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Extraction and Optimization of Oil from Sesame Seed By using n hexane Solvent

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Declaration

Thesis is prepared by me. The thesis is entitled "Extraction and optimization of oil from sesame seed by n hexane solvent", It is submitted in partial fulfilment of the requirement for the Degree of Masters Science in Addis Ababa Institute of Technology School of chemical engineering complies with the regulation of the university and meets the accepted standards with respect to originality and quality. I declare that it is done by myself and that it is not copied from other work.

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List of acronyms

GCMS Gas chromatography mass spectroscopy

DT desolventizer Toaster

FFA Free fatty Acid

ALA alpha linolenic acid

RDA recommended daily allowance

KEYWORDS: Sesame seed, extraction, response surface methodology, oil yield, Design Expert.

Abstract

Optimization of sesame oil extraction using response surface methodology (RSM) was carried out in this study. The effects of three factors (moisture content of seed, extraction temperature and extraction time) on the oil yield obtained from the seed were considered. With the aid of Design Expert and using the central composite design of the response surface methodology, twenty (20) experiments were generated and run. Using n-hexane as the solvent, each experimental run was carried out in 100ml Soxhlet extraction apparatus. The results obtained from the experiments together with the factors considered during the experiments were modelled and analyzed using a quadratic model based on observations made during the model analysis. The reults obtained from the quadratic model for this extraction were R-squared, the adjusted R-squared and the predicted R-squared values of the developed and modified quadratic model were 0.9986, 0.9975 and 0.9955. Also, with the aid of the Design Expert, the optimum parameters of the process were estimated to be moisture content of 6%, extraction temperature of 83 ºC and extraction time of 7 hr. It was finally concluded that the optimum values of the parameters given by the software were genuine ones, because the validation of the optimum values obtained experimentally showed a very good conformity between the predicted optimum oil yield and the experimental one. The quadratic models developed predicted 55.8% as the maximum oil yield while the one obtained from the experiment was found to be 50.17%. Therefore, the application of response surface methodology to optimize oil extraction from sesame seed has been successfully demonstrated.

Chapter one

Introduction

1.1 Background

Sesame (Sesamum indicum) from Pedaliaceae, is an important oil seed crop being cultivated in the tropics and the temperate zone of the world (Biabani and Pakniyat, 2008). It is one of the oldest oil crops and is widely cultivated in Asia and Africa (Ali et al., 2007). It was a highly prized oil crop of Babylon and Assyria at least 4000 years ago (Ross, 2005). This plant is called “sesame” internationally, while it is called “benniseed” in West Africa, “simsim” in East Africa and“Till” in India. Within Ethiopia, it is called “selit”. This crop is grown mostly for the oil extracted from its seed which is edible and use for industrial and pharmaceutical purposes (Ogbonna and Ukaan2013). It ranks ninth among the top thirteen oilseed crops which make up 90% of the world production of edible oil (Adeola et al., 2010).

Natural sesame oil derived from good quality seed has a very pleasant flavour and can be consumed without further purification. The natural sesame oil has excellent stability due to the presence of high level of natural antioxidants (Lyon, 1972).The oil is also useful in the industrial preparation of perfumery, cosmetics (skin conditioning agents and moisturizers, hair preparations, bath oils, hand products and make-up), pharmaceuticals, insecticides and paints and varnishes (Warra, 2011). Generally, oil is extracted from sesame seed using methods like Soxhlet extraction, supercritical extraction, etc. Soxhlet method is one of the most popular methods used for lipid extraction from natural products and foodstuffs. According to this method, lipids are extracted from solids by multiple rinsing out with an organic solvent like hexane or any low-boiling petroleum ether (Carvalho et al., 2012). However, the efficiency of Soxhlet extraction, usually evaluated using oil yield has been discovered to be affected by operating conditions such as temperature of extraction, seed size and extraction time. So, in order to obtain good yield of oil from this seed, there is the need to obtain the optimum values of these parameters affecting the extraction of oil from the seed.

1.2 Problem of statement

Edible oil is mainly produced from cotton seed, rape seed, lint seed, nigger seed, sesame seed, palm and vegetables. In Ethiopia, the first two types are dominant in the market followed by lint and nigger seed. Vegetable and palm oil are imported products while sesame oil is not easily available in the market. Sesame is one of the major export earning oil seeds. Due to the highest price offer in the international market as against the depressed price of domestic edible oil, the manufacturing of edible oil is mainly processed from cotton seed and rape seed, which have lesser prices against sesame and other oil seeds.

Edible oil is part of everyday food expenditure in urban areas as well as in most of the rural areas. Sesame oil is also used as an input for industrial production of pharmaceuticals, soap and cosmetics.

1.3 Objective

1.3.1 General Objective

Extraction of sesame oil from sesame seed using hexane solvent and optimization using surface response method.

1.3.2 Specific Objective

- To investigate the effect of moisture on the extraction process.
- To investigate the effect of temperature on the extraction process.
- To Optimize of Soxhlet extractor for different temperature, seed moisture content and extraction time by using hexane solvent.
- To Characterization of final oil at optimum condition such as acid value, FFA, iodine value, density.

1.4 Significance

When this thesis project scale up to industry can create a job for many people. The establishment of such factory will have a foreign exchange saving effect to the country by substituting the current imports. The project will also create forward and backward linkage with the manufacturing sector and also generates income for the Government in terms of tax revenue and payroll tax.

- This study will contribute to a significant improvement from traditional methods to technological manufacturing of the oil for the use of sesame extracts.

- This work will show the possibility of getting the oil and this can be used us an additive in soap and cosmetic industries to improve the performance quality of the product.

- The experimental work showed that the possibility of getting the appropriate operating conditions for extraction of sesame oil.

