical regions, often in proximity to the seashore. The leading coconut-producing countries include the Philippines, Indonesia, India, Malaysia, and Thailand. In tropical Africa, the crop is predominantly grown in Mozambique, Cote d'Ivoire, Tanzania, and Nigeria. (Onwualu, 2005)

After pollination, a mature coconut plant produces fruits. These fruits are known as drupes and are composed of a seed, which is the coconut itself, surrounded by a thick fibrous husk. The husk, also known as the mesocarp, is covered by a thin, leathery layer. Within the hard and thick shell of the coconut, known as the endocarp, there is a layer of firm, white, oily endosperm called copra. Additionally, the central cavity of the coconut is partially filled with sugary coconut water..

Sheanut (Butyrospermum paradoxum (gaertn.F.) Hepper)

In Africa, sheanut trees grow naturally in the wild. The regions with the largest populations of sheanut trees are Mali, Burkina Faso, Northern Togo, Ghana, Niger, Nigeria, Cote d'Ivoire, and Benin. Burkina Faso has the potential to produce approximately 460,000 tons of sheanuts annually, while the annual production in Cote d'Ivoire and Mali ranges from 17,000 to 20,000 tons. (Onwualu, 2005)

Palm(Arecales)

Palm oil is a significant and versatile raw material extensively used in both the food and non-food industries. It has gained popularity worldwide and currently ranks as the second most consumed oil, thanks to its competitive pricing compared to other edible oils. The oil palm tree has been around since ancient times and every part of the tree holds economic and domestic value. While it is found in South America, Southeast Asia, the South Pacific, and to a lesser extent in other tropical regions, historically, the palm tree was primarily confined to West and Central Africa, where it grew in the wild, semi-wild, and cultivated states.

Palm oil is among the 17 key oils and fats that are produced and traded globally. Over the course of the last 40 years, palm oil has experienced remarkable growth and has become the fastest-growing oil worldwide. It is expected to become the largest oil produced globally, although it currently holds the second position, trailing behind soybean oil. (Mohammadreza Koushki, 2008)

Olive oil

Olive oil is widely used as a base oil in soap making today. It has been a popular choice for centuries, and most soap makers incorporate at least some amount of olive oil in their blends. Olives belong to a fruit category known as drupes, which are fleshy fruits with a hard seed at the centre. Initially, the olives are crushed and ground to form a paste. The oil is then separated from the paste using various methods. The oil obtained from the very first crush is known as "virgin" olive oil. The residue left after the initial extraction is called "pomace."

- Oleic 55-83%
- Palmitic 7.5-20%
- Linoleic 3.5-21%
- Stearic 0.5-5%

Olive oil provides soap with desirable properties such as hardness, stable lather, a slippery feel, conditioning, and moisturizing effects. It has the ability to attract external moisture to the skin, helping to maintain its softness and suppleness. In soap making, pomace olive oil contains a higher proportion of unsaponifiable ingredients. This slightly alters its SAP (Saponification Value) and gives the oil, as well as the soaps made with it, a greenish color. For soap making purposes, pomace oil is often preferred over grade A olive oil. (Dimitrios Boskou, 2006)

2.1.3. Physicochemical Analysis of some selected Industrial oils

Table 2-1 species name and oil content of edible and non-edible plant (Marchetti, 2017)

Type of oil	Common name	Species name	Oil content of seed /kernel (wt. %)	
			seed	Kernel
Edible	Coconut	Cocosnucifera L.	63-65	63.1(±2.8)
	Corn	Zea mays	24.44	-
	Hemp seed	Cannabis sativa L	22-38	-
	Mustard seed	Brassica nigra	33	-
	Olive	Olea europaea	45-70	-
	Palm	Elaeis guineensis	30-60	-
	Peanut	Arachishypgea L.	45-55	47-61
	Pumpkin seed	Cucurbita maxima	31.5	43.69(±3.92)
	Rape seed	Brassicanapius	38-46	-
	Rice bran	Oryza sativa	15-23	-
	Saf flower seed	Carthamustinctorius	35	-
	Sesame seed	Sesamum indicum	58	-
	Soybean	Glycine max	18-20	-

	sunflower	Helianthus annuus	25-35	-
Non-edible	Castor	Ricinus communis l.	45-50	-
	Cotton seed	Gossypium hirsutum L.	18-25	31.42
	Desert date	Balanites aegyptiaca	45-50	36-47
	Jatropha	Jatropha curcas L.	20-60	40-60
	Jojoba	Simmondisa chincnsis	45-50	-
	Karanja	Pongamia pinnata	30-40	30-50
	Linseed	Linum usitatissimum	35-45	-
	Mahua	Madhuca indica	35-40	50
	Neem	Azadirachta indica	20-30	25-45
	Polanga	Calophyllum inophyllum	65	22
	Rubber seed	Hevea brasiliensis	40-60	40-50
	Tobacco	Nicotiana tabacum L.	30-43	-
		Nicotiana tabacum	36-41	17
		Zanthoxylum bungeanum	24-28	25
	Tung	Vernicia montana	16-18	-
	Ethiopian mustard	Brassica carinata	42	2.2-10.8
	Sea mango	Cerbera odollam	54	6.4
	Croton oil plant	Croton tiglium	30-45	50-60

2.2. Frankincense (boswellia thurifera)

Similar to its well-known biblical counterpart, myrrh, frankincense oil is derived from resin, specifically the dried sap of trees native to Somalia. The resin is obtained from the deciduous boswellia thurifera, a member of the burseraceae family, which sheds its leaves annually. During its blooming phase, the tree is lush with oblong leaves that alternate towards the upper branches. To extract the sap, a deep incision is made on a section of the tree trunk that has been stripped of its bark, resulting in the release of a milky sap-like substance. (Jones, 2011)

Recently, increasing interest in natural dietary and therapeutic preparations used as dietary supplements has been observed. One of them is frankincense. This traditional medicine of the East is believed to have anti-inflammatory, expectorant, antiseptic, and even anxiolytic and anti-neurotic effects. (Ali Ridha Mustafa Al-Yasiry, 2016)

This is just the beginning of long process that can take three months from start to end. Because the sap oozes slowly and is allowed to harden on the tree by exposing it to air before the original incision is late deepened.in three months the sap usually has attained the required degree of consistency, hardening into yellowish tear shaped oleo gum resin that is scraped and carried away in baskets. After this, the oil is extracted by steam distillation. (Jones, 2011)

2.2.1 Physio-chemical Characteristics of frankincense

Frankincense essential oil is a base note with woody, balsamic-yet spicy smell.it has a pale yellow to pale green hue. Also, bear in mind that because frankincense oil comes from tree sap that tends to be very thick.it is very visicous.the oils components include incense acetate, which is said to lower anxiety. According to physiochemical research, oil of Boswellia resin contains.

Table 2-2. Physical properties of frankincense (Botros R. Mikhaeil, 2002)

Colour	pale yellow
Odour	slightly spicy and lemony odor
specific gravity(25 ºC)	0.875
refractive index (20 ºC)	1.446
acid value	12.05

ester value	127.46
saponification value	139.51

2.2.2 Applications of frankincense oil

a) In Aromatherapy

- Acne, Anxiety, Colds, Coughs, Indigestion and Ulcers
- Support healthy cellular function
- Increase blood flow and circulation
- Reduce inflammation
- Reduce skin discoloration and imperfections
- Promote feelings of wellness and relaxation
- Balance hormones
- Improve memory
- In some studies, have been shown to suppress proliferation of cancer cells
- have an anti-microbial activity for skin and surfaces
- Helps Reduce Stress Reactions and Negative Emotions

b) In Perfumery & Cosmetics

Frankincense oil is used in perfumery and cosmetics products.Although frankincense oil is widely used in aromatherapy for its aroma in incense burners, it seems to be one of the more intense therapeutic grade essential oils.in 2009, researchers from the university of Oklahoma health sciences center and Oklahoma City veteran’s affairs medical center published findings showing that frankincense oil can kill cells that cause of the bladder. (Jones, 2011)

Frankincense extracted from the *Boswellia serrata* (BS) tree have numerous potentials and are also used in traditional medicine in many countries. BS extracts exhibit antibacterial, antifungal and anticancer activities. (Philip, 2020)

2.3. Methods of Essential oil Extraction

Essential oil is produced from the resin of *Boswellia* by the following Steam Distillation, Hydro Distillation, Supercritical Fluid Extraction, Microwave-Assisted Hydro distillation and Frankincense Infusion. Each technique will be discussed below.

2.3.1 Steam Distillation

Steam distillation is used in the manufacture and extraction of essential oils, the botanical material is placed in a still and steam is forced over the material. The hot steam helps to release the aromatic molecules from the plant material since the steam forces open the pockets in which the oils are kept in the plant material. The molecules of these volatile oils then escape from the plant material and evaporate into the steam. The steam is produced at greater pressure than the atmosphere and therefore boils at above 100^0C which facilitates the removal of the essential oil from the plant material at a faster rate and in so doing prevents damage to the oil. (Najam, 2018)

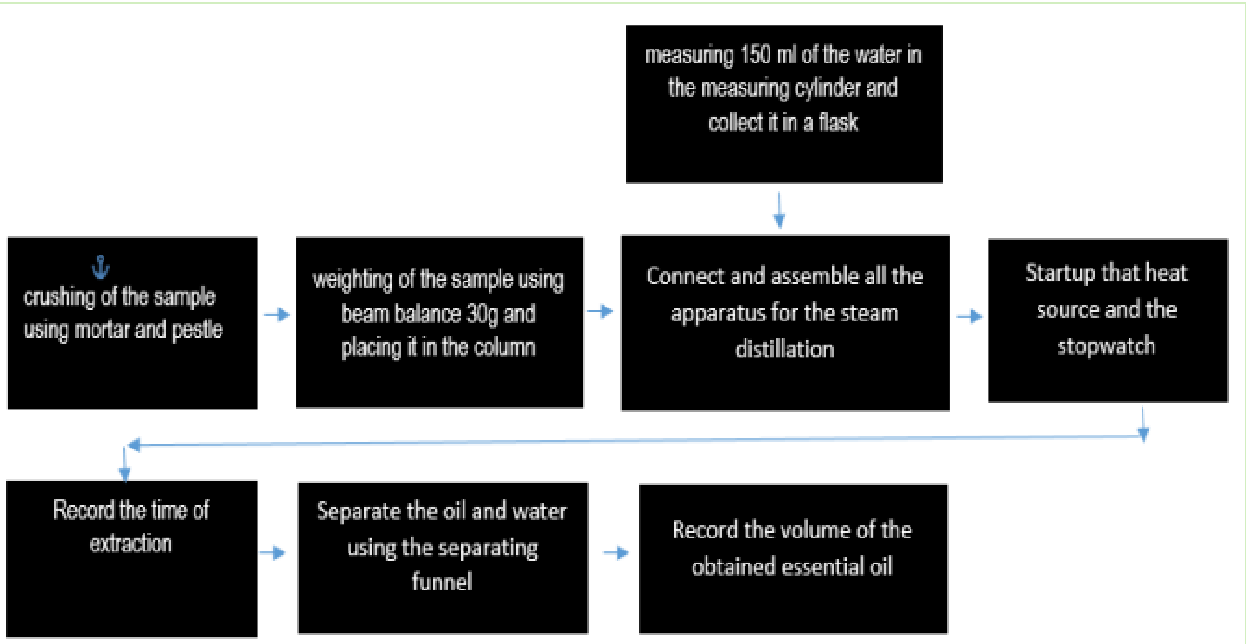


Figure 2-1 flow diagram of steam distillation (Abdalla Ayoub Abdalla, 2020)

2.3.2 Hydro distillation

Using frankincense essential oil obtained from hydro distillation of *Boswellia sacra* gum resins, our goals are to determine optimal preparation conditions that induce potent cytotoxic effects in cultured human pancreatic cancer cells, to establish a relationship between *Boswellia sacra* essential oil chemical composition and anti-cancer activity, and to evaluate *Boswellia sacra* essential oil anti-tumor activity *in vivo*. Our results demonstrated that anti-tumor activity of *Boswellia sacra* essential oil depends upon hydro distillation duration and hydro distillation temperature; and high molecular weight compounds in essential oil may be responsible for its anti-tumor properties.

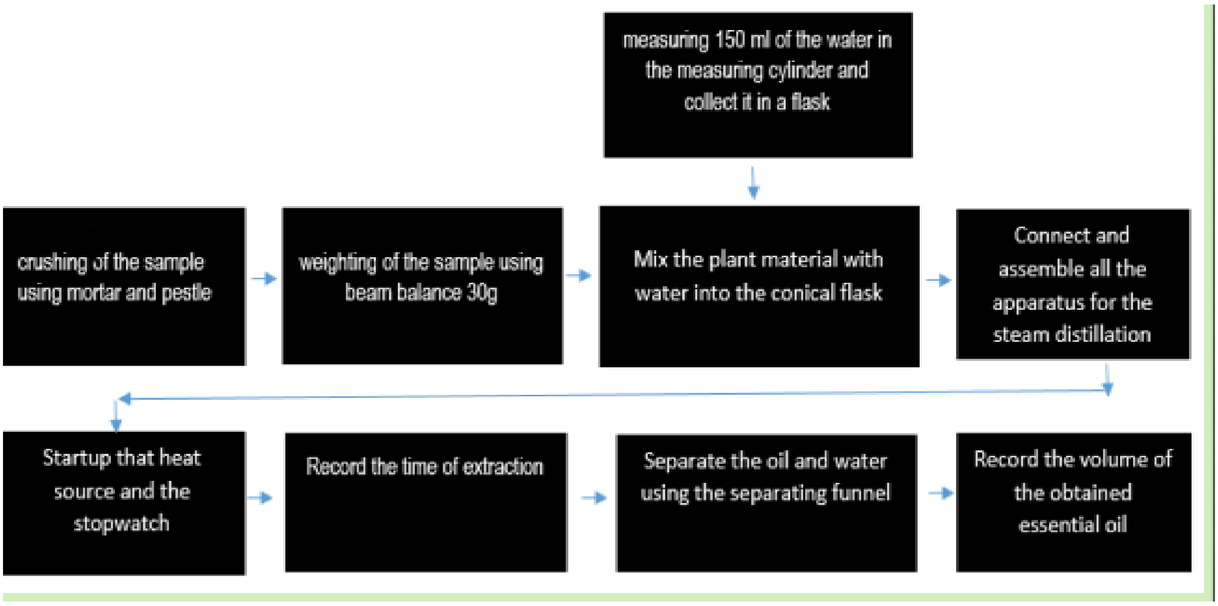


Figure 2-2 hydro distillation process (Abdalla Ayoub Abdalla, 2020)

2.3.3. Supercritical Fluid Extraction

Supercritical fluid extraction (SFE) of essential oils is a modern technique, currently being applied in the process industry, which competes with conventional processes such as steam distillation and organic liquid (solvent) extraction. It has been widely accepted by many investigators that SFE provides a rapid and quantitative method for extracting essential oils from aromatic plants that compares favourably with steam distillation. (Abdalla Ayoub Abdalla, 2020)