1.5 Scope of the Study

The thesis studies in maximizing the yield of edible oil using soxhlet extraction. By using surface response methodology to optimize the product, to characterize the oil obtained at optimum parameter using gas chromatography mass spectroscopy.

Chapter Two

Review of Literature

2.1 History of sesame seed

Sesame seeds are the seeds of the tropical annual Sesamum indicum. The species has a long history of cultivation, mostly for its yield of oil. The oil plant has been grown since the beginning of arable cultivation, and originates from the dry bush savannah of tropical Africa, and spread from there to India and China, where it is still widely cultivated. (Naturland, 2002).The original area of domestication of sesame is obscure but it seems likely to have first been brought into cultivation in Asia or India (www.nigeriamarkets.org). Archeological records indicate that it has been known and used in India for more than 5,000 years and is recorded as a crop in Babylon and Assyria some 4,000 years ago (Borchani et al., 2010).

Sesame was cultivated during the Indus valley civilization and was the main oil crop. It was probably exported to Mesopotamia around 2500 BCE and was known in Akkadian and Sumerian as 'ellu'. Prior to 600 BC, the Assyrians used sesame oil as a food, salve, and medication, primarily by the rich, as the difficulty of obtaining it made it expensive. Hindus used it in votive lamps and considered the oil sacred.

2.2 Production of sesame seed

Global production of sesame seed is estimated by FAO at 3.15 mn tonnes per year (2001) having risen from 1.4 mn tonnes in the early 1960's. However only a small proportion of the global sesame harvest enters international trade. For the most part, the oil is expressed locally and used locally for cooking or the seeds themselves are eaten, particularly after being fried. Sesame is grown in many parts of the world on over 5 million acres (20,000 km2). The largest producer of the crop in 2007 was India, China, Myanmar, Sudan, Ethiopia, Uganda and Nigeria. Seventy percent of the world's sesame crop is grown in Asia, with Africa growing 26% (Hansen, 2011)

The largest producers are China and India, each with an annual harvest around 750,000 tonnes followed by Myanmar (425,000 tonnes) and Sudan (300,000 tonnes). These figures are only rough estimates of the situation as sesame is a smallholder crop and much of the harvest is consumed locally, without record of the internal trade and domestic processing (CBN, 2009).

2.3 Sesame seed cultivation

Sesame (Sesamum indicum L.) is a self-pollinating annual plant having a straight, pubertal stem with branching and is known one of the most important oilseed crop. Sesame is a blossoming plant and belongs to the genus Sesamum. Various wild relatives found in Africa and a minor number in India and other parts of the world. It is extensively naturalize in tropical areas in the world. It is grown for their edible seed that grows in pods. Its flowers are light yellow, although flowers are also differ in color with various being purple or blue (Morris, 2002).

Sesame cultivation can be carried out on a wide range of soils but optimum are well-drained, loose, fertile and sandy alluvial soils that have a pH value between 5.4 and 6.75. Very low pH values have a drastic effect on growth, whereas some varieties can tolerate a pH value up to 8 (Naturland, 2002). Good drainage is crucial, as sesame is very susceptible to short periods of waterlogging. Sesame is intolerant of very acidic or saline soils (Bennet, 2011). The total amount of water required to grow sesame crop ranges from 600 to 1000 mm, depending on the cultivar and the climatic conditions (Hansen, 2011). Good harvests can be expected when rainfall of 300-600 mm is optimally spread throughout the vegetation period. During each of its development stages, the plant is highly susceptible to water-logging, and can therefore only thrive during moderate rainfall, or when irrigation is carefully controlled in drier regions (Naturland, 2002). The water requirement can be met from available soil moisture at sowing, rainfall during the growing season and irrigation (Hansen, 2011). This is because due to its tap roots, the plant is highly resistant to drought, and can provide good harvests even when only stored soil water is available. When irrigated, or during summer rain spells, sesame grows better in sandy than in heavy soils. This is due to its sensitivity to high soil moisture contents (Naturland, 2002).

Sesame needs long periods of sunshine, and is generally a short-day plant, where by varieties exist which are unaffected by the length of the day (Naturland, 2002). Sesame needs a constant high temperature, the optimum range or growth, blossoms and fruit ripeness is 26-30°C. The minimum temperature for germination is around 12°C, yet even temperatures below 18°C can have a negative effect during germination (Naturland, 2002). Pollination and the formation of capsules is inhibited during heat-wave periods above 40°C. In regions visited by strong, hot winds, the plants only form smaller seeds with a lower oil content. Sesame is sensitive to strong winds when the main stem is fully grown. Tall varieties should not be planted in regions which have strong winds during the harvesting season (and, if necessary, hedges to protect against the wind should be planted) (Naturland, 2002). The response of sesame to both temperature and daylength indicates that it should be cultivated in the wet season in the tropics, or in the summer in the warmer temperate areas. While there is some variation between cultivars, the base temperature for germination is about 16oC (Bennet, 2011). This warm-season annual crop is primarily adapted to areas with long growing seasons and well-drained soils (Hansen, 2011, www.nutrition-and-you).

Sesame is growing to 50 to 100cm (1.6 to 3.3ft) tall in length and it is an annual plant with conflicting leaves 3 to 14cm (1.5 to 5.6 inch) extended with an whole edge; leaves are broad lanceolate, to 5cm (2 inch) wide at the bottom of the plant, lessening to just 1cm (0.4 inch) broad to the blossoming stem. The floras are purple to white, cylindrical, 2 to 5cm (1.1 to 1.9 inch) long, lobed mouth with a four edges. The seed cake is an excellent source of protein supplement in the animal feed industry. Sesame seed are pear-shaped, ovate, small (about 2.5-3mm in length, 1.5mm in width), slightly flattened and thinner at the helium. Weight of 1000 seeds is approximately 3g. Light-colored seeds yield better quality oil than dark color but lower oil content (Mukhopadhyay and Ray, 1999).