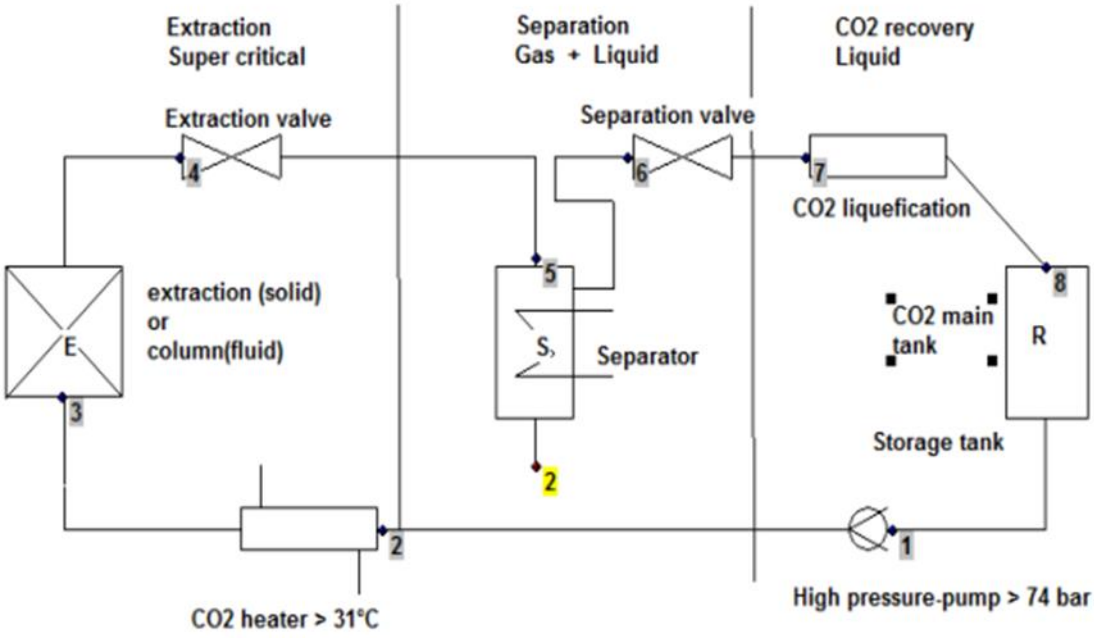


Figure 2-3 supercritical fluid extraction (Abdalla Ayoub Abdalla, 2020)

2.3.4 Microwave-Assisted Hydro distillation

Microwave energy is a superior alternative to several thermal applications owing to its efficient volumetric heat production. The volumetric heating or heating of the bulk as opposed to transferring heat from the surface, inwards, is more efficient, uniform and less prone to overkill or supererogation. Controllability is by far the greatest advantage of microwaves over conventional thermal technologies. In processing applications, the ability to instantaneously shut 18 the heat source makes enormous difference to the product quality and hence the production economics. The raw material is heated directly by microwaves and this brings about quality consistency and minimizes the impact on the environment as opposed to using fossil fuels or less efficient, indirect electrical heating systems. (Abdalla Ayoub Abdalla, 2020)

2.3.5. Frankincense Infusion

Frankincense is a resin that comes from trees of the genus Boswellia. When the bark is cut or tapped, it exudes a syrupy substance that hardens into what is known as the tears. This resin is rich in a complex variety of chemical compounds. Studies are on-going, but it is suggested that it may have great value for human health and may hold, at least in part, cure for cancer. (lisalise, 2019)

A frankincense extract is far superior to using essential oils, mainly because with an oil extract you get to have the benefit of the boswellic acids. These acids are heavy terpenes which do not come over into the essential oil. It is thought that boswellic acids are effective at treating inflammation and reversing effects associated with photo ageing. (lisalise, 2019)

To make frankincense infusion, grind it in a mortar and freeze. Heat oil in a pot of hot water and add ground resin. Simmer for 45-36 hours. Remove from heat, steep for a few days, or filter immediately. Filter the infusion through a cheese cloth or coffee filter.. (lisalise, 2019)

2.4. Castor oil (Ricinus communis L.)

Castor oil has long been used commercially as a highly renewable resource for the chemical industry. It is a vegetable oil obtained by pressing the seeds of the castor oil plant (Ricinus communis L.) that is mainly cultivated in Africa, South America, and India. Major castor oil-producing countries include Brazil, China, and India. This oil is known to have been domesticated in Eastern Africa and was introduced to China from India approximately 1,400 years ago.4 India is a net exporter of castor oil, accounting for over 90% of castor oil exports, while the United States, European Union, and China are the major importers, accounting for 84% of imported castor oil. Castor plant grows optimally in tropical summer rainfall areas. It grows well from the wet tropics to the subtropical dry regions with an optimum temperature of 20°C–25°C. The high content of the oil in the seeds can be attributed to the warm climate conditions, but temperatures over 38°C can lead to poor seed setting. Additionally, temperatures low enough to induce the formation of frost is known to kill the plant.

2.4.1 Castor Oil and its Properties

Table 2-3 physical properties of castor oil (FC, 2011)

PHYSICAL PROPERTIES	
Viscosity (centistokes)	889.3
Density (g/mL)	0.959
Thermal conductivity (W/m°C)	4.727
Specific heat (kJ/kg/K)	0.089
Flash point (°C)	145
Pour point (°C)	2.7
Melting point (°C)	-2 to -5
Refractive index	1.480

The unique structure of castor oil offers interesting properties, making it appropriate for various industrial applications. Castor oil is known to consist of up to 90% ricinoleic, 4% linoleic, 3% oleic, 1% stearic, and less than 1% linolenic fatty acids. Castor oil is valuable due to the high content of ricinoleic acid (RA), which is used in a variety of applications in the chemical industry. The hydroxyl functionality of RA makes the castor oil a natural polyol providing oxidative stability to the oil, and a relatively high shelf life compared to other oils by preventing peroxide formation. The presence of the hydroxyl group in RA and RA derivatives provides a functional group location for performing a variety of chemical reactions including halogenation, dehydration, alkoxylation, esterification, and sulfation. As a result, this unique functionality allows the castor oil to be used in industrial applications such as paints, coatings, inks, and lubricants. (Vinay R. Patel, 2016)

2.4.2 Uses of castor oil

Castor oil is a multipurpose vegetable oil that people have used for thousands of years. It's made by extracting oil from the seeds of the Ricinus communis plant. These seeds, which are known as castor beans, contain a toxic enzyme called ricin. However, the heating process that castor oil undergoes during production deactivates the ricin, allowing the oil to be used safely. Castor oil has a number of medicinal, industrial, and pharmaceutical uses.It's commonly used as an additive in foods, medications, and skin care products, as well as an industrial lubricant and biodiesel fuel component. A powerful laxative: Perhaps one of the best-known medicinal uses for castor oil is as a natural laxative. It's classified as a stimulative laxative, meaning that it increases the movement of the muscles that push material through the intestines, helping clear the bowels. Castor oil is approved by the Food and Drug Administration (FDA) as a simulative laxative (2Trusted Source). stimulative laxatives act rapidly and are commonly used to relieve temporary constipation or to clean out the bowel before medical procedures. Here's generally how it works: When you consume castor oil by mouth, it's broken down in the small intestine, releasing ricinoleic acid, the main fatty acid in castor oil. The ricinoleic acid is then absorbed by the intestine, stimulative a strong laxative effect. Several studies have shown that castor oil can relieve constipation.

Natural moisturizer: Castor oil is rich in ricinoleic acid, a monounsaturated fatty acid. These types of fats can be used to moisturize the skin. They act as occlusive moisturizers, which prevent or reduce water loss through the outer layer of the skin. Castor oil is used in cosmetics to promote hydration. Manufacturers often add it to products like lotions, makeup, and cleansers. You can also use this rich oil on its own as a natural alternative to store-bought moisturizers and lotions. Many popular moisturizing products found in stores contain potentially harmful ingredients like preservatives, perfumes, and dyes, which may irritate the skin and harm overall health.

Fuel and biodiesel: Castor is considered to be one of the most promising nonedible oil crops, due to its high annual seed production and yield, and since it can be grown on marginal land and in semiarid climate. Few studies have been done regarding castor fuel-related properties in pure form or as a blend with diesel fuel, primarily due to the extremely high content of RA.

Polymer materials: Castor oil and its derivatives can be used in the synthesis of renewable monomers and polymers. In one study, castor oil was polymerized and cross-linked with sulphur or disocyanates to form the vulcanized and urethane derivatives, respectively. In another study, full-interpenetrating polymer networks (IPNs) were prepared from epoxy and castor oil-based polyurethane (PU), by the sequential mode of synthesis.

Soaps, waxes, and greases: Castor oil has been used to produce soaps in some studies. Some studies also utilize castor oil in waxes. One study by Dwivedi and Sapre utilized castor oil in total vegetable oil greases. Total vegetable oil greases are those in which both the lubricant and gallant are formed from vegetable oil. Their study utilized a simultaneous reaction scheme to form sodium and lithium greases using castor oil. (Vinay R. Patel, 2016)

Lubricants, hydraulic, and brake fluids: Castor oil has also been used for developing low pour point lubricant base stocks through the synthesis of acyloxy castor polyol esters. Low pour point property helps to provide full lubrication when the equipment is started and is easier to handle in cold weather. (Vinay R. Patel, 2016)

Fertilizers: Production of castor oil generates two main by-products: husks and meal. For each ton of castor oil, 1.31 tons of husks and 1.1 tons of meal are generated. A study by Lima et al62 showed that blends of castor meal and castor husks used as fertilizer promoted substantial plant growth up to the dose of 4.5% (in volume) of meal. However, doses exceeding 4.5% caused reduction in plant growth and even plant death. (Vinay R. Patel, 2016)

2.5. Extraction methods

Preparation of feedstock and various oil extraction methods are discussed in the following parts.

2.5.1 Feedstock preparation

- **Castor Seed Samples**

For this study castor seeds were purchased from Eden green. They collected from southern region of Ethiopia especially called wendogenet.

- **Castor seed preparation (pre –treatment)**

The pre-requisite for oil extraction is seed preparation. The preparation of seeds involves removal of outer layers of the fruit to expose the kernels or seeds, and it's drying to reduce moisture content.

- **Castor seed cleaning**

The seeds are removed from the fruits, and the fruits that do not naturally open are opened by hand. The separated seeds or kernels are then sifted, cleaned, and stored at normal room temperature.

- **Castor seed drying**

Seeds can be dried either by using an oven or by exposing them to the sun until they reach the desired moisture level. The kernels or seeds need to be prepared in a manner that ensures they have the ideal moisture content for extracting a high amount of oil. For example, it has been discovered that preparing the seed kernels of the beauty leaf (C. inophyllum) to a moisture content of 15% resulted in the highest oil yields when using both mechanical and solvent extraction techniques. It is important to closely monitor the drying process by periodically weighing the trays throughout the day, if possible. Once the desired level of dryness is achieved, the trays should be stored in a room with refrigeration facilities Mechanical expellers or presses can be supplied with either intact seeds, kernels, or a combination of both. However, it is common practice to use only seeds when using these methods. On the other hand, when it comes to chemical extraction, only kernels are typically utilized. (Jahirul MI, 2013)

2.5.1 Oil Extraction methods

(Bhuiya M, 2016) Once the raw material has been prepared, it is ready for the extraction of oil. There are three primary methods that have been identified for this purpose:

a) mechanical extraction

b) chemical or solvent extraction

c) enzymatic extraction

In addition, accelerated solvent extraction (ASE), supercritical fluid extraction (SFE), and microwave-assisted extraction (MAE) methods are also utilized, although they are not as widely recognized or commonly employed as the previously mentioned methods. It has been noted that mechanical pressing and solvent extraction are the predominant methods employed for commercial oil extraction. The primary outputs of the extraction process are crude oil, while significant by-products include seed or kernel cakes. These seed cakes can serve various purposes, such as soil enrichment as fertilizers, feed for poultry, fish, and swine, and some oil cakes even find applications in fermentation and biotechnological processes. (Warra, 2011)

2.5.1.1 Mechanical oil extraction

The mechanical press method is the traditional and widely used technique for oil extraction. It involves the use of either a manual ram press or an engine-driven screw press. Oil extraction efficiencies determined from data reported in recent studies generally fall within these ranges, with engine-driven screw presses exhibiting a broader efficiency range of 70-80%. The oil obtained through mechanical presses requires additional processing, such as filtration and degumming, to produce a purer end product. Another limitation of conventional mechanical presses is that their design is optimized for specific types of seeds, which can affect the oil yield when used with different seedsIt has been observed that pre-treating seeds before using a mechanical extractor can enhance the oil recovery. For example, by cooking castor seeds in water at 70°C for one hour and utilizing screw pressing, Beerens achieved an oil yield of 89% after a single pass and 91% after a double pass. In comparison, the oil yield recovery for untreated seeds was 79% and 87%, respectivelyHence, in order to enhance the oil extraction yield, various alternative methods have been recently suggested, including solvent extraction, enzymatic extraction, and microwave-assisted techniques.

2.5.1.2 Solvent oil extraction (chemical extraction)

Solvent extraction, also referred to as leaching, is the method by which oil is extracted from a solid material using a liquid solvent The most commonly used solvent for oil extraction, which yields the highest oil yield, is n-hexane. This method has been employed to extract oil from Australian native beauty leaf seeds (Calophyllum inophyllum). However, despite the low cost, the mechanical screw press method is ineffective due to its relatively lower oil yields. In contrast, the chemical oil extraction method has proven to be highly effective due to its ability to consistently achieve high oil yields. The oil extraction process involved using a Soxhlet extractor and n-hexane (with a boiling point of 40-60°C) to extract oil from 5.0g of ground seed kernels of Indigenous castor. After the six-hour extraction process, the oils were obtained by removing the solvent under reduced temperature and pressure. To eliminate any excess solvent used in the oil, refluxing at 70°C was performed. The extracted seed oil was then stored in a freezer at -2°C for subsequent physicochemical analysis. In the case of cotton seeds, apart from solvent extraction, the crude cotton seeds were also clarified using a 0.5M NaOH solution. The oil yield was determined by completely distilling most of the solvent on a heating mantle, and the resulting oil was transferred to a measuring cylinder. The measuring cylinder is then placed over water bath for complete evaporation of solvent for about 2-3 hours in accordance with the method reported and volume of the oil was recorded and expressed as oil content(%) as follow :

Oil content (%) = oil weight/ sample weight*100

2.5.1.3 Enzymatic extraction

As an alternative to solvent extraction (SE), aqueous extraction has been employed. This method relies on the low solubility of triglycerides in aqueous media. Unlike SE, this method does not encounter the same issues, and it yields high-quality oil. However, the drawback is that the yields are typically very low, as a significant portion of the oil bodies remain within the seed structure. The solution to this issue involves breaking down the structures that trap the oil bodies, namely the cell walls and the membranes of liposome bodies. To achieve this objective, glycosidase and proteases can be employed. While in certain cases, using a single enzyme may yield satisfactory results, the most effective approach appears to be using a combination of enzyme cocktails that can target various components of the cell walls, such as pectic polymers, lignin, cellulose, hemicellulose, and proteases. (Pablo Díaz-Suárez a 1, 2021)

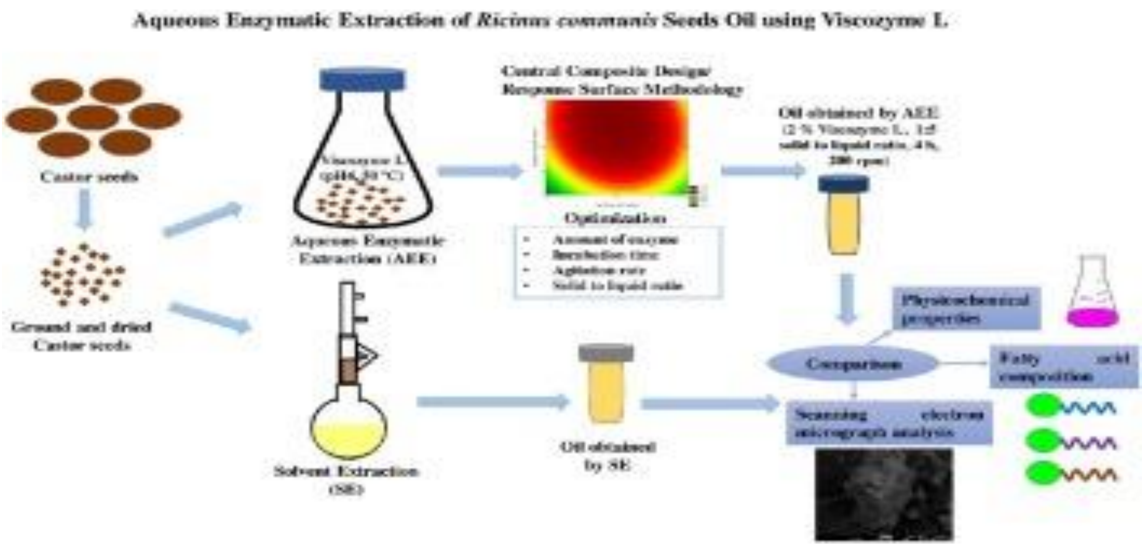


Figure 2-4 enzymatic extraction of castor seed (Pablo Díaz-Suárez a 1, 2021)

2.6 Castor oil refining

2.6.1 Castor Seed Sample Preparation for Oil Content Determination

Castor oil seeds typically contain around 30% to 50% oil by weight. The extraction of castor oil from castor beans can be carried out through mechanical pressing, solvent extraction, or a combination of both methods. Once harvested, the seeds are dried to facilitate the splitting open of the seed hull, which allows the seed to be released. The extraction process starts with the removal of the hull from the seeds, which can be done mechanically using a castor bean dehuller or manually by hand.

Figure 2-5 Screw press

Afterwards, the cleaned castor seeds are further processed to eliminate any impurities, such as sticks, stems, leaves, sand, or dirt. A set of rotating screens or reels is typically employed to separate these materials. Additionally, magnets placed above the conveyer belts can effectively remove any iron contaminants to facilitate extraction the seeds can undergo a heating process to enhance the hardness of their interior. This involves warming the seeds in a steam-jacketed press to remove moisture. The hardening process plays a crucial role in enhancing the efficiency of extraction. Following this, the cooked seeds are dried before commencing the extraction process. A continuous screw or hydraulic press is utilized to crush the castor oil seeds, facilitating the extraction of the oil (as illustrated in Figure 5The initial stage of the extraction process is referred to as prepressing, which typically involves the use of a screw press known as an oil expeller. The oil expeller is a continuous screw press that applies high pressure to extract the oil. While mechanical pressing can be performed at low temperatures, it results in only about 45% oil recovery from the castor beans. However, higher temperatures can enhance the efficiency of extraction. By employing high-temperature hydraulic pressing, yields of up to 80% of the available oil can be achieved in the extraction process.

The temperature during the extraction process can be regulated by circulating cold water through a pressing machine specifically designed for cold pressing the seeds. Cold-pressed castor oil exhibits lower acid and iodine content and has a lighter color compared to solvent-extracted castor oil. Once the extraction is complete, the oil is collected and filtered. The filtered material is then mixed with fresh seeds for subsequent extraction. This process is repeated multiple times, allowing the bulk filtered material to accumulate and undergo several extraction cycles in combination with new bulk material.

Eventually, this material is expelled from the press and referred to as castor cake. The castor cake from the press contains up to approximately 10% castor oil content. After crushing and extracting oil from the bulk of the castor oil seeds, further extraction of oil from the leftover castor cake material can be accomplished by crushing the castor cake and by using solvent extraction methods.

To extract oil from the castor cake, a Soxhlet apparatus or a commercial solvent extractor is employed. Organic solvents like hexane, heptane, or petroleum ether are commonly used to dissolve and remove the remaining oil that is still inaccessible within the residual seed bulk. This extraction process effectively extracts most of the residual oil from the castor cake. (Vinay R. Patel, 2016)

2.6.2 Castor oil filtration/purification

After the oil has been extracted using a press, there may still be impurities present in the extracted oil. To eliminate these remaining impurities, filtration systems are commonly utilized. These filtration systems are capable of removing particles of various sizes, dissolved gases, acids, and even water from the oil. The filter press is the typical equipment employed for this filtration process. Initially, crude castor seed oil has a pale yellow or straw color, but it can be rendered colorless or nearly colorless through refining and bleaching. Furthermore, the crude oil may possess a distinct odor, which can be eliminated through the deodorization process during refining. (Vinay R. Patel, 2016)

2.6.3 Castor oil refining

The crude or unrefined oil undergoes a refining process after filtration. This refining process aims to eliminate impurities such as colloidal matter, phospholipids, excess free fatty acids

(FFAs), and coloring agents from the oil, ensuring its stability during extended storage. The refining process involves several steps, including degumming, neutralization, bleaching, and deodorization. Degumming involves adding hot water to the oil, allowing the mixture to settle, and then removing the aqueous layer. This process can be repeated if necessary. Following degumming, a strong base like sodium hydroxide is added for neutralization. The base is then separated from the oil using hot water, and the water layer is removed.After neutralization, the oil undergoes a bleaching process to remove any remaining color, phospholipids, and oxidation products. Deodorization is the final step, aimed at eliminating any odor from the oil.

Refined castor oil typically has a long shelf life of about 12 months, as long as it is not exposed to excessive heat. The process of refining crude castor oil is further discussed in the subsequent section. (Vinay R. Patel, 2016)

2.6.4 Crude Castor Oil Refining

While the previous section briefly discussed the general overview involved in a castor oil refining step, this section thoroughly explains each of the processes involved in it. Unrefined castor oil leads to rapid degradation due to the presence of impurities as mentioned in "Castor oil refining" section, making it less suitable for most applications. Hence, a refining. Process has to be conducted prior to the derivatization of the oil. The order of the steps performed in the refining process, which includes degumming, neutralization, bleaching, deodorization, and sometimes winterization, should be taken into consideration for efficient oil refining and are described extensively and specifically in a castor oil industry setting in "Degumming", "Neutralization", "Bleaching", "Deodorization". (Vinay R. Patel, 2016)

2.6.5 Degumming

The first step in the castor oil refining process, called degumming, is used to reduce the phosphatides and the metal content of the crude oil. The phosphatides present in crude castor oil can be found in the form of lecithin, cephalic, and phosphatides acids. These phosphatides can be classified into two different types: hydratable and non-hydratable, and accordingly, a suitable degumming procedure (water degumming, acid degumming, and enzymatic degumming) has to be performed for efficient removal of these phosphatides. In general, crude vegetable oil contains about 10% of non-hydratable phosphatides. However, the amount may vary significantly depending on various factors such as the type of seed, quality of seed, and conditions applied during the milling operation. While hydratable phosphatides can be removed in most part by water degumming, non-hydratable phosphatides can only be removed by means of acid or enzymatic degumming procedures. (Vinay R. Patel, 2016)

2.6.6 Neutralization

Good quality castor seeds stored under controlled conditions produce only low FFA content of approximately 0.3%. (Okullo AA, 2012) Occasionally, oil seeds that are old or stored for more than 12 months with high moisture content produce a high FFA content of about 5% level. This excess FFA present in the castor oil does not provide the same functionality as the neutral oil and has the ability to alter its reactivity with different substances. Hence, it is highly essential to remove the high FFA content so as to produce a high-quality castor oil. This process of removal of FFA from the degummed oil is referred to as neutralization. (Okullo AA, 2012)

In general, the refining process can be divided into two methods: chemical and physical refining. Physical refining is usually done by maintaining a high temperature above 200°C with a low vacuum pressure. Under these processing conditions, the low boiling point FFA is vacuum distilled from the high boiling point triglycerides. However, physical refining is not recommended in the case of castor oil, due to its sensitivity to heat as it normally starts disintegrating above 150°C, which can result in the hydrolysis of the hydroxyl groups. On the other hand, chemical refining is based on the solubility principle of triglycerides and soaps of fatty acids.FFAs (acid) react with alkali (strong base) to form soaps of fatty acids. The formed soap is generally insoluble in the oil and, hence, can be easily separated from the oil based on the difference in specific gravity between the soap and triglycerides. The specific gravity of soap is higher than that of triglycerides and therefore tends to settle at the bottom of the reactor. Most of the modern refineries use high-speed centrifuges to separate soap and oil mixture. Alkali neutralization or chemical refining reduces the content of the following components: FFAs, oxidation products of FFAs, residual proteins, phosphatides, carbohydrates, traces of metals, and a part of the pigments. The degummed castor oil is first treated with an alkali solution (2% caustic soda) between 85°C and 95°C with constant stirring for approximately 45–60 minutes. (Bhosle BM, 2005)

At this stage, the alkali reacts with FFAs and converts them into soap stock. The obtained soap has a higher specific gravity than the neutral oil and tends to settle at the bottom. The oil can be separated from the soap either by gravity separation or by using commercial centrifuges. Small-scale refiners use gravity separation route, whereas large capacity plants utilizes commercial vertical stack bowl centrifuges. The separated oil is then washed with hot water to remove soap, alkali solution, and other impurities. (Bhosle BM, 2005)

Typically, batch neutralization of castor oil requires about four to six hot water washes so as to bring down the soap level to below 100 ppm. (Okullo AA, 2012)

The oil, thus obtained, is vacuum dried and is transferred to the next process, bleaching. Castor oil neutralization is a high loss-refining step. This loss is presumably due to the small difference in specific gravity of the generated soap and neutral viscous castor oil. (Bhosle BM, 2005)

2.6.7. Bleaching

Castor oil is used for many applications where the final product's appearance is extremely important. For instance, cosmetics formulations, lubricant additives, and biomaterial manufacturing all demand the final product's color to be within a certain limit. Although castor oil obtained after degumming and neutralization processes yield a clear liquid by appearance, it may still contain colored bodies, natural pigments, and antioxidants (tocopherols and tocotrienols), which were extracted along with the crude oil from the castor beans. The color pigments are extremely small ranging from 10 to 50 nm, which cannot be removed from the oil by any unit operation. However, an adsorption process called "bleaching" can be used to remove such colored pigments and remaining phospholipids, using activated earths under moderate vacuum conditions between 50 and 100 mmHg. The reduction in the oil color can be measured using an analytical instrument, called a tintometer. (Okullo AA, 2012)

Bleaching of castor oil can be done under vacuum at around 100°C while constantly stirring the oil with an appropriate amount of activated earths and carbon. The bleaching process requires around 2% bleaching earth and carbon to produce desirable light colored oil. Under these processing conditions, colored bodies, soap, and phosphatides adsorb onto the activated earth and carbon. The activated earth and carbon are removed by using a commercial filter. The spent earth-carbon, thus obtained, retains around 20%–25% oil content. Bleaching castor oil containing higher phosphatide and soap content often leads to high retention of oil due to the large amount of activated earth used and thus causes filtration issues. Although this retained oil on the spent earth can be recovered by boiling the spent earth in water or by a solvent extraction method, the recovered oil from the spent earth is highly colored with high FFA and high peroxide content, normally greater than 10 mg KOH/g and 20 meq/kg, respectively. (Kheang LS, 2006)

2.7.8 Deodorization

Deodorization is simply a vacuum steam distillation process that removes the relatively volatile components that give rise to undesirable flavours, colors, and odors in fats and oils. Unlike other vegetable oils, castor oil requires limited or no deodorization, as it is a nonedible oil where slight pungent odor is not an issue for most of its applications, with the only exception being pharmaceutical grade castor oil. Deodorization is usually done under high vacuum and at high temperature above 250°C to remove undesirable odors caused by ketones, aldehydes, sterols, triterpene alcohols, and short-chain fatty acids.

Pharmaceutical grade castor oil is deodorized at low temperatures, approximately 150°C–170°C under high vacuum for 8–10 hours to avoid hydrolysis of hydroxyl group of RA. (FC, 2011)

2.8. Characterization of castor oil

2.8.1. Physical analysis

- **PH value**

The pH value of oil was determined with the aid of a pH meter (Model Delta 320, Mettler Toledo, China).

- **Viscosity**

Castor oil is a hydroxy monounsaturated triglyceride that has a high viscosity and a viscosity index of 9012. The viscosity of castor oil at 10 °C is 2,420 centipoise, which is much higher than most other oils. However, castor oil also tends to form gums in a short time, which limits its use to engines that are regularly rebuilt, such as racing engines. The viscometer was placed into a holder and inserted to a constant temperature bath set at 29°C and allowed approximately 10 minutes for the sample to come to the bath temperature at 29°C.

- **Specific gravity**

Specific gravity (also referred to as relative density) is the ratio of the density of a material compared to the density of water at 4 °C4 °C (39.2 °F39.2 °F). People usually choose that temperature as it is when water is at its densest. The specific gravity equation is:

$SG = \rho_{object} / \rho_{reference}$………………..Equation 1

Where:

- SG is the specific gravity (dimensionless)
- ρ_{object} is the density of the object you're measuring (in kg/m^3 or g/cm^3)
- $\rho_{reference}$ is the density of the reference material (usually water at 4°C, which is approximately 1000 kg/m^3 or 1 g/cm^3)

Since the specific gravity formula consists of one density divided by another, the specific gravity units don't exist. It is a unit less quantity, like most ratios in physics.Specific gravity values are usually given at one atmosphere of pressure (1013.25 kPa). Remember, that you have to check the temperature of both the material and the reference (usually water) when quoting specific gravities. Standard temperatures vary depending on industry and the material you are measuring. The specific gravity of water has a value of 1, as its being compared to itself, and the same number divided by it is 1. (Dominik Czernia, 2023)

- **Refractive index**

The index of refraction, also called the refractive index, describes how light propagates through a medium. It is a dimensionless quantity, and it determines how much light is bent (or refracted) when entering a different medium. In essence, refraction means a change in the speed and wavelength of the wave. Refractometer was used in this determination. (Dominik Czernia, 2023)

2.8.2 Chemical Analysis

- **Saponification value**

Saponification value (SV) or saponification number (SN) represents the number of milligrams of potassium hydroxide (KOH) required to saponify one gram of fat under a specific condition. The saponification value is a significant parameter used to characterize and evaluate the quality of edible fats and oils. Also, the saponification number provides information about the fatty acids' average molecular weight. The higher the number, the lower the molecular weight of all fatty acids.

Saponification value was calculated from the equation

$SV = \sum (WI * Mwi * 56.1) / 1000$…………………………. Equation 2

Where:

Wi = Weight percentage of each fatty acid (i) in the sample

Mwi = Mean molecular weight of each fatty acid (i) (g/mol)

56.1 = Equivalent weight of KOH (mg/eq)

1000 = Conversion factor (mg to g)

(Warra, 2011)

- **Iodine value**

Iodine value, also called iodine number, in analytical chemistry, measure of the degree of unsaturation of an oil, fat, or wax; the amount of iodine, in grams, that is taken up by 100 grams of the oil, fat, or wax. Saturated oils, fats, and waxes take up no iodine, and therefore their iodine value is zero, but unsaturated oils, fats, and waxes take up iodine. (Unsaturated compounds contain molecules with double or triple bonds, which are very reactive toward iodine.) The more iodine is attached, the higher is the iodine value, and the more reactive, less stable, softer, and more susceptible to oxidation and rancidification is the oil, fat, or wax. In performing the test, a known excess of iodine, often in the form of iodine monochloride, is allowed to react with a known weight of the oil, fat, or wax, and then the amount of iodine remaining unreacted is determined by titration. (Erik-Gregersen, 2022)

The iodine value (I.V) is given by the expression $I.V. = 12.69C (V1-V2)/M$ ……Equation 3

Where C =concentration of sodium

V1 = Volume of sodium thiosulphate used for blank

V2 = Volume of sodium thiosulphate used for determination

M = Mass of the sample.