Figure 2.1 A Picture of the Sesame Plant with the Seeds

2.4 Sesame seed description

Sesame has been considered crop that can be used as animal feed and human foods as a whole seed, oil, and meal (Hahma *et al*., 2009). The sesame is not an excellent source of starch. The sesame seeds contain approximately a quarter of soluble fibers out of the total fiber present in sesame. The main part of soluble fiber is mucilaginous gum and its composition ranges from 8-11g per 100 g. The alpha linolenic acid (ALA) which is an omega-3 fatty acid forms the major part of polyunsaturated fatty acids in sesame. It is present about 50% of the total fatty acids. ALA is essential amino acid and cannot be synthesized by the human body from any other substance therefore it is considered as an essential fatty acid. The essential fatty acids requirements for the human body can be fulfilled by intake of sesame products (Morris, 2004). A hundred grams of sesame provides 100% of the recommended daily allowance (RDA) for manganese and potassium, 57-65% of the RDA of phosphorus and iron, and 13-35% for zinc, calcium and copper while its recommended daily intake is 25 to 50 grams (Anon, 2006). Specialty grains (bulgar wheat, barley, sesame, sprouted grains and rice extract) are used in breakfast cereals to add novel textures, flavors and colors in these products.

The unique flavor and its opposition to oxidative corrosion make the sesame oil an outstanding salad oil. On an average, two third sesame seeds of the worlds are processed into oil and meal (Kang *et al*., 2003). The quality of the oil and variety of yield continue to raise (Bandyopadhyay and Ghosh, 2002). In addition to using sesame oil for cooking foods, it may be used for nutraceutical food (Morris, 2002). In 2003, the reserved area for sesame planting in the world was 6.57 million hactares; with a production rate of 3,096 million tonnes per year and an average yield of 471.2kg/ha (Ofosuhene *et al*., 2010). In recent times, a nutraceutical diversity of sesame uses have been open to the elements from such as sesamolin, assesamin, sesamol are lipidsoluble antioxidants and sesaminol, lignan glucosides and sesame lignans are originate to be antioxidative and health benefiting. Sesame meal is a significant protein resource for the utilization of mankind because of the presence of sulfur-containing AA. It has protein content ranging from ~45–55%. Sesamolin which is derived from seeds of sesame inhibit expansion of leukemia cells in human (Bandyopadhyay and Ghosh, 2002).

The consumption of whole sesame seed shows the increase plasma γ-tocopherol and improved vitamin E action, which is believed to reduce heart disease and cancer. Sesame oil is considered to satisfy compound organ failure and boost endurance speed during endotoxemia in rats, while defending aligned with lipopolysaccharidemotivated oxidative and influencing constructively the blood glucose, glycosylated hemoglobin, antioxidant levels and lipid peroxidation in diabetic. Sesame intake also affects confidently antioxidant status, blood lipids and sex hormones in postmenopausal women (Wu *et al.*, 2006).

Sesame is cultivated broadly from humid to moderate regions of the world. Sesame seeds contain normally 25– 35% of protein and 55% of oil. The oil is rich in unsaturated fat. The richest fatty acids in sesame oil are linoleic acid (37–42%) and oleic acid (41–45%) (Kang *et al.*, 2000: Kim *et al.*, 2002). The effects of germination on the chemical composition and biochemical constituents of seeds differ to a great degree with the plant kind, seed cultivars or varieties and the germination circumstances. Such changes are important to the marketable applicability of sesame, more so if the health-improving functional constituents are sought. Nevertheless, sesame in recent times recognized as a chief cause of allergic reactions between infant and young children and a young woman that is amazing as sesame oil is used widely in the cosmetic and pharmaceutical industries because of its apparently low antigenicity (Caminiti *et al.*, 2006). Allergens that belong to seed storage proteins, ordinary food allergens have been recognized in sesame seeds. Therefore it is thus significant to check the protein level in sesame. As the investigation of biochemical and chemical ingredients provided by germinated seeds leftovers deficient, the benefits and limitations of germinating seeds utilized for human have not been recognized. To maintain the advantageous level of sesame seeds for marketable utilizations, it is vital to outline their compositional changes during and after germination
(Beyer *et al.*, 2002).

Sesame is grown for its seeds and the primary use of the sesame seed is as a source of oil for cooking. The young leaves may also be eaten in stews and the dried stems may be burnt as fuel with the ash used for local soap making but such uses are entirely subordinate to seed production (Table 2.1). The crop of sesame is commercialized in a number of forms. Most sesame seeds are processed directly into oil by the grower or within the producing region but are also sold in various stages of processing, for various uses, such as meal, paste, confections and bakery products (Salunkhe et al., 1991).

Table 1.1 Products of sesame and its uses

Part used Seeds	Products	Description
Seeds	Confectionery and Biscuits	Fried seeds bound together with sugar syrup, whole seeds baked into Biscuits, popular in northern Europe either incorporated into breads or as decorative toppings, a paste of sesame seeds is used as an ingredient in eastern Mediterranean and Middle Eastern foods
Oil	Varied uses	To treat ulcers and burns, low grade oil is used in making soaps, paints, lubricants, and illuminants
Cake	Food and feed	Protein rich useful supplement, used in some Indian cooking

The Carbohydrates in sesame seed are composed of 3.2% glucose, 2.6% fructose and 0.2% sucrose while the remaining quantity is dietary fibers. The nutrient composition of sesame seeds is enlisted in Table 4 and 5. Also, they have desirable physiological effects including antioxidant activity, blood pressure and serum lipid lowering potential as proven in experimental animals and humans (Sirato-Yasumoto et al., 2001). The major protein fraction (globulin) in sesame contains about 95% of 13S globulin and seems to be a simple, salt soluble, very susceptible to heat denaturation and similar in subunit structure to soybean 11S globulin with more hydrophobic properties. The last property limits the use of sesame proteins in certain food formulation, particularly in fluids and beverages, which indicates the need to modify the functionality of sesame proteins before it can be used in processing of imitated dairy products.