- **Acid value**

The acid value (AV), also known as acid number, neutralization number, or acidity, is a numerical measurement used to determine the level of acidity in a specific chemical substance. It represents the amount of base, typically potassium hydroxide (KOH), required to neutralize the acidic components in 1 gram of the substance. The acid value can be calculated using the formula: (Warra, 2011)

AV = (V * N * 56.1) / m………………………………Equation 4

Where:

V = Volume of standard KOH solution used in the titration (mL)

N = Normality of the KOH solution (eq/L)

56.1 = Equivalent weight of KOH (mg/eq)

m = Mass of the sample (g)

- **Volatile matter**

A sterile and flat petri dish was dried in an oven, allowed to cool in a desiccator, and then weighed to obtain the initial weight. A precise amount of 2 g of the oil was carefully measured using a dropping pipette and added to the petri dish. The dish, along with its contents, was then weighed and the weight recorded. The dish was placed in an air oven set at a temperature of 105°C and left to dry for approximately 3 hours. After drying, the dish was transferred to a desiccator to cool down and then weighed again. This drying process was repeated until a constant weight was achieved, and the final constant weight was recorded. The percentage of volatile matter was calculated based on these measurements. As:

% VM = (W2 – W3 / W2 – W1)*100…………………Equation 4

Where W1 = Weight of empty dish

W2 = Weight of dish with contents

W3 = Final weight of dish with contents

2.9. Soap-making processes

Soap production on an industrial scale typically involves continuous processes, where fat is continuously added and the soap product is continuously removed. On the other hand, smaller-scale production follows traditional batch processes. There have been different approaches to soap production, including the decomposition of fat or oil into fatty acids and glycerine, followed by the conversion of these acids into soap through treatment with sodium or potassium carbonate. Three common methods of soap making are typically employed: semi-boiling, full boiling, and cold processed.

2.9.1. Semi – boiling

The process of soap making is well-suited for both soft and hard oils, as well as their blends. In this process, the fat is initially melted and then treated with a mild caustic soda solution, typically with a concentration of 9-10%. The quantity of caustic soda required for saponifying the oil is around 14-15% of the oil's weight. To prepare the caustic soda solution, the weight of caustic soda is dissolved in ten times its weight of water, resulting in a 9% solution. The saponification process begins when the caustic solution is added to the oil, resulting in the formation of an emulsion as the mixture is stirred. Additional caustic solution is gradually added to prevent the mixture from becoming too thick. This step-by-step addition ensures complete saponification. The boiling of the mixture continues until the soap becomes clear. Throughout the boiling process, moderate heat is maintained, and each addition of caustic soda solution must be given sufficient time to react with the oil before the next addition is made. This careful and gradual approach ensures successful saponification.

Rapid addition of caustic soda solution during the early stages of the soap making process can hinder saponification, while adding it too quickly towards the end of saponification can lead to the soap drying out. On the other hand, careful and calculated addition of the solution helps maintain a smooth and uniform emulsion throughout the process. If any signs of separation or graininess appear in the soap, additional water can be added to restore the mass to a homogeneous state. While this process is not suitable for the production of toilet soap, it can be effectively used for manufacturing laundry soap and other types of soft and liquid soaps. One limitation of this process is that it does not allow for the removal of waste alkali, which contains glycerine produced during soap making. As a result, the glycerine, which can reduce the hardness of the soap and enhance its cosmetic properties, remains in the final product. However, this method offers advantages over the other two methods as it allows for the production of large quantities of high-quality soap in a relatively short period of time. Additionally, this process enables the incorporation of a higher percentage of fillers into the soap, resulting in increased bulk.

2.9.2. Full Boiling

The process can be divided into four stages: saponification of the oil using alkali, separation of the soap through graining, strengthening the soap through boiling, and finally, filling the soap into appropriate containers.

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.

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.

2.9.3. 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 glycerol 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 generates little heat to complete the saponification reaction. In this process, it is absolutely necessary 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 discolour. 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 the temperature 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 be stirred 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 solution is used to blend the caustic soda solution which greatly improves the appearance of the given soap, making it smoother and milder.

2.9.4. Types of Soaps

There are several types of soap available, each with its own unique formulation and purpose. Here are some common types of soap:

Bar Soap: Bar soap is a solid soap that comes in various shapes, sizes, and formulations. It is typically made by combining fats or oils with an alkali, such as sodium hydroxide (in the case of solid soap) or potassium hydroxide (in the case of liquid soap).

Liquid Soap: Liquid soap is a soap formulation that is in a liquid or gel form. It is often used for hand washing, body washes, or as a base for creating other cleaning products.

Castile Soap: Castile soap is a type of soap made from vegetable oils, traditionally olive oil. It is known for its mildness and gentle cleansing properties.

Glycerine Soap: Glycerine soap is made with glycerine, a natural by-product of the soap-making process. It is known for its moisturizing properties and is suitable for those with sensitive or dry skin.

Antibacterial Soap: Antibacterial soap contains additional ingredients, such as triclosan or benzalkonium chloride, which are intended to kill or inhibit the growth of bacteria. However, regular hand washing with plain soap and water is generally sufficient for maintaining good hygiene.

Exfoliating Soap: Exfoliating soaps contain abrasive ingredients, such as ground coffee, oatmeal, or pumice, which help to remove dead skin cells and promote smoother skin.

Scented Soap: Scented soaps are infused with fragrances or essential oils to provide a pleasant scent during use. Various scents, such as lavender, citrus, or floral notes, are used to enhance the bathing experience. Uses of scented soap:

- Cleansing: Scented soap can effectively remove dirt, oil, and sweat from the skin.
- Moisturizing: Some scented soaps contain ingredients that can help to hydrate and moisturize the skin.
- Aromatherapy: Scented soap can be used for aromatherapy, which is a holistic healing treatment that uses the therapeutic properties of essential oils.
- Mood-boosting: Some scents, such as lavender and citrus, have been shown to have mood-boosting effects.
- Personal care: Scented soap is often used for personal care, such as handwashing, showering, and bathing.
- Gift-giving: Scented soap makes a great gift for friends, family, and loved ones.

Benefits of using scented soap:

- Pleasing scent: Scented soap can add a touch of luxury and enjoyment to your daily routine.

- Mood-boosting: Some scents can help to improve your mood and reduce stress.
- Aromatherapy benefits: Scented soap can provide aromatherapy benefits, such as relaxation, stress relief, and improved sleep.

Considerations when choosing scented soap:

- Fragrance type: Choose a fragrance that you enjoy and that is appropriate for your skin type.
- Scent strength: If you have sensitive skin, choose soap with a mild scent.
- Ingredients: Check the ingredients list to make sure that the soap does not contain any irritants or allergens.
- Brand reputation: Choose soap from a reputable brand that uses high-quality ingredients.

Specialty Soaps: There are also specialty soaps available for specific purposes, such as acne-fighting soaps, moisturizing soaps, baby soaps, and shaving soaps, each formulated to address specific needs.

These are just a few examples of the many types of soap available on the market. The choice of soap depends on personal preference, skin type, and specific requirements.

2.9.5 History of making natural body soap in Ethiopia

The origins of natural body soap can be traced back to ancient times, where civilizations across the globe utilized natural ingredients for body cleansing. In Ethiopia, the cultural tradition of body cleansing has been passed down through generations. A renowned natural body soap in Ethiopia is known as "Ambassel," which is crafted from a blend of herbs, roots, and spices like neem, eucalyptus, cinnamon, and ginger. These ingredients are carefully selected for their cleansing and moisturizing properties. The process of making traditional Ethiopian soap involves a meticulous approach. The herbs and spices are finely ground into a powder and mixed with water to form a paste. This paste is then shaped into bars or balls and left to naturally dry and harden. Once prepared, the soap is used for body cleansing, leaving behind a refreshing and fragrant aroma. The use of natural body soap holds deep cultural significance in Ethiopia, cherished for its gentle and nourishing attributes. It not only cleanses the skin but also helps maintain its natural moisture balance. Many individuals in Ethiopia continue to prefer these traditional natural body soaps due to their connection to local heritage and the belief in their positive effects on the skin.

2.9.6 Rationale for making natural based body soap

In the present day, there is a global surge in the demand for environmentally friendly and natural products, leading to an increased popularity of natural body soaps. These soaps are highly valued for their simplicity and purity, as they are devoid of harsh chemicals and synthetic additives. Consequently, traditional Ethiopian natural body soaps are gaining recognition and appreciation beyond the borders of Ethiopia. Their appeal lies in their commitment to providing a wholesome and natural cleansing experience.

2.9.7 Factors affecting soap making process

- Ingredient ratios
- Temperature
- Mixing and emulsification
- Additives and fragrance
- Curing and drying
- Water quality

- **Ingredient Ratios**

The precise measurement and ratio of fats (oils or butters) to alkali (such as sodium hydroxide or potassium hydroxide) are crucial. Deviating from the recommended ratios can result in either a lye-heavy or super fatted soap, affecting its quality and properties.

- **Temperature**

Both the temperature of the ingredients and the environment play a role. Maintaining the correct temperature during the saponification process ensures proper chemical reactions and influences the soap's texture, hardness, and curing time.

- **Mixing and Emulsification**

Thoroughly mixing the fats and alkali is essential for proper saponification. Insufficient mixing can lead to uneven distribution of ingredients and affect the soap's consistency.

- **Additives and Fragrances**

Incorporating additives like colorants exfoliates, or fragrances can impact the soap's appearance, scent, and texture. It's important to consider the compatibility of these additives with the soap base and their safe usage levels.

- **Curing and Drying**

Allowing the soap to cure and dry properly after molding is crucial for its final quality. Factors like humidity, temperature, and air circulation can affect the curing process and the soap's hardness, longevity, and lather.

- **Water Quality**

The quality of water used in soap making can influence the outcome. Hard water with high mineral content may affect lathering and produce soap scum, while soft water might require adjustments to the recipe.

2.9.8. Equipment and Techniques

The type of equipment used, such as molds and mixing tools, can impact the soap's shape and texture. Additionally, different soap-making techniques, like cold process or hot process, can yield variations in the final product.

2.9.9 Characterization of frankincense scented castor oil body soap

2.9.8.1. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis can provide information about the soap's chemical composition and identify functional groups present in the soap. It helps in confirming the presence of specific ingredients, such as castor oil and the components responsible for the frankincense scent.

2.9.8.2. Physical analysis

- **PH value**

The pH value is a measure of the acidity or alkalinity of a substance. In the case of soap, its pH value indicates how basic or alkaline it is. Soaps typically have a pH value between 8 and 10, which makes them slightly alkaline. This alkaline nature helps soap to effectively clean by breaking down oils and removing dirt from surfaces. It's important to note that maintaining the proper pH balance in soap is crucial to ensure its mildness and skin-friendliness. (Okullo AA, 2012)

- **Moisture content**

Moisture content refers to the amount of water or moisture present in the soap formulation. The moisture content in soap is a critical factor that affects its quality, stability, and performance. Soap manufacturers carefully control the moisture content during the soap-making process. Excess moisture can lead to issues such as softness, reduced lather, and decreased shelf life. On the other hand, insufficient moisture can result in a dry and brittle soap that does not lather well. To ensure the desired moisture content, soap makers typically use a combination of precise measurements and drying techniques. They may use specialized equipment or allow the soap to cure and dry naturally over a specific period. The goal is to achieve an optimal moisture content that results in a balanced and long-lasting soap bar. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015)

2.9.8.3 Chemical analysis

- **Alcohol insoluble**

Alcohol insoluble in the context of soap refers to the portion of soap that does not dissolve or mix with alcohol. It is a measure of the soap's ability to remain insoluble in alcohol, which is often used as a solvent for testing soap properties. When soap is mixed with alcohol, it typically forms a clear solution due to its solubility. However, certain components or impurities present in the soap may remain insoluble and separate from the alcohol solution. These insoluble substances can include fatty acids, unsaponifiable matter, or other impurities. (Abdalla Ayoub Abdalla, 2020)

The alcohol insoluble content of soap is an important parameter that can affect its quality and performance. Soap with a high alcohol insoluble content may indicate the presence of impurities or excess fatty acids, which can impact its cleansing ability, lather formation, or stability. To determine the alcohol insoluble content, a sample of soap is dissolved in alcohol, and the insoluble residue is collected, dried, and weighed. The weight of the insoluble residue is then expressed as a percentage of the initial soap sample, providing an indication of its alcohol insoluble content. (Bhuiya M, 2016)

- **Saponification value**

SAP value stands for "Saponification Value." It is a measure of the amount of alkali needed to completely saponify a specific amount of fat or oil. Saponification is the chemical reaction that occurs when fats or oils are mixed with a strong alkali, such as sodium hydroxide or potassium hydroxide, to produce soap. (Okullo AA, 2012)

The SAP value is usually expressed in milligrams of potassium hydroxide (KOH) or sodium hydroxide (NaOH) required saponifying one gram of fat or oil. It is an important parameter in soap making as it helps determine the appropriate amount of lye needed to convert fats or oils into soap. Different fats and oils have different SAP values due to their varying chemical compositions. Soap makers use SAP values as a guide to formulate soap recipes and ensure the right balance of oils and alkali for the desired properties of the final soap product. (Mohammadreza Koushki, 2008)

- **Free caustic (alkalinity test)**

Alkalinity test for soap refers to a procedure used to measure the alkaline content in soap products. This test determines the amount of alkali, such as sodium hydroxide, remaining in the soap after the saponification process. It helps assess the effectiveness of soap in cleansing and its potential to irritate the skin. The test is typically conducted by dissolving a sample of the soap in water, adding an indicator solution, and then titrating it with an acid solution until the color of the indicator changes. The amount of acid required to neutralize the alkaline content indicates the soap's alkalinity. (Okullo AA, 2012)

- **Soap stability**

Soap stability refers to the ability of a soap product to maintain its desired properties and performance over time. It involves assessing the soap's resistance to physical, chemical, and microbiological changes that can occur during storage or use. (Okullo AA, 2012)

- **Total fatty matter**

The total fatty matter (TFM) in soap refers to the percentage of fats and oils present in the soap formulation. TFM is an important characteristic as it determines the quality and effectiveness of the soap. In Ethiopia, traditional soap-making techniques, such as "Ambasha" or "Dabo" soap, often involve using locally available oils like sesame, palm, or coconut oil. The TFM content in Ethiopian soaps may vary depending on the specific recipe and production methods employed by local soap manufacturers. (Vinay R. Patel, 2016)

- **Foam height**

Foam height refers to the amount of lather or bubbles that are produced when soap is agitated or mixed with water. When soap is applied and rubbed against the skin or other surfaces, it creates a layer of foam that helps to lift dirt, oils, and impurities. The foam height can vary depending on various factors such as the type of soap, water temperature, and the amount of agitation applied. Generally, a good soap will produce rich and creamy foam that effectively cleanses and nourishes the skin. In Ethiopia, like anywhere else, the foam height of soap is an important characteristic that people often consider when choosing soap for their personal hygiene needs. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015; Abdalla Ayoub Abdalla, 2020)

- **Foam stability**

Foam stability for soap refers to the ability of soap to maintain a stable foam structure over time. When soap is agitated or mixed with water, it forms bubbles that create foam. The stability of this foam is important because it determines how long the bubbles will last before collapsing or breaking down. Soap molecules have both hydrophilic (water-attracting) and hydrophobic (water-repelling) properties. These properties allow soap to reduce the surface tension of water and form stable bubbles. The hydrophobic part of the soap molecule attaches to the air inside the bubble, while the hydrophilic part remains in contact with the water. (Baskar, 2022)

Factors that affect foam stability include the concentration of soap, water hardness, temperature, impurities in the water, and the presence of additives. Higher soap concentrations generally lead to more stable foam, while hard water and impurities can reduce foam stability. Temperature can also impact foam stability, with higher temperatures often leading to faster bubble breakdown. To enhance foam stability, some soap formulations may include additives such as glycerine or certain surfactants. These additives can help to increase the longevity of the foam and improve its overall stability. (Warra, 2011)

In conclusion, foam stability for soap is the ability of soap to maintain a lasting and well-formed foam structure. It is influenced by various factors, and soap formulations may include additives to enhance foam stability. (Vinay R. Patel, 2016)

CHAPTER THREE

3. Materials and Methods

3.1 Materials

Ethanol (99.9%, Fisher Scientific, UK), HCl (37%99%, Sigma-Aldrich, France), sodium hydro oxide (99%, Sigma-Aldrich, France), Methyl orange and phenolphthalein indicator (98%), sodium silicate (99%, Sigma-Aldrich, France), sodium chloride, sulphuric acid (98%, Ltd pool England), perfume, colorant, Hydrogen per oxide (99%, Sigma-Aldrich, France) were used. In all experiments distilled water was used for dilution of samples. Castor seed has been used as raw material for the production of soap.