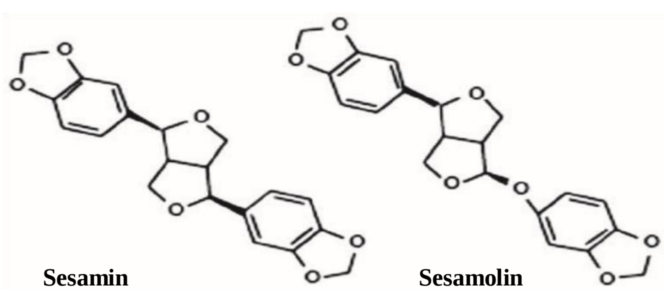


Figure 2.2 Chemical structure of bioactive compounds from sesame

Functional properties reflect the intrinsic physical attributes of the protein as influenced by interactions with food components and the processing treatments. Sesame is rich in sulfur containing amino acids and limited in lysine and contains significant amounts of oxalic (2.5%) and phytic (5%) acids (Kapadia et al., 2002).

Because oxalic acid is present in the hulls, decortication can remove most of it. Decorticated sesame seeds have the following composition: 45-63% oil, 19-31% (averaging about 25%) proteins, about 14% carbohydrates and about 3% ash. Unlike many oilseeds, sesame meal is devoid of anti-tryptic compounds. Sesame oil is very rich in polyunsaturated fat used in margarine production and cooking oils.

The valuable components of sesame seed are the oil and protein and the contents of these have been determined for several varieties of sesame which were grown in the southern and southwestern parts of the U.S. (10). Oil contents varied from 45-63% and averaged 54%. Protein contents varied from 17-32% and averaged 26%. The average protein content of the oil-free meals was 57 (Williams, 2005).

Table 2.2 Nutrient composition of sesame seeds

Nutrient	Quantity (%)
Moisture	4.0-5.3
Protein	18.3-25.4
Oil	43.3-44.3
Saturated Fatty Acids (% in oil)	14.0
Monounsaturated Fatty Acids (% in oil)	39.0
Polyunsaturated Fatty acids (% in oil)	46.0
Ash	05.2-06.2
Glucose	03.2
Fructose	02.6
Sucrose	0.2
Phytosterols	0.4

2.5 Sesame oil description

Sesame oil is highly stable and rarely turns rancid in hot climates. It is rich in unsaturated fatty acids where the fatty acids composition is 14% saturated, 39% mono-unsaturated, and 46% poly-unsaturated fatty acids (Toma and Tabekhia, 1979).

Edible oils are resulting from plants and animals. Oils from plants are termed as vegetable oil. The principal sources of vegetable oils are nearly plants, which include sunflower, soybeans, cotton seed, groundnut, rapeseed, corn, melon and sesame seed. Other sources are oil bearing perennial plants such as shear, olive, cashew coconut and palm. Sesame seed is rich in oil content with about 53% quality edible oil, 42% cake and 5% moisture seed (Akinoso, 2006).

Sesame seed oil may be used directly exclusive of refining is an ordinary salad oil, which could need small or no winterization and is one of the marginal vegetable oils. Clinical studies have shown that sesame oil consumption has too many benefits to health like minimizing the occurrence of cancer, contributes to the avoidance of degenerative processes; therefore decrease the death rate through cerebro and cardio -vascular diseases. Health benefits of sesame, such as antioxidative, hypocholesteremic, antihypertensive, immunoregulation and anticancer are mainly accredited to lignans and their glycosides (Hany *et al*., 2000).

The level of oil of the sesame seeds ranges from 41.6 to 62.5%, the average being 53.2%. Sesame contains nearly 85% unsaturated fatty acids. Seed contained 48.4% crude oil, 5.6% moisture, 20.2% crude proteins, 7.69% carbohydrate (by difference) and 9.3% crude fiber. Mineral profile (mg/100g) of seed is Calcium 415.38 ± 3.14, Phosphorus 647.25 ± 3.52, Potassium 851.35 ± 3.44, Magnesium 579.53 ± 0.42 and Sodium 122.50 ± 4.21. Chemical study of cake extract of sesame showed 4.1% ash, 51.0% fat, 19.7% sugar 6.8% protein and a total of 14.3% lignans and lignan glucosides (Nzikou *et al.*, 2009).

Fatty acid profile of sesame oil showed that major component was linoleic acid containing 41.8–45.1% of the total fatty acids, followed by stearic 32.6–24%, palmitic 8.2–7. %, oleic 4.6–5.6% and these four comprised on 96% of the total fatty acids. About 83% of total means of linoleic and oleic acids were as unsaturated fatty acids of sesame. Sesame oil fit for human consumption because high amount of unsaturated fatty acids increases the quality of the oil. Saturated fatty acids of sesame oil were palmitic and stearic acids with a range of 9.1–10.4 and 3.2– 5.9%. Phenolic compounds donate a hydrogen atom o serve up as significant antioxidants because of their donating ability in order to form stable radical intermediates. Hence, they phenolic compounds help in prevention the oxidation of different biological molecules (Were *et al*., 2006).

Applied pressure, heating temperature, heating duration, moisture content, particle size, handling and storage are factors influencing yield and quality of vegetable oil expression. The degree of influence varies with kind of oilseeds and method of oil expression. Effects of some of these parameters on yield and quality of oil expressed from sesame using expeller were studied. Moisture content, duration of heat treatment and pre heating temperature were studied (Akinoso *et al.*, 2006).