3.1.2 Equipment

In this experiments Screw press, boiling pan, 3000 ml round- bottom flask, beaker, stirrer rod filter paper, measuring cylinder, drying oven (Beschickung, loading model 100-800, Heating mantle , Separating funnel, analytical balance (SARTORIUS AG, BP110, Germany), water bath (SBS40, UK), pH meter 3510, JENWAY, UK) ,Thermometer , viscometer were used in this study.

3.2 Experimental methods

3.2.1 Extraction of castor oil

The mechanical extraction method using a screw press was employed at the food engineering laboratory of Addis Ababa Science and Technology University (AASTU) to extract oil from castor seeds. The process involved cleaning the screw press machine before continuously supplying the castor seeds to the top of the machine. As the seeds moved through the machine, the mechanical pressure and friction caused the oil to separate from the solid material. The extracted oil was continuously withdrawn from the oil-out part of the machine, while the solid residue, known as the cake, was continuously removed in the cake-forming section. This method allowed for efficient and continuous oil extraction from the castor seeds, with the potential for further processing and utilization in various industries. The amount of oil extracted was calculated using the equation below:

%yield of oil = $mass\ of\ oil\ obtained / mass\ of\ seed\ used \times 100\%$..................... Equation 5

3.2.2. Purification of crude castor oil

Filtration

To remove impurities from the oil obtained through screw pressing, vacuum filtration was employed. The impurities in the oil, including seed fragments and meal fines that are insoluble in oil, can be effectively separated through this filtration process. Vacuum filtration involves passing the oil through a filter under reduced pressure, which enhances the filtration efficiency. The filter captures the impurities, allowing the purified oil to pass through. This method ensures the removal of contaminants, resulting in a cleaner and higher-quality oil suitable for various applications. (Vinay R. Patel, 2016)

Degumming

After the vacuum filtration process, the purified oil was subjected to degumming to remove phospholipids. To initiate the degumming process, the oil was heated to 80 °C. Boiled water was then added to the oil, and the mixture was stirred for 2 minutes to ensure thorough mixing. The mixture was then allowed to settle in a separating funnel, where a clear separation occurred between the oil and the aqueous layer containing the degummed impurities. The aqueous layer, containing the majority of the phospholipids, was carefully extracted from the separating funnel. To ensure optimal removal of phospholipids, the degumming process was repeated, possibly with fresh water, to further enhance the purification of the crude castor oil. This step helps to obtain a refined oil with reduced phospholipid content, improving its quality and stability for various applications. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015)

3.2.3. Characterization of purified castor oil

3.2.3.1 Physical analysis

Determination of specific gravity

To determine the specific gravity of castor oil, 25ml pycnometers was properly washed with soap and water before being dried and weighed and record its mass the bottle or cylinder then filled with a known volume of castor oil (25ml used). Ensured that there was no air bubbles trapped within the oil. Weigh the bottle or cylinder with the oil and record its mass.Filled another identical bottle or cylinder with the same volume of water and weigh it. (Baskar, 2022)

Calculate the specific gravity using the following formula: The equation for specific gravity (SG) is quite simple:

$SG = \rho_{object} / \rho_{reference}$……………..Equation 6

Where:

SG is the specific gravity (dimensionless)

ρ_{object} is the density of the object you're measuring (in kg/m^3 or g/cm^3)

$\rho_{reference}$ is the density of the reference material (usually water at 4°C, which is approximately 1000 kg/m^3 or 1 g/cm^3)

The specific gravity of castor oil typically ranges from 0.950 to 0.965, meaning it is slightly less dense than water. This value can vary depending on factors such as temperature and impurities present in the oil.

Determination of viscosity

The viscosity of the oil was determined using a viscometer. The viscometer was charged with the castor oil by inverting the tube's thinner arm into the liquid sample and drawing suction force up to the upper timing mark of the viscometer, after which the instrument was turned to its regular vertical position and set to 40°c temperature. The value was then recorded from the viscometer display. (Baskar, 2022)

Determination of PH value

To determine the pH value of castor oil, pH meter was used. Ensured pH meter were cleaned and calibrated according to the manufacturer's instructions. A clean, dry 250ml beaker was filled with 100g of the sample. It was then chilled to 25°C in a cold-water bath. Carefully immersed the electrode of the meter into the castor oil sample. (Baskar, 2022)

Determination of Colour of castor oil

To determine the colour of castor oil Visual inspection used. (Baskar, 2022)

3.2.3.2 Chemical analysis

Determination Saponification value of castor oil

To determine the saponification value of castor oil, 2g of oil was weighed then transferred the castor oil to a clean and dry flask. Then added 25ml0.1NKOH a standardized potassium hydroxide (KOH) solution to the flask containing the castor oil. Heated the flask gently on a hot plate or in a water bath, while stirring, until saponification was complete. The solution stirred and boiled for 60 minutes. Once saponification was complete four drops of phenolphthalein indicator solution to the flask was added then Titrated the excess KOH solution in the flask with a standardized (0.5N) HCL solution. (Baskar, 2022)

Calculate the saponification value using the following formula:

SV = (B - S) * N * 56.1 / m……………………….....Equation 7

Where:

B = Volume of standard acid used in blank titration (mL)

S = Volume of standard acid used in sample titration (mL)

N = Normality of the standard acid solution (eq/L)

56.1 = Equivalent weight of KOH (mg/eq) (adjust to 40 for NaOH)

m = Mass of the sample (g)

Determination of acid value

The acid value of the castor seed oil was evaluated using the method provided by in a 250ml beaker, 25ml of diethyl ether and 25ml of ethanol were combined. The resulting mixture was put to 5g of oil in a 250ml conical flask, along with three drops of phenolphthalein. The mixture was titrated with 0.1M NaOH to the end point with steady shaking, resulting in a pink color and a volume of 0.1M NaOH (V0). (Abubakar, 2016)

AV = (V * N * 56.1) / m…………….…Equation 9

Where:

V = Volume of standard KOH solution used in the titration (mL)

N = Normality of the KOH solution (eq/L)

56.1 = Equivalent weight of KOH (mg/eq)

m = Mass of the sample (g)

Determination of iodine value

The amount of unsaturation in fats and oils is frequently calculated using iodine values. The (Abubakar, 2016) approach was employed for this investigation. 20ml of carbon tetra chloride was added to 0.4g of the sample in a conical flask to dissolve the oil. A safety pipette was then used in the fume chamber to add 25ml of Dam's reagent to the flask. The flask's contents were then forcefully swirled once the stopper was placed. After that, the flask was left in the dark for two hours. Using a measuring cylinder, 125ml of water and 20ml of 10% aqueous potassium iodide were added at end of this time. With 0.1M sodium-thiosulphate solutions, the content was titrated until the yellow color nearly vanished. The titration was then continued by adding thiosulphate drop by drop until the blue coloring vanished after vigorous shaking. A few drops of 1% starch indicator were added first. For the blank test and other samples, the same process was applied.

Iodine value (I. V) =$12.6(v1-v2) \times Nm$………Equation 8

Where C = Concentration of sodium thiosulphate used; V1 = Volume of sodium thiosulphate used for blank; V2 = Volume of sodium thiosulphate used for determination, m = Mass of the sample.

3.3. Soap making process

The saponification process is the chemical reaction that occurs when oils or fats (known as triglycerides) are mixed with a strong alkaline solution, such as sodium hydroxide (NaOH) for solid soap or potassium hydroxide (KOH) for liquid soap. This reaction carried out using castor oil as base oil and sodium hydroxide as alkaline solution. Before the saponification process occurred infusion of castor oil with frankincense was done. As the saponification reaction progresses, the mixture thickened and reached a stage known as "trace." This was when the soap batter has thickened to a consistency similar to pudding or custard. At this point, the soap was ready to be poured into molds. After pouring the soap into molds, it undergoes a curing period, typically 24hrs during which it hardened and completed the saponification process. This allowed any remaining lye to fully react with the oils/fats and ensured a mild and stable soap.

3.3.1 Preliminary experiment

These experiments were done to identify which factors had effect on moisturizing, hardness and stable lather of body soap.

Table 3.1experimental design

Run	Castor oil (in volume)	Water to lye ratio	Temperature (^{0}C)	Mixing time (min)	Moisturizing (visual assesements)
1	Actual(5ml)	0.6g/1.5ml	90	15	5
2	Actual(6ml)	0.7g/1.7ml	90	15	10
3	Actual(7ml)	0.8g/2ml	90	15	7
4	Actual(8ml)	0.9g/2.3ml	90	15	7
5	Actual(9ml)	1.0g/2.6ml	90	15	8.5
6	Actual(10ml)	1.2g/2.9ml	90	15	10

3.3.2. Response surface methodology Experimental design

Design Expert software 6.0.8 with box Behnken design (BBD) was used to design the trials and study the impacts of process factors on frankincense scented castor oil soap through saponification reaction. The water to lye ratio, castor oil concentration, and temperature were chosen as process variables. Design Expert software 6.0.8 with Box-Behnken design offers significant advantages in terms of efficient experimental design, reduced experimental runs, optimal factor settings, response optimization, and visualization. It is a valuable tool for researchers, engineers, and scientists involved in process optimization, product development, and quality improvement.

Table 3-1. Factors and levels

FACTORS	LEVELS	
	HIGH	LOW
TEMPERATURE(ºc)	90	85
WATER TO LYE RATIO	2	1.5
CONCENTRATION OF CASTOR OIL(ml)	10	5

Table 3-2 BBD experimental design

Std	Run	Block 1	Temperature (ºc)	water to lye ratio	Concentration of castor (ml)	Hardness	Stable lather	Fluffy lather	moisturizing
1	11	Block 1	85	1.5	7.5	4.2	9.5	1.1	8.5
2	3	Block 1	90	1.5	7.5	5	10	2	10
3	7	Block 1	85	2	7.5	5.1	2	4.7	8.2
4	4	Block 1	90	2	7.5	5.5	5.4	5	7
5	2	Block 1	85	1.75	5	7	8	2.5	6
6	12	Block	90	1.75	5	8	8.5	2	5

		1							
7	16	Block 1	85	1.75	10	3	6.2	1.2	8
8	10	Block 1	90	1.75	10	4.8	8.5	1.3	10
9	8	Block 1	87.5	1.5	5	3	10	2	8.5
10	9	Block 1	87.5	2	5	1.2	7	5	4.2
11	14	Block 1	87.5	1.5	10	5.2	8.9	9	10
12	6	Block 1	87.5	2	10	1.1	4	3	7
13	15	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
14	5	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
15	1	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
16	17	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
17	13	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10

3.3.3. Synthesis of frankincense scented castor oil soap

Table 3-3 Synthesis of frankincense scented castor oil soap

sample	Oil base(castor oil) ml	NAOH Needed(kg)	Water Needed(kg)	Lye solution strength (%) (Using lye solution strength calculator)	Additives(g) (frankincense)
1	7	0.807	1.498	35	3.35
2	8	0.922	1.713	35	3.83
3	9	1.037	1.927	35	4.31
4	10	1.153	2.141	35	4.79

Note: To calculate lye solution strength

Oil weight: Weigh the total amount of oils you'll be using in grams.

Lye type: Identify the type of lye you'll be using (e.g., sodium hydroxide (NaOH) or potassium hydroxide (KOH)).

Super fat percentage: Choose your desired super fat percentage. This refers to the excess oil remaining in the soap after saponification, contributing to moisturizing properties but decreasing cleansing power. Common ranges are 5-10% for beginners and 0-5% for experienced soap makers. Lye Concentration (as a percentage of water weight)This method expresses the lye concentration as a percentage of the total water weight used in the recipe. However, it's important to note that the actual lye concentration in the final soap solution will vary depending on the saponification values of your chosen oils.

Formula: Lye concentration (%) = (Lye weight / Water weight) * 100

Calculate the required lye weight using a soap calculator or the saponification value of your oils. Choose a desired lye concentration (common range: 25-35%).Based on the concentration and lye weight, calculate the necessary water weight using the formula above.

3.3.4. Characterization of frankincense scented castor oil body soap

Determination of pH value

To determine the pH of soap, dissolve 1g of soap in 99ml distilled water. Use a calibrated pH meter or indicator strips to dip the electrode into the soap solution, note the pH reading, repeat the process two more times, and calculate the average pH readings for a more reliable value. The pH indicates acidity, neutrality, or alkalinity. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015)

Determination of moisture content

To determine the moisture content in soap, weighed an empty container, place a 2g sample, place it in an oven at 100-105°C for 1-2 hours, and let it cooled. After dried, weigh the dried soap sample and calculate the moisture content using the formula. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015)

MC (%) = ((W1 - W2) / W1) * 100………………….Equation 9

Where:

W1 is the initial weight of the sample (g)

W2 is the weight of the sample after drying (g)

Determination of Alcohol insoluble matter in frankincense scented castor oil body soap

To determine the alcohol insoluble matter of castor soap, weighed 5g of soap, chop it into small pieces, and dissolved it in a solvent like ethanol (97%). Stir the mixture gently, filter the solution, and collect the alcohol-soluble components. After washed the filter residue with same solvent allowed the solvent to evaporate, and place the alcohol-insoluble matter in an oven set at low temperature. After cooling, weighed the alcohol-insoluble matter and calculate the percentage of alcohol-insoluble matter using the formula.

Alcohol Insoluble Matter (%) = (Weight of alcohol-insoluble matter / Weight of soap) x 100………Equation 10

Determination of saponification test

To determine saponification value 2g of oil sample was weighed into a conical flask; 25ml 0.1N KOH was then added. The content was constantly stirred and allowed to boil gently for 60minutes. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5N HCL to the end point until the pink colour of the indicator just disappeared. (Bhavani1, 2023)

Saponification Value = MW x N (V0-V1)/M…………………..Equation 11

Where V0 = the volume of the solution used for the blank test (L).

V1 = the volume of the solution used for determination (L).

M = mass of the sample (g).

N = Actual normality of KOH (mol/L)

MW = Molecular Weight of KOH (g).

Determination of free acid content

6g of soap sample was dissolved in 70ml hot neutral alcohol and titrated against 2M H_2SO_4 using phenolphthalein indicator. (Bhavani1, 2023)

The free alkali/acidity was calculated as;

Free acid content=3.1MV/W………………………Equation 12

Where: M – Molarity of H_2SO_4 solution, $mol \cdot L^{-1}$

V – Volume of H_2SO_4 solution used in titration, ml

W – Weight of the soap sample

Determination of foam height of frankincense scented castor oil body soap

2g of soap was dissolved in a liter volumetric flask and made to mark with tap water, 50ml of the solution was introduce into a measuring cylinder such that it followed the walls of the column to avoid foaming. 200ml of the solution was taken in a conical flask and poured into a funnel, which was already clamped with the outlet closed. The measuring cylinder was then put directly beneath the funnel while the level (height) of the foam generated was read from the cylinder immediately the funnel outlet was opened. (A.1, Characterization and Utilization of castor bean seed oil extract , 2015)

Determination of total fatty matter of frankincense scented castor oil body soap

To determine total fatty matter (TFM) in soap, weighed 5g of sample, chopped it into small pieces, then added ethanol or isopropanol, heat, filter, transfer to an evaporating dish, evaporate, and weigh the remaining fatty matter. (Warra, 2011)

Calculate the percentage of total fatty matter using the formula

TFM (%) = (Weight of fatty matter / Weight of soap) x 100……………………..Equation 13

FTIR analysis

FTIR, or Fourier transform infrared spectroscopy, is a widely used technique in infrared spectroscopy that involves the absorption and measurement of infrared radiation passing through a sample. By analyzing the emitted radiation, FTIR spectroscopy can identify the chemical functional groups present in a surfactant. This is achieved by comparing the observed spectrum with reference spectra of known compounds, enabling the characterization of the surfactant's molecular composition and aiding in its identification and analysis. Berthomieu and Hienerwadel's 2009 study likely provides specific examples and applications of FTIR spectroscopy in surfactant analysis. (Berthomieu and Hienerwadel 2009).

CHAPTER FOUR

4. Results and discussion

4.1 Extraction and characterization of castor oil

4.1.1 Extraction of castor oil

The oil extraction process was performed by using a screw press machine for 2 hours to extract 3.2 liters of oil from 10 kg of dried castor seed. 33.4% yield was obtained, which is in the range of 30–55% as (A.1, Characterization and Utilization of castor bean seed oil extract , 2015) reported. This assures castor oil can be selective oil for the synthesis of frankincense scented castor oil body soap.

4.2. Characterization of castor oil

Physio-chemical characteristics determined for extracted castor seed and oil were moisture content of seed, specific gravity of oil, viscosity, pH, acid value, %FFA, iodine value and saponification value. Their values and comparison with ASTM standard value were listed in Table 4.1 Physiochemical properties of extracted castor oil

Property	**Unit**	**ASTM standard value**	**Experimental values of this study**
Moisture content	……	1.83-5.4	4.55±0.04
Specific gravity at 25°C	g/ml	0.957-0.968	0.963
Kinematic Viscosity at 40°C	mm2/sec	…….	250
pH	……..	6.00–7.00	6.14
Acid Value	mg KOH/g	0.4-4.0	0.95±0.02
%FFA	……..	0.3–0.7	0.475
Iodine Value	mg I2/100g	82-88	80.22±1.28

Saponification Value	mg KOH/g	175-187	174.84±5.58

It can be observed from table 4.1 that the specific gravity of castor oil in this study was 0.963g/ml which is between ASTM quality standard while kinematic viscosity was found to be 250 mm/s2 this indicated the extracted castor oil was viscous to be used for further reaction. Furthermore, the iodine value of refined oil was near to the ASTM standard. This illustrated the presence of high amount of unsaturated fatty acids (double bonds) in refined castor oil and assured castor oil is non-drying oil. And also moisture content and pH were between the ASTM standard levels.

4.3. Saponification process

Before proceeding to the optimization experimental design (RSM), some preliminary experiments were conducted in order to investigate the effect of an individual parameter independently and to select the ranges of optimization parameters. was selected for conducting preliminary experiments, considering concentration of castor oil, reaction temperature, and water to lye ratio as reaction parameters selected from previously reported literatures and observed during the trials. These experiments were used to identify factors affecting moisturizing, hardness, stable lather and fluffy lather.

Table 4.2 Experiment 1.Effect of water to lye ratio

Run	Concentration of frankincense (gm)	Castor oil conc (ml)	Water to lye ratio	hardness	Fluffy lather	Stable lather	moisturizing
1	0.5	10	1.5	5.2	9	8.9	10
2	0.5	10	1.75	4.8	1.3	8.5	10
3	0.5	10	2	1.1	4	3	7

Table 4.3 Experiment 2 Effect of temperature

Run	Concentration of frankincense (%)	Castor oil conc (%)	Water to lye ratio	temperature	hardness	Fluffy lather	Stable lather	moisturizing
1	0.5	10	1.75	85	3	1.2	6.2	8
2	0.5	10	1.75	87.5	1.1	3	4	10
3	0.5	10	1.75	90	4.8	1.3	8.5	8.75

Table 4-**4** Effect of castor oil concentration on moisturizing

Run	Castor oil (ml)	Water to lye	Temperature	Mixing time	moisturizing
1	Actual(4.8ml)	0.58g/1.4ml	90	15	5
2	Actual(5.8ml)	0.67g/1.7ml	90	15	10
3	Actual(6.8ml)	0.80g/2ml	90	15	7
4	Actual(7.7ml)	0.92g/2.3ml	90	15	7
5	Actual(8.6ml)	1.03g/2.6ml	90	15	8.5
6	Actual(9.5ml)	1.15g/2.9ml	90	15	10

4.4 Quality parameters of soaps produced from castor oil

4.4.1 Effects of concentration of castor on the quality parameters

Quality parameter	standard
P^H	5.5-7.0
Specific gravity	1.0-1.2
Total fatty acids	75-85%
Unsaponifiable matter	10-20%
Free fatty acids	0.5-1.0

4.4.2 Effects of water to lye ratio on the quality parameters

The water to lye ratio (WLR) plays a crucial role in determining the quality parameters of frankincense-scented soap. Here's a breakdown of its key effects:

1. Saponification and Final Product Form:

- Ideal Ratio: A balanced WLR (around 3:1 or slightly higher) ensures complete saponification, transforming fats and lye into soap while minimizing unreacted lye (caustic soda), which can irritate skin.
- Low Ratio: Insufficient water (<3:1) leads to incomplete saponification, resulting in a soft, greasy soap with excess lye, unsuitable for use.
- High Ratio: Excessive water (>4:1) produces a hard, crumbly soap with potentially lower lathering ability. The higher water content can also dilute the fragrance of frankincense.

2. Viscosity and Curing:

- Ideal Ratio: A balanced WLR yields a medium-viscosity batter that sets and cures properly, resulting in a bar soap with desirable firmness and texture.
- Low Ratio: Thicker batter due to less water can be challenging to mold and may cure unevenly, potentially leading to cracks or inconsistencies.

- High Ratio: Runny batter with high water content might require longer curing times and may result in a softer, less-defined soap bar.

3. Lather and Cleansing:

- Ideal Ratio: A balanced WLR promotes optimal lathering without compromising cleansing power.
- Low Ratio: Soaps with less water tend to lather less due to the presence of unreacted lye, which can also be harsher on the skin.
- High Ratio: Soaps with high water content might lather more abundantly but may have reduced cleansing effectiveness due to dilution of cleansing agents.

4. Fragrance Retention:

- Ideal Ratio: A balanced WLR allows for better incorporation and retention of the frankincense scent during the saponification process.
- Low Ratio: Less water might lead to stronger fragrance initially, but the scent might fade faster due to incomplete blending with the soap base.
- High Ratio: Excessive water can dilute the fragrance oil, resulting in a weaker scent in the final soap.

4.4.3 Optimizing and quality analysis of soap from castor oil

In this experiment 1.5L castor oil was prepared through extraction and factors to be studied where basic ingredients of synthetic detergents (castor oil, NaOH,) with levels two by taking water and perfume as constant factor. The response can be measured after mixing of all those ingredients using stirrer at 100mps for 30min agitating hour. The physical characteristics: pH value, foam ability and cleaning power of the detergent were determined as the procedure discussed under section above.

Table 4.5 standard requirement for laundry soap in world (community, 2011)

Samples	Moisture content %	Free alkali %	Matter insoluble in alcohol %	Total fatty matter %	PH
Sunlight	13.24	0.06	53	41.76	9.85
Mp3	14.05	Nil	45	40.95	10.04
Canoe	16.04	0.22	43	40.96	10.51
4B	14.94	0.04	46	39.06	10.31
Glide	11.00	Nil	54	35	10.09
Lux	16.65	NIL	48	35.35	10.23
Joy	11.96	0.08	58	30.04	10.10

Table 4-6 standard requirement for soap in Ethiopia

Quality parameters	Grade 1	Grade 2	Grade 3
PH	9-11	9-11	10-11
Moisture %(m/m),max	26	28	30
TFM %(m/m),min	60	50	45
AIM %(m/m),max	2.5	15	20

Table 4.7 response of frankincense scented castor body soap

Castor oil volume (5-10ml)	Water to lye ratio	P^H value	Alcohol insoluble	Total fatty matter	Moisture content	Foam height
Actual(4.8ml)	0.6g/1.4ml	11.17	2.07	62	15.65	1
Actual(5.8ml)	0.7g/1.71ml	10.53	2.07	63	12	2
Actual(6.7ml)	0.8g/2ml	10.22	2.07	64	10.45	2.1
Actual(7.6ml)	0.9g/2.2ml	10.52	2.07	70.49	11	3.2
Actual(8.62ml)	1.0g/2.5ml	10.25	2.07	72	19	4.9
Actual(9.5ml)	1.1g/2.8ml	10.35	2.07	76.52	25	5.5

4.5. Analysis of Experimental Results

Table 8 shows the Hardness, Fluffy lather, stable lather and moisturizing, at different temperature, ratio of water to lye and concentration of castor. To analyse the experimental results, Design expert® 7.0.0 software was used. Regression analysis and analysis of variance (ANOVA) was done by using Design Expert 7.0 program. The software program was used to generate surface plots, using the fitted equation obtained from the regression analysis, keeping one of the independent variables constant.

Table 4.8 Analysis of experimental results

Std	Run	Block 1	Temperature (°c)	water to lye ratio	Concentration of castor (%)	Hardness	Stable lather	Fluffy lather	moisturizing

1	11	Block 1	85	1.5	7.5	4.2	9.5	1.1	8.5
2	3	Block 1	90	1.5	7.5	5	10	2	10
3	7	Block 1	85	2	7.5	5.1	2	4.7	8.2
4	4	Block 1	90	2	7.5	5.5	5.4	5	7
5	2	Block 1	85	1.75	5	7	8	2.5	6
6	12	Block 1	90	1.75	5	8	8.5	2	5
7	16	Block 1	85	1.75	10	3	6.2	1.2	8
8	10	Block 1	90	1.75	10	4.8	8.5	1.3	10
9	8	Block 1	87.5	1.5	5	3	10	2	8.5
10	9	Block 1	87.5	2	5	1.2	7	5	4.2
11	14	Block 1	87.5	1.5	10	5.2	8.9	9	10
12	6	Block 1	87.5	2	10	1.1	4	3	7
13	15	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
14	5	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
15	1	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
16	17	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10
17	13	Block 1	87.5	1.75	7.5	4.4	8.5	1.3	10

4.5.1. Analysis of Variance (ANOVA

Response:	Moisturizing					
ANOVA for Response Surface Quadratic Model						
Analysis of variance table [Partial sum of squares]						
	Sum of		Mean	F		
Source	Squares	DF	Square	Value	Prob > F	
Model	56.6	9	6.29	14.81	0.0009	significant
A(temperature)	0.21	1	0.21	0.5	0.5034	
B(water to lye ratio)	14.04	1	14.04	33.07	0.0007	
C(concentration of castor)	15.96	1	15.96	37.59	0.0005	
A2	3.22	1	3.22	7.59	0.0283	
B2	2.06	1	2.06	4.86	0.0633	
C2	14.8	1	14.8	34.86	0.0006	
AB	1.82	1	1.82	4.29	0.077	
AC	2.25	1	2.25	5.3	0.0548	
BC	0.42	1	0.42	0.99	0.3518	
Residual	2.97	7	0.42			
Lack of Fit	2.97	3	0.99			
Pure Error	0	4	0			
Cor Total	59.57	16				

Optimization of the soap production condition was carried out using a 2 level factorial design with three factors (temperature, water to lye ratio, and concentration of castor oil) and four responses which were hardness, fluffy lather, stable lather and moisturizing. Statistical analysis was carried out to determine the correlation coefficients of the model as a function of the responses. The sequential model sum of squares for soap production is summarized in Table 4.9. From table 4.9 due to the highest value of the "Adjusted R-Squared" and the "Predicted R-Squared" and the model is not aliased, the quadratic model is suggested among the other models.

Table 4.9. The model summary statistics

Source	**Std. deviation.**	**R-squared**	**Adjusted R-squared**	**Predicted R-squared**	**PRESS**	
Linear	1.75	0.1633	-0.0298	-0.7514	83.48	
2FI	1.96	0.1952	-0.2877	-3.1347	197.08	
quadratic	1.59	0.6267	0.1468	-4.9726	284.68	suggested
Cubic	0	1	1	N/A	Not defined	Aliased

Note: R-squared this statistic indicates the percentage of the variance in the dependent variable that the independent variables explain collectively. R-squared measures the strength of the relationship between your model and the dependent variable on a convenient.

Adjusted R-square: The adjusted R-squared is a modified version of R-squared that accounts for predictors (input variables) that are not significant in a regression model. In other words, the adjusted R-squared shows whether adding additional predictors improve a regression model or not. Compared to a model with additional input variables, a lower adjusted R-squared indicates that the additional input variables are not adding value to the model.

Predicted R-square: The predicted R-squared indicates how well a regression model predicts responses for new observations. Can be negative but always lower than R-squared.

negative "Pred R-Squared" implies that the overall mean is a better predictor of your response than the current model.

4.5.2 ANOVA for Response Surface Quadratic Model

To determine whether or not the quadratic model is significantly affected by the parameters Listed in the design it was crucial to perform an analysis of variance (ANOVA). To check the significance of each coefficient, which also showed the interaction strength of each parameter, the probability values (P-values) were used to perform as a device. The bigger the significance of the corresponding coefficient resulted from the smaller the p-values. The Model F-value of 14.81 implies the model is significant.

There is only a 0.09% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case B, C, A2, C2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

Table 4-1 model adequacy measure

Std. Dev.	0.65164627	R-Squared	0.95010121
Mean	8.37647059	Adj R-Squared	0.88594563
C.V.	7.77948493	Pred R-Squared	0.20161943
PRESS	47.56	Adeq Precision	12.4552344

Development of regression model equation: The model equation that correlates the response (moisturizing) to the process variables in terms of actual value after excluding the insignificant terms was given in (Equation 1 and .2). A model equation is a mathematical expression in which the whole model was expressed in a single equation that helps to maximize response. The predicted model for the moisturizing in terms of the actual factors and coded factors is given in (Equation 1 and 2). The final equation in terms of actual factors:

Final Equation in Terms of Actual Factors:

Moisturizing = -1193.50000+25.55500* temperature+124.50000 *water to lye ratio 6.34500 * concentration of castor -0.14000* $temperature^2$ -11.20000 * water to lye $ratio^2$ -0.30000 * concentration of $castor^2$ -1.08000* temperature * water to lye ratio +0.12000 temperature * concentration of castor +0.52000 * water to lye ratio * concentration of castor………………….(Equation 14final equation in terms of actual factors

Final Equation in Terms of Coded Factors:

Moisturizing =+10.00+0.16 * A -1.32 * B+1.4* C-0.88 * A^2-0.70 * B^2-1.87* C^2 -0.68*A*B+0.75 * A * C+ 0.33 * B * C……………Equation 15 final equation in terms of coded factors

The equation developed from the regression model terms of coded factors represented the moisturizing response. The moisturizing was as response and affected by linear terms such as temperature (A), water to lye ratio (B) and castor oil concentration (C), and pure quadratics terms (A^2, B^2 and C^2) and interaction quadratic terms (AB, AC, and BC). Based on the coefficients in (equation 2).