Moisture content has highest influence on sesame seed oil yield. To increase oil yield, moisture content is reduced while roasting duration and roasting temperature are increased. Temperature has less effect on increasing free fatty acid values while moisture may have a stronger effect. The hydrolysis of meadow foam triglycerides improved as moisture level of the seeds would sustain. The free fatty acid values of the oil samples show improvement with enhancing temperature and moisture. Optimum condition was achieved at seed moisture content of 4.6% wet basis, roasting time and temperature of 13.0 minute and 124.2^{0}C respectively. At these conditions, oil yield was 50.4% of substance, corresponding to 90.1% effectiveness of oil expressed. Free fatty acid was 1.1% m/m while oil impurity was 0.1% and color rating was 6.2 lovibond units (Mohammadzadeh *et al*., 2009).

Table 3.3 Amino acid and fatty acid composition in sesame seeds Source (Williams, 2005)

Nutrient Amino acid (g/16g N)	Quantity
Threonine	3.1-3.7
Valine	3.9-4.6
Cysteine + methionine	2.8-4.8
Isoleucine	4.0-4.2
Phenylalanine + tyrosine	6.4-9.6
Histidine	2.7
Tryptophan	1.3-1.5
Lysine	2.6-2.7
Argenine Fatty acid (%)	12.0

Palmitic acid (16:0)	11.7
Stearic acid (18:0)	0.52
Oleic acid (18:1)	41.4
Linoleic acid (18:2)	39.4
Linolenic acid (18:3)	0.4
Arachidic acid (20:0)	0.4
Behenic acid (22:0)	0.6

The high proportion of oil content in the seeds makes them feasible for commercial extraction. The oil was found to be cyanide free, therefore appropriate for human utilization. The protein level for enzyme isolated seeds being remarkably lower than that of solvent-isolated might be accredited to the removal of protein in the aqueous stage as is evidenced by the occurrence of substantial amounts of protein (13.9–25.7%) (Mohammed *et al*., 2007).

2.6 Extraction technology

2.6.1 n-Hexane solvet extracion

Solvent extraction is the most effective method for the recovery of oil (almost 98%),   especially with materials with low oil content, like soybeans. This method is not commonly directly used to oilseeds with high oil content, like peanut and sunflower. Such oilseeds require a prior step of pressing of the seeds and then the cake produced is extracted with a solvent (Williams, 2005).

For most oilseeds, one of the disadvantages of the extraction is that the solvent extracts some none triglycerides, which does not occur in the expelling (Williams, 2005). Another serious problem is the presence of volatile organic impurities in the final product, which can compromise the quality and go against the new profile of consumers who are seeking for natural products aiming to have a healthier diet (Michulec & Wardenki, 2005). This entails refining extracted oil using complicated process is mandatory.

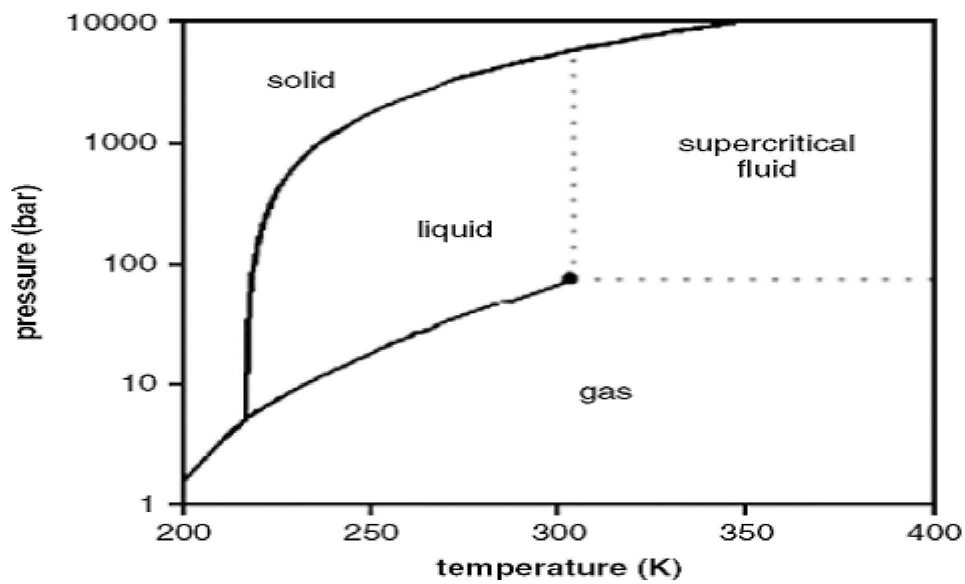
In oil production, pre-press process is designed to prepare the high oil content raw material to the solvent extraction. Mechanical pressing reduces the oil by half to two thirds of its original level facilitating solvent extraction by reducing the amount of oil to be extracted, the size of extractor equipment and also the volume of solvent used (Walkelyn & Wan, 2006).

In extraction, successive washing of oleaginous material with solvent is performed until equilibrium is reached or near equilibrium between the oil content of the solid and that of the solvent, i.e., when the solvent absorbed by the solid and solvent in the free miscella containing the same amount of oil dissolved. When this occurs, the miscella is drained and another washing takes place

The ability to extract an oil from an oilseed depends on the nature of the oil, the nature of the solvent, the temperature and the contact time between solvent and grain mass, flake thickness and conditions of pretreatment of the seeds. Attah & Ibemesi (1990) evaluated the solvent capacity and solvent effects in the extraction of oils from four native plants. The following solvents were used: petroleum benzene (60-80 ^{o} C), cyclohexane, isopropyl ether, ethyl acetate, tetra hydro furan, propan-2-ol and acetone. The authors found that oil extraction performance of each solvent appears to be generally dependent on the nature of the oil, i.e. ethyl acetate and tetra hydro furan gave the highest oil yields in rubber their oil yields in melon gave next to lowest value. Hexane is used as a solvent to commercially extract oil from rice bran. However, hexane poses potential fire, health, and environmental hazards. Short-chain alcohols, especially ethanol and isopropanol, have been proposed as alternative extraction solvents due to their greater safety and reduced probability of regulation. Alcohols tend to extract more nonglyceride materials than hexane, due to their greater polarity. Typically, alcohol-extracted oils contain more phosphatide and unsaponifiable compounds. Another important consideration was that solvent did not affect the increase in the level of free fatty acid of extracted oils (17).