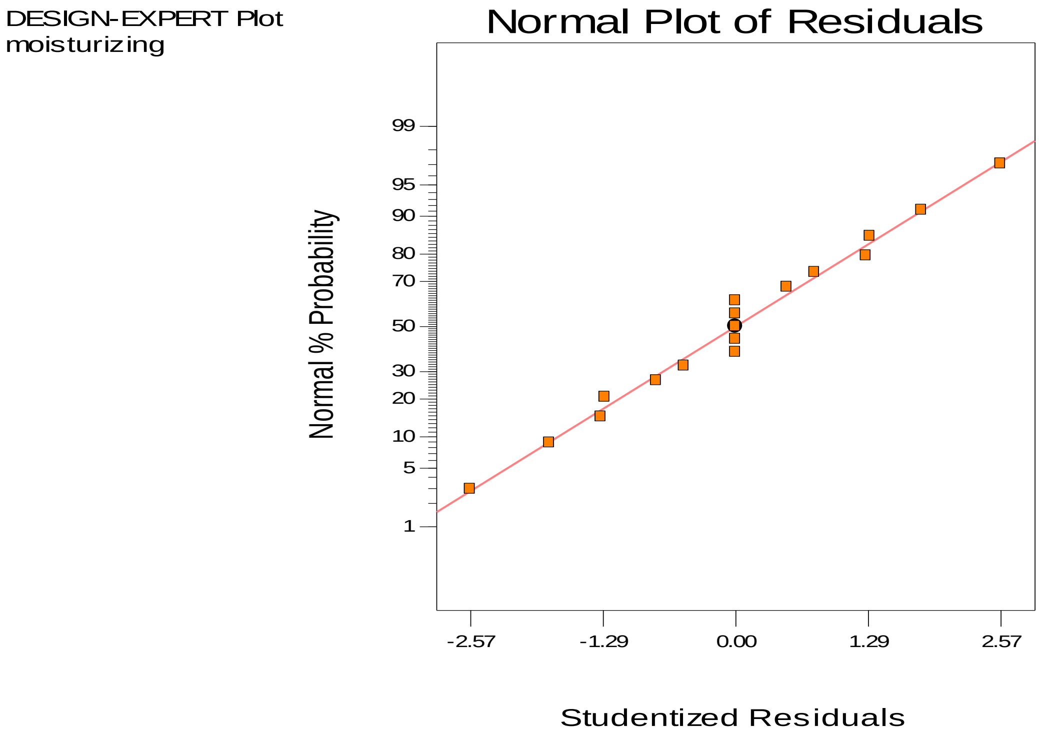
Based on the coefficients in (equation 2), it was clear that the percentage of moisturizing response increases with the increase of temperature (A) and concentration of castor (C). While water to lye ratio (B) have a negative linear effect on moisturizing yield. While temperature(A),water to lye ratio(B) and concentration of castor (c) have negative quadratic effect of moisturizing response.

Interaction of temperature and water to lye ratio have also a negative effect on the response while the interaction of mixing time and temperature (AC) and also the interaction of water to lye ratio and concentration of castor (BC) has positive effect on moisturizing response.

4.5.3. Normal probability plot

In the case of this experimental data the points in the plots are in a good fit to the straight line as shown in the normal probability plot, (Figure 7) and it indicates the residuals following by the normal % probability distribution, and this shows that the quadratic polynomial model satisfies the analysis of the assumptions of ANOVA implying the error distribution is approximately normal.

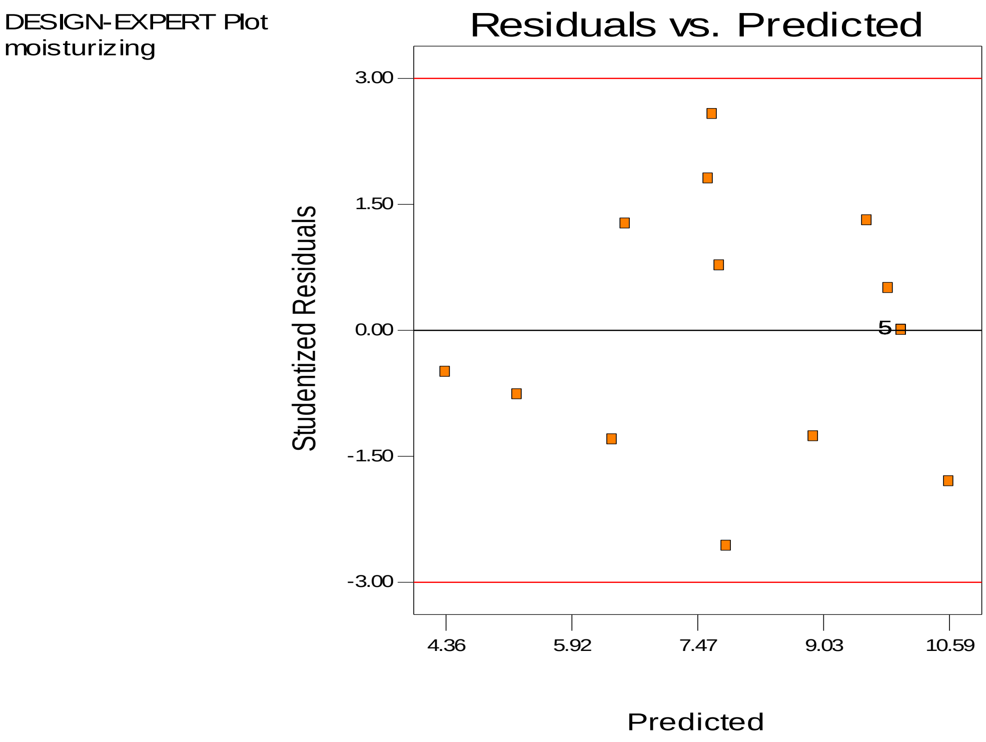
Figure 4-1 normal probability of residuals versus studentized residuals value of soap production



4.5.4 Residual versus Predicted Plot

If the model is correct and the assumptions are satisfied, the residuals should be structured less; in particular, they should be unrelated to any other variable including the predicted response. For correct model and satisfied assumptions, the residual value must be structure less; in particular, they should be unrelated to any other variable including the predicted response. A simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the assumption of constant variance. The plot shows random scatter or structure less which satisfied the assumption of the constant variance as shown in Figure 8. A simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the predicted response values tests the assumption of constant variance. The plot shows random scatter which justifying no need for any alteration to minimize personal error.

Figure 4-2 studentized residuals versus predicted values of soap production



4.5.6 Effect of temperature on moisturizing of frankincense scented castor oil body soap

As shown figure 4.3 effect of temperature on moisturizing of frankincense scented castor oil body soap had optimum point at 87.50^{0}c. Further increment of temperature showed decrement impact on moisturizing behaviour of the body soap. And also slight decrease in temperature showed decrement.

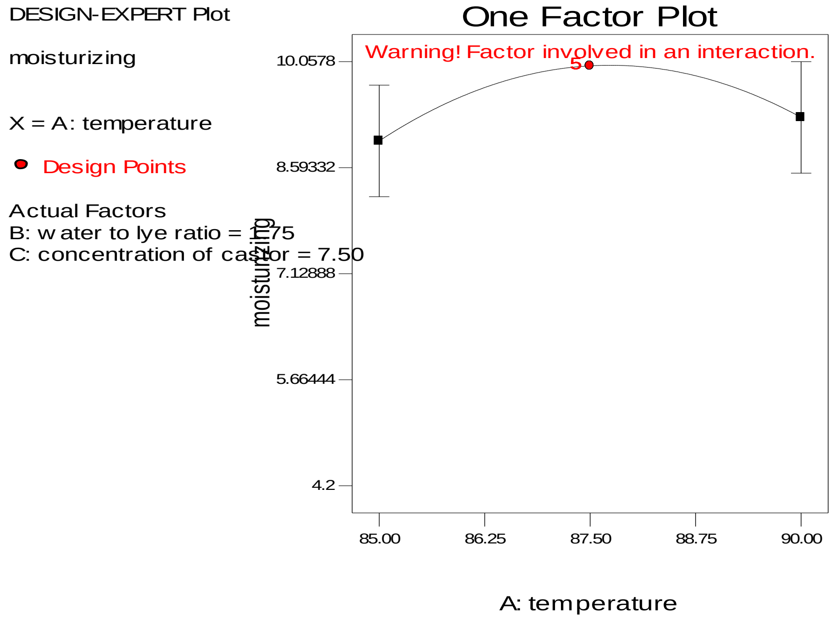


Figure 4.3 Effect of temperature on moisturizing

4.5.7 Effect of water to lye ratio on moisturizing

As shown figure 4.4 there was optimum point at 1.75 the water to lye ratio in soap making plays a crucial role in determining the moisturizing properties of body soap. The ratio affects the amount of lye that reacts with oils or fats to create soap. Higher water to lye ratio, also known as a "lye-heavy" ratio, can result in a soap that is more cleansing but may be less moisturizing. This is because excess lye can potentially strip away natural oils from the skin, leading to dryness. On the other hand, lower water to lye ratio, also known as a "lye-deficient" ratio, can create a soap that is more moisturizing. This is because a lower amount of lye allows for a higher proportion of oils or fats to remain in the final soap, providing additional nourishment and moisturization to the skin. It's important to find the right balance in the water to lye ratio to achieve a soap that cleanses effectively without drying out the skin. This can vary depending on the specific recipe and the desired characteristics of the soap. Experimentation and understanding the needs of your skin can help in finding the optimal water to lye ratio for moisturizing body soap.

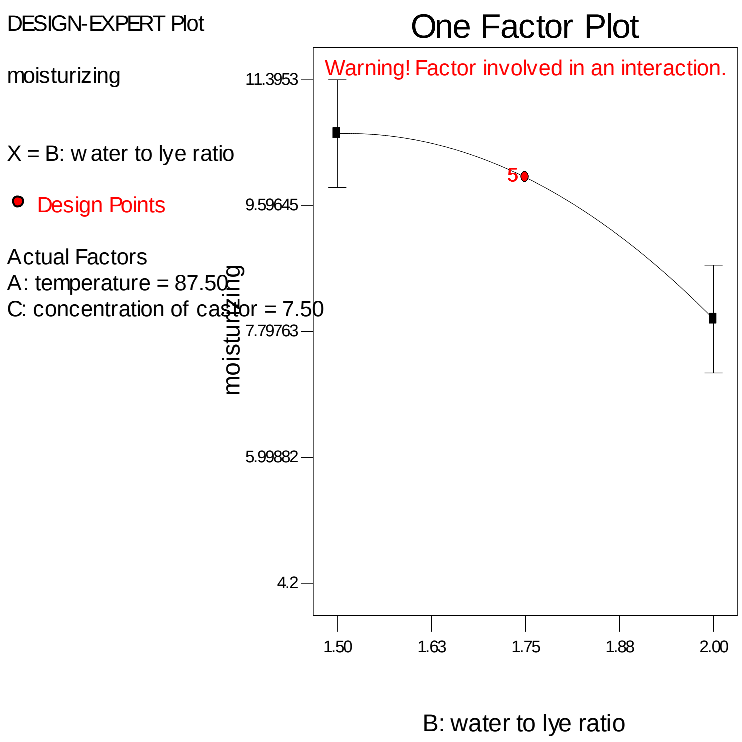


Figure 4-4 Effect of water to lye ratio on moisturizing

4.5.8 Effect of concentration of castor oil on moisturizing

As shown in figure11 the concentration of castor oil in body soap can have a significant impact on its moisturizing properties. Castor oil is known for its moisturizing and emollient properties, making it a popular ingredient in skincare products. Higher concentrations of castor oil in body soap can increase its moisturizing effects. Castor oil helps to retain moisture in the skin by forming a protective barrier that prevents water loss. It also has humectant properties, which means it attracts and retains moisture from the environment, further enhancing its moisturizing benefits. However, it's important to note that using castor oil in high concentrations may also affect the overall texture and lather of the soap. Castor oil has a unique chemical composition that can contribute to a softer and more conditioning bar of soap, but excessive amounts may result in a sticky or slimy texture. Finding the right concentration of castor oil in body soap depends on personal preference, skin type, and the desired characteristics of the soap.

It's recommended to start with smaller concentrations and gradually increase or decrease based on the desired level of moisturization and the overall performance of the soap. The optimum value of concentration of castor is at 8.5 percent.

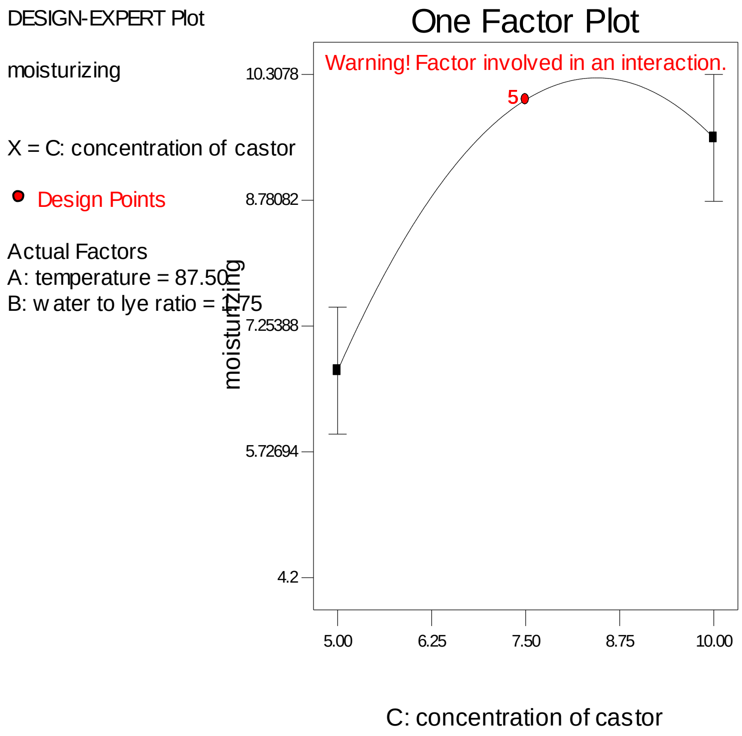


Figure 4-5 Effect of castor oil concentration on moisturizing

4.5.9 Interaction Effects of Experimental Variables on moisturizing for Soap Production

In soap production the best way of showing the effects of this parameter on the yield of moisturizing is to generate response surface plots of the equation. The three-dimensional response surfaces effect was plotted in (Figures 8-9) as a function of the interactions of any two of the variables by holding the other value of the variable at the center point. In order to analyse the regression equation of the model, three-dimensional surface and 2D contour plots were obtained by plotting the response on the Z-axis against any two variables while keeping the other variable at the center level. These plots are created to analyse the change in the response surface.

4.5.9.1 Interaction effects of temperature and water to lye ratio with moisturizing value to produce frankincense scented castor oil soap production

In the Interaction effect of temperature and water to lye ratio with moisturizing as shown in (Figure 4.6), moisturizing yield obtained at near to the center and then the yield decreases at any direction in the model. This might be due to the reason that oil was converted to other by product like glycerine. Generally, as the graph indicates the levels of water to lye ratio and temperature resulted in higher yield of moisturizing.

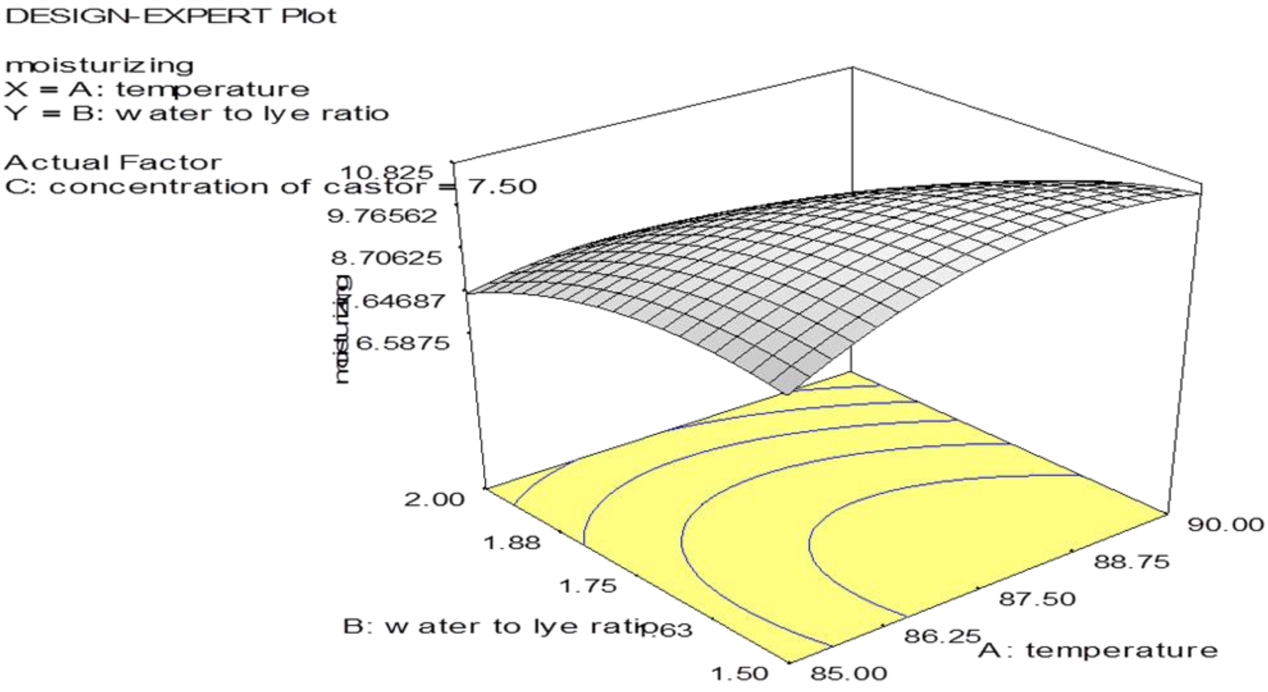


Figure 4-6 normal response plots of the effect of water to lye ratio and temperature with moisturizing of the soap

4.5.9.1 Interaction effects of temperature and water to lye ratio with stable lather to produce frankincense scented castor oil soap production

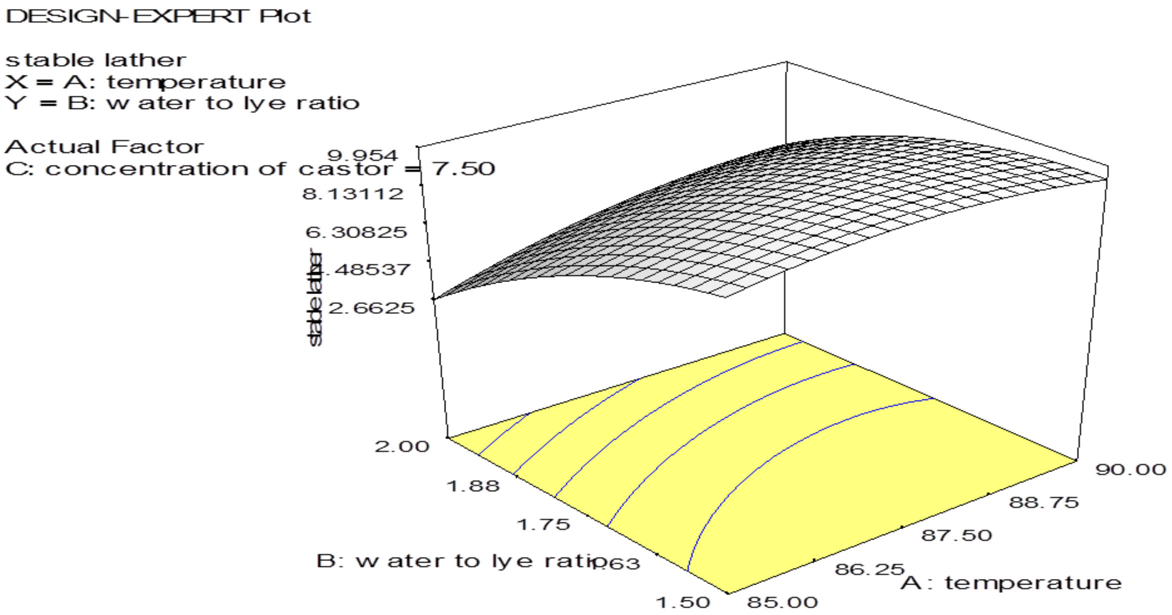


Figure 4-7 normal response surface plots of the effect of water to lye ratio and temperature with stable lather

4.6 Optimization of Soap Production Parameters Using Response Surface Methodology

Response surface methodology is a collection of mathematical and statistical techniques useful for improving, developing, and optimizing processes. It also has important applications in the improvement of existing product designs as well as in the design, development, and formulation of new products. The optimization of soap production parameters from castor seed oil was summarized in table

The yield of moisturizing can be optimized by manipulating the process parameters in soap production. In order to optimize the response, the function of desirability was applied using Design Expert software version 7.0. In this study, numerical optimization was chosen which presents a comprehensive and up-to-date description of the most effective methods in process optimization. To do so, the upper and lower limit of each the process parameters in soap making from castor oil and its response as predicted by the model 60 were provided based on the contour and overlay plot obtained. The goal of optimization was to maximize economic benefit and increasing the TFM content by minimizing process cost and model variables to investigate the optimum values of sop production from castor oil as summarized in table 8

Table 4-2 constraints for optimization of soap production

Name	Goal	Lower limit	Upper limit
temperature	In range	85	90
Water to lye ratio	In Minimum	1.5	2
Concentration of castor	Maximize	5	10
Hardness	Minimum	1.1	8
Stable lather	Maximum	2	10
Fluffy lather	Maximum	1.1	9
moisturizing	Maximum	4.2	10

4.6.1 Model Validation

Using the optimized condition obtained from the two level factorial designs (i.e. table 17), an experiment was conducted. The moisturizing yield of 10 % was obtained. So, it is possible to say that this is in good agreement with the predicted one. Therefore, the model is considered to be accurate and reliable for predicting the soap production. The desirability lies between 0 and 1 and it represents the closeness of a response to its ideal value. If a response falls within the unacceptable intervals, the desirability is 0, and if a response falls within the ideal intervals or the response reaches its ideal value, the desirability is 1. So in this case the desirability value of 0.727 indicates the closeness of the response to the ideal value.

Table 4-3 the optimized solution production of soap

Number	temperature	water to lye ratio	concentration of castor	Hardness	stable lather	fluffy lather	moisturizing	Desirability	Column
1	87.77	1.5	10	3.23358	9.76514	6.962	9.99997	0.89	Selected
2	87.71	1.5	9.98	3.21301	9.75615	6.92405	9.98311	0.889	
3	87.69	1.5	9.96	3.21621	9.75472	6.87145	9.99998	0.887	
4	86.8	1.5	10	3.04333	9.49715	6.7691	9.32224	0.868	

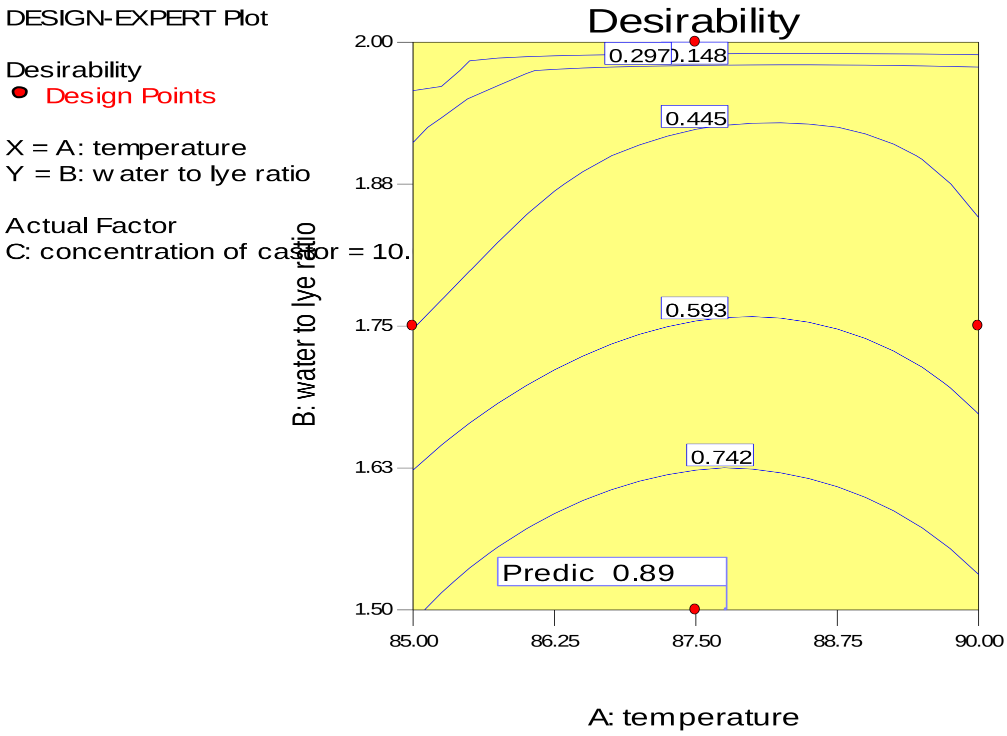


Figure 4-8 contour plots for the optimization process

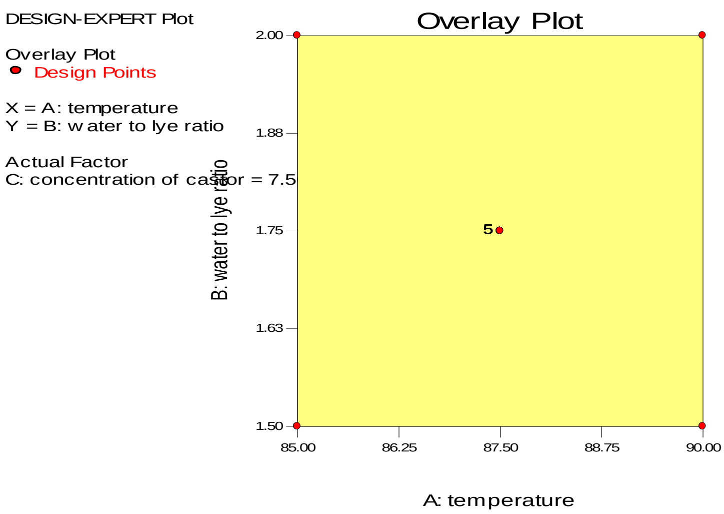


Figure 4-9 overlay plots for the optimization process of the effects soap production parameters

4.7 characterization of frankincense scented castor oil body soap

Table 4-4 characterization of frankincense scented castor oil body soap

Base oil	TREATMENT	PH VALUE	ALCHOL INSOLUBLE	TOTAL FATTY MATTER	MOISTURE CONTENT	foam height
Castor oil volume (5-10ml)	Water to lye ratio					
Actual(4.7ml)	0.5g/1.429ml	11.17	2.07	62	15.65	1
Actual(5.7ml)	0.6g/1.714ml	10.53	2.07	63	12	2
Actual(6.7ml)	0.8g/2ml	10.22	2.07	64	10.45	2.1
Actual(7.6ml)	0.9g/2.2ml	10.52	2.07	70.49	11	3.2
Actual(8.6ml)	1.0g/2.5ml	10.25	2.07	72	19	4.9
Actual(9.5ml)	1.1g/2.8ml	10.35	2.07	76.52	25	5.5

4.7.1. FTIR Analysis

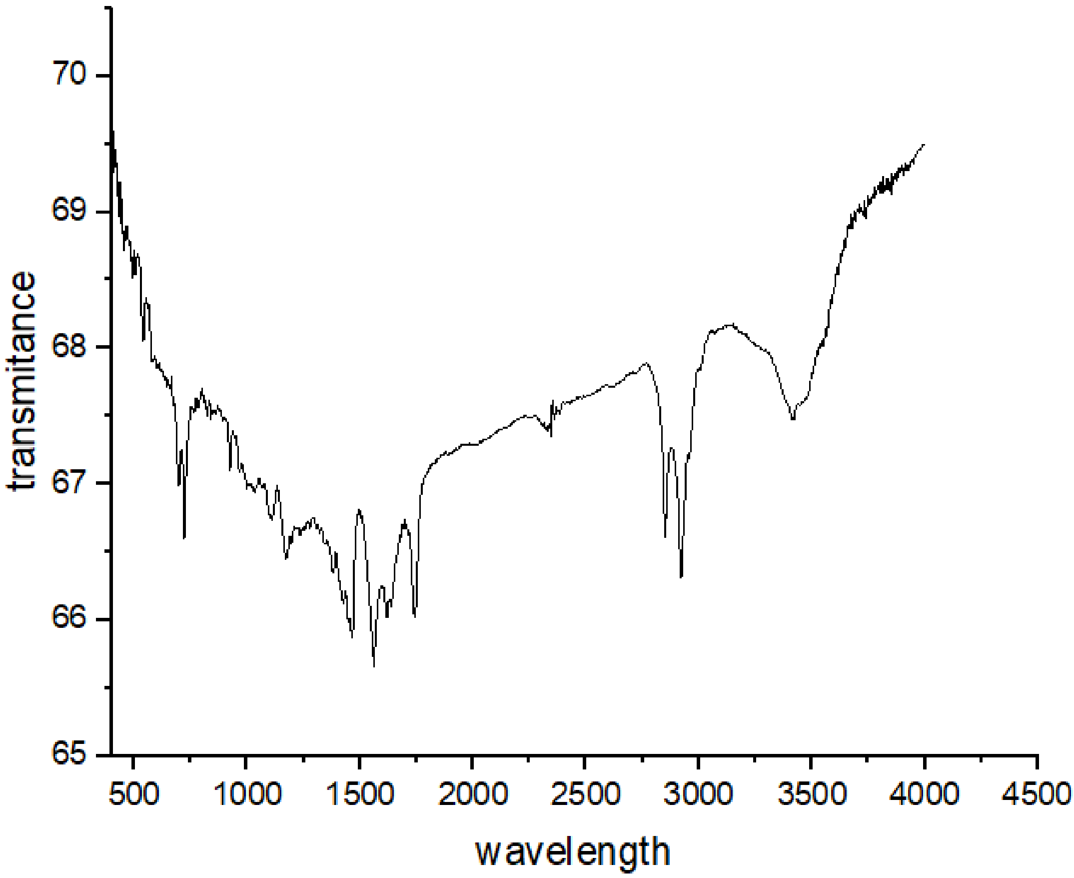


Figure 4-10 FTIR plot of frankincense scented castor oil soap

As shown in figure 4.10 FTIR analysis, namely 2852.6, 2921.8, 1745.6, 1462.2, 1563.8, 1175.6, and 719.8 cm^-1, suggest the presence of specific functional groups in the soap's chemical composition. The wavenumbers 2852.6 and 2921.8 cm^-1 indicate the presence of aliphatic hydrocarbon chains or fatty acids, while 1745.6 cm^-1 suggests the presence of a carbonyl-containing compound. The wavenumber 1462.2 cm^-1 provides further evidence of hydrocarbon chains, while 1563.8 cm^-1 suggests the presence of unsaturated compounds. The wavenumber 1175.6 cm^-1 indicates the presence of ether or alcohol groups, and 719.8 cm^-1 suggests the presence of aromatic rings. This confirms the synthesised soap. (Mirghania, 2002)

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

In summary, in this study the soap has been produced from castor seeds and frankincense oil by using pre-treatment of castor seed, extraction of castor oil, neutralization and saponification to the literature; using screw press 35% up to 55 %, oil was extracted. The low yield was attributed to the nature of the seeds. Moreover, the saponification process and neutralization gave a powered soap of high enough efficiently as seen from the result of the foam ability test resulting in a foam height of 2.5 cm that persisted for about 4 minutes since the efficiency of washing power was assessed through the amount of foam produced. The reaction proceeds at temperatures between 85-90°C. PH tests showed that the soap exhibited basic property. All basic soap parameter such as TFM, FC, alcohol insoluble matter and moisture content of produced laundry soap from content, TFM, AIM, FCA and Titrate value found in all of the batch studied were 10.7, shown that using castor seed oil for soap production is feasible and be scaled soap production.

5.2 Recommendation

Ethiopia is known for its rich biodiversity and traditional knowledge of herbal medicine. One recommendation for natural soap production in Ethiopia is to incorporate locally sourced ingredients with beneficial properties should be used for a good quality of laundry soap and castor oil investigated in the purification of saponification waste and conversion into value-added . Prioritize organic and sustainable practices: Ethiopia has a strong focus on organic agriculture, so consider using organic ingredients and adopting sustainable production methods. This can include using eco-friendly packaging, minimizing waste, and utilizing renewable energy sources in your production process. Ingredients like optical brightness and EDTA should be used for a good quality of body soap.

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APPENDIX

Appendix A: Laboratory works

Appendix A1: Pictures showing experimental works and laboratory results (Author)