2.6.2 Supper critical extraction

The state of a substance is called supercritical fluid (SCF) when both temperature and pressure exceed the critical point values, see figure 2.6



Extractions with supercritical fluid (SCF) solvents have emerged in recent years as highly promising environmentally benign technologies for the production of natural extracts with high potency of active ingredients colors, nutraceuticals or herbal medicines industries. Supercritical carbon dioxide (SC CO2) at near desirable SCF solvent for extraction of natural products today as non-flammable, and non-polluting. It's near ideally suitable for thermally labile natural products. It is generally regarded as safe (GRAS) and yields contamination-free, tailor enhanced shelf life. The supercritical fluid extraction (SCFE) technique ensures high consistency and reliability in the quality and safety of the bioactive heatit does not alter the delicate balance of bioactivity of natural molecules. Mukhopadhyay, 2008)

Extraction using supercritical fluid, the oil produced has very high purity; however the operating and investment cost is high.

Supercritical fluid extraction (SFE) eliminates the disadvantages of conventional solvent extraction (CSE) which leads to degradation of heat sensitive compounds and leaves traces of toxic sol-vents in the solute. This is a concern for food and medicinal extracts. By changing the operating pressure and/or temperature, the low solubility of oil in supercritical fluids (SCFs) can be in-creased and also improves the extraction rates. Therefore, new functional and/or nutritional characteristics for use in creating new formulated foods can be improved using SFE. Extraction of oils from different seeds with supercritical CO2 (scCO2) is widely re-ported (Özkal et al., 2005; Roy et al., 1996).

Extraction of sesame oil from sesame seeds using supercritical CO2 was carried out. The effect of operating parameters such as pressure, temperature, and supercritical CO2 flow rate and particle size on extraction yield were investigated. An increase in the pressure and the supercritical CO2 flow rate improved the extraction yield and also shortened the extraction time. The extraction yield increased as the particle size decreased depending on decreasing intra particle diffusion resistance.

Sesame oil has a mild odor and a pleasant taste and, as such, is a natural salad oil requiring little or no winterization. It is used as a cooking oil, in shortening and margarine, as a soap fat, in pharmaceuticals and as a synergist for insecticides. Sesame oil is very popular as cooking oil in many countries, and more expensive than other vegetable oils (Hai and Wang, 2006; Budowsk and Markely, 1951). An analysis of the operating cost of SFE indicated that use both of CO2 and entrainer is much less expensive than CSE (Salgınet al., 2004).

The investigated conditions which would give maximum oil yield from raw sesame seeds by scCO2 extraction. They examined the effect of three parameters including pressure, temperature and co-solvent concentration on extraction yield and also used Response Surface Methodology to analyze the experimental data. They reported that the presence of 10 vol.% ethanol gave maximum yield of 89.40% at 276 bar and 70 ^{o}C relative to yield obtained by Soxhlet extraction using hexane (Odabas and Balaban 2002).

The mathematical modeling of experimental data obtained in SFE has the objective of determining parameters for process design, such as equipment dimensions, solvent flow rate, particle size, and the characterization of SFE to enable the prediction of the viability of SFE processes on an industrial scale, through the simulation of overall extraction curves (Machmudah et al., 2006). The distribution of the natural materials within the solid structure affects the selection of models. Extractable materials easily accessible on the surface of the solid matrix, adsorbed on the outer surface, located within pores, or probably distributed within plant cell homogeneously. There are many mathematical models used for extraction of solutes from natural matrices in the literature to correlate over-all extraction curves

(Hortaçsu, 2000). The authors are aware, there are no investigation reported in the literature mathematical modeling of sesame oil extraction from raw seeds.

2.6.3 Mechanical press extraction

The mechanical expression of oil from oilseeds is the most common method used for the removal of oil from oil-bearing materials. This method is economical for small and medium scale oil processors in the developing countries (Singh et al.1984). Although the use of a hydraulic press during expression results in lower oil removal efficiency, it is preferred to the screw press by small scale producers because of lower initial and running costs. The amount of oil expressed using the hydraulic press is affected by the pre-pressing and pressing conditions such as particle size, moisture content, heating temperature, heating time, applied pressure, and pressing time (Khan and Hanna 1983). Steinbock (1948) explained, that with the exception of very small sized seeds like sesame, reduction of oilseeds to flakes by rolling is essential with the size and hardness of the seeds determining the number of stages for the flaking operation. Heating is essential because it completes the breaking down of oil cell walls, lowers the viscosity of the oil to be expelled, coagulates the protein in the meal, and adjusts the moisture content of the meal to the optimum level for pressing (Ward 1976). Oil yield has been shown by several researchers to be affected by the temperature and duration of heating (Ajibola et al. 1990; Fasina and Ajibola 1989; Sivakumaran et al. 1985; Singhet al. 1984).

High heating temperatures and long heating times may have negative effects on the quality of expressed oil and cake residue (Anjou 1972; Ohlson 1976). It is therefore important to identify the optimal heat treatment required for processing sesame seed.

The moisture content of oilseeds before pressing affects the yield and quality of oil expressed. Cloudy oil is obtained from seeds with high moisture content. Therefore, moisture content adjustment of the seeds is necessary before pressing. Vaughan (1970) explained that the effect of moisture content on expression efficiency can be related to mucilage development in the outer epidermal cells of the seeds. The mucilage coating can impede the flow of oil from the cotyledon tissue. High oil yields ranging between 37.0 and 39.6% were obtained from samples of conophor nut with post-heating moisture content between 8 and 10% (dry basis) (Fasina and Ajibola 1989), while maximum oil yields of about40% were obtained when the moisture contents of melon seed samples were reduced by about 5% (dry basis) (Ajibola et al.1990). Singh et al. (1984) observed that the moisture content of sunflower seed was the most significant factor affecting the expression of oil from the seed.

An important parameter related to the pressing efficiency is the determination of residual oil in the cake. High pressures can lead to cakes with less than 10% of oil content, which leads to higher crude oil production. A reduction in speed of rotation of the shaft, for example, can reduce the oil yield, increasing the oil content in the cake and solids in the oil (Jariene et al., 2008).

After pressing, the crude oil must be purified. A very common method of purification, especially for small producers, is to allow the oil to stand undisturbed for a few days, removing the upper layer (purified oil) and discard the bottom layer, consisting of fine pulp, water and resins. If this step is not enough, one can filter the oil using a press filter or using a centrifuge, but both methods are costly (Bachmann, 2004).

In addition to obtaining the oil, mechanical expelling produces a very important by-product named cake or meal. Some oilseeds cakes have high nutritional value and can be used as human food. Some are not suitable as food, but serve to complement the diet of chickens, pigs and cattle. It is important to emphasize the need for proper storage of seeds and cakes. They must be protected against the action of moisture, rodents and insects. High moisture content will generate mold, which can alter the taste of cakes, being rejected by the animals. Another problem is the development of mycotoxins such as aflatoxin, which in high concentrations can be poisonous to humans and animals. (Bachmann, 2004).

2.7 Factors that Affect solvent extraction process

There such parameters that affect the performance of the solvent extractor apparatus. These some parameters are outlined below.

2.7.1 Solvent Type

The choice of solvent takes into account a number of factors, notably, solvent extraction capacity, effects of solvent on oil properties, process safety, solvent volatility and stability, and economic considerations (Attah & Ibemesi, 1990).

In 1947, it was published a study evaluating the various types of solvents such as benzene, aviation gasoline, methanol, ethanol, isopropanol, carbon disulfide, diethyl ether, ethylene dichloride, carbon tetra chloride, trichloroethylene and the various petroleum naphtha. However, in 1947, the most commonly used solvents in the United States were light paraffinic petroleum fractions, such as the hexanes, heptane and pentanes. The hexane was finally chosen because of its ease to evaporate and left no residual hateful odors or tastes. There are two types of hexane: normal, also known as n-hexane; and commercial, named extraction-grade hexane. The n-hexane is pure and boils at 69 ^{o}C, while the extraction-grade hexane is not pure. So, the boiling temperature could be slight higher or lower than 69 ^{o}C.

2.7.2 Extractor Temperature

As the temperature of the miscella increases, its rate of diffusivity through the cell walls of the oleaginous material increases. As the prepared oleaginous material/ oil contained seed/ enters the extractor at approximately 60 ^{o}C, and both the oil and meal fractions are heated in excess of 100 ^{o}C in subsequent process steps, there is no extra thermal energy required for operating the extractor at a warm temperature. As a result, optimizing extraction results requires operating the extractor as warm as possible.

There is an upper limit for the extractor operating temperature. The solvent must remain safely in a liquid state. As the boiling range of commercial hexane is typically 64–69 ^{o}C at sea level, the maximum possible temperature to prevent boiling is 63 ^{o}C. operating on the edge of the boiling range could cause rapid evaporation during an upset condition. Rapid evaporation can cause pressurization of the extractor, leading to excessive solvent loss, a safety hazard. Therefore, most processors operate the extractor at 60 ^{o}C to provide several degrees of safety margin below the lower end of the solvent boiling range.

If the prepared material temperature is too low, or heat loss in the extractor is too high, then it may not be possible to achieve an extractor temperature of 60 ^{o}C. The materails should be heating to the temperature close to extraction temperature. It is necessary to ensure additional contact time between solvenet the soil seed flakes is provided to achieve desired results. The size of the extractor is proporational to contact. However, by insulating the conveying system and extractor, the heat loss can be prevented and operation can be maintained at 60 ^{o}C. This helps to reduce the capital cost of the extractor, avoiding oversizing the extractor to compensate for low operating temperature.

2.7.3 Extraction Time

The total time that the oleaginous material spends in the extractor is residence time. Residence time can be subdivided into wash time and drain time. Wash time is the time the oleaginous material spends under the washing nozzles of the extractor, and drain time is the time the oleaginous material spends draining prior to discharge.

Wash time can be further subdivided into contact time and dormant time. Contact time is the time a particle of oleaginous material spends in the washing zone of the extractor where the particle is in contact with miscella. Extraction only takes place during contact time. Dormant time is the time a particle of oleaginous material spends in the washing zone of the extractor where it is not in contact with miscella. The ratio of contact time to dormant time varies with extractor design.

Extractors with deep material bed depth and small bed surface area are generally operated by immersing the oleaginous materials in miscella. This is accomplished by providing a sufficient miscella flow rate per unit of material bed surface area to ensure that miscella fills all of the voids around the oleaginous material particles as it passes down through the material bed. This type of extractor operation provides a very high ratio of contact time to dormant time in the washing zone.

Extractors with shallow bed depth and large bed surface area are generally operated by percolating the miscella down through the oleaginous materials. With most oleaginous materials, the oleaginous material particles occupy 40% to 50% of the material bed and 50% to 60% of the material bed is composed of void spaces between the particles. In percolation, the oleaginous material particles are surrounded by both solvent vapor and miscella as the miscella rains downward.

2.7.4 Flakes Size (particle size)

Various oleaginous materials are prepared for extraction using different process steps, but with virtually all oleaginous materials, one process step is flaking. The principle purpose of flaking is to reduce the thickness of the oleaginous material to reduce the distance and number of cell walls that miscella needs to diffuse through to reach the oil bodies. Note that for soybeans, a typical 0.38-mm-thick flake is approximately 20 cells thick. By reducing the particle thickness, the time required for miscella within the cellular structure of the oleaginous material to reach equilibrium with the miscella surrounding the oleaginous material is reduced. By reducing particle thickness, desired results can be achieved with less contact time. If all other extraction parameters remain constant, reduced particle thickness will allow a smaller extractor to be used.

Reducing the particle thickness represents additional cost. For example, on soybeans, reducing particle thickness from 0.38 mm down to 0.30 mm will increase flaking mill electricity requirements by 1 to 2 kwh/ton of soybeans processed. As this is a significant ongoing operating expense, it is not economically feasible to undersize an extractor and reduce particle thickness. Conversely, by increasing particle thickness, desired results will demand more contact time. An extractor can be oversized to obtain desired results with increased particle thickness, thereby reducing ongoing operating cost.

For all oleaginous materials, the economic balance between the initial cost of the extractor and the ongoing electricity costs required for flaking can be analyzed and the optimum particle thickness can be determined.

2.8 Oil refinery

Crude oils obtained by pressing and/or extraction are sometimes used directly for food and feed purposes. In most cases, however, the crude oils are refined Crude oil refining entails the removal of gums or crude lecithin's and that of free fatty acids (FFA) from the oil to get a neutral taste of the edible oil while maintaining the nutritional value and ensuring the quality and stability of the product.

2.8.1 Degumming: chemical and physical refining

Degumming is the first step of refining and involves the removal of the gums/crude lecithins from the oil. To that effect, the crude oil is treated with water, enzymes or food grade acid at elevated temperatures. The hydrated gums are removed at the end of this step or after neutralization. Gums are a raw material for the production of lecithin's.

2.8.2 Neutralization: chemical refining

FFAs are responsible for oil acidity. Chemical refining is the traditional method of oil refining and involves a neutralization step of these FFA's in the crude oil. During neutralization, the oil is treated with a food grade alkali solution (caustic soda) that reacts with the FFA to form soap stock. The soap stock -together with the precipitated gums, if still present- is separated from the oil by centrifugation. Typically, soap stocks contain 40% water and 60% fatty matter (FFA, triglycerides). In facilities that both crush oilseeds and refine the seed oils (integrated crushing and refining), the soap and gums can be added back to the meal or expellers at inclusion levels of around 1.5%. Soap stock can also be sold to the market as feed material under the denomination "soap stock" or can be split by means of an acid into acid oils.

The production of gums and soaps stock in integrated crushing refining is a process of continuously removing the gums and free fatty acids from the oils and continuously adding these as gums or soap stock to the meal or expellers. The components in the soap stock are part of the natural composition of seeds or beans. This means that only natural components separated from the seeds and beans are returned back into the crushing process. Whether integrated crushing refining plants add soap stocks back to the meal or expellers is determined by the design of the facility. It is not subject to daily management decisions. In their meeting on 17 and 18 January 2013, the Standing Committee on the Food Chain and Animal Health, section Animal Nutrition confirmed the feed material status of meals and expellers to which soap stocks have been added in integrated crushing and refining plants.

2.8.3 Bleaching: chemical and physical refining

The purpose of bleaching (or decolorizing) is to reduce the levels of pigments such as carotenoids and chlorophyll, but this treatment also further removes residues of phosphatides, soaps, traces of metals, oxidation products, and proteins. These trace components interfere with further processing. They reduce the quality of the final product and are removed by adsorption with activated clay or silica. In integrated crushing / refining plants the used bleaching earth may be brought back into the meal. Bleaching earth originating from stand-alone refining plants and / or hardening plants, the latter which can contain nickel is excluded from recycling into feed and is disposed of outside the feed chain. If heavy polycyclic aromatic hydrocarbons are present in crude oil, activated carbon shall be used for their removal. The bleaching clay containing activated carbon is disposed of outside the feed sector.

2.8.4 Winterization

Optionally both chemical and physical refining during winterization waxes are crystallized and removed in a filtering process to avoid clouding of the liquid fraction at cooler temperatures. The filter cake that remains after the filtering process consists of oil, waxes and filter aid. The filter cake can be recycled to the toaster and added to the meal (in an integrated crushing/refining plant) or sold as such as a feed material (refining standalone). The term winterization was originally applied decades ago when cottonseed oil was subjected to winter temperatures to accomplish this process. Winterization processes using temperature to control crystallization are carried out on sunflower and maize oil. This process is also referred to as dewaxing.

2.9 Minor Components of Sesame Oil

Many of the special properties of sesame oil are due to the presence of 0.4-1.1% sesamin, 0.3-0.6% sesamolin and traces of sesamol in the oil (4, 18). As shown in Figure 2.3. Sesamin and sesamolin have the same 2,7-dioxabicyclo- (3,3,0)-octane nucleus and both have two 3,4-methylene dioxyphenyt substituents. In sesamin these are attached directly to the nucleus, while in sesamolin one is attached directly and the other through a connecting oxygen atom. Sesamol is the free 3, 4-methylenedioxyphenol. Thus sesa- molin is an acetat-type derivative of sesamot. The absolute configurations of sesamin and two other sterioisomers are known (17).

The superior oxidative stability of sesame oil relative to other oils is largely due to sesamol, which is usually present in traces but may be released from sesamolin by hydrogena- tion, by acid or acid bleaching earths or other conditions of processing or storage. Sesamol may also be removed by some bleaching earths or by deodorization. The effectiveness of sesamol as an antioxidant on the keeping quality of lard has been reported (20).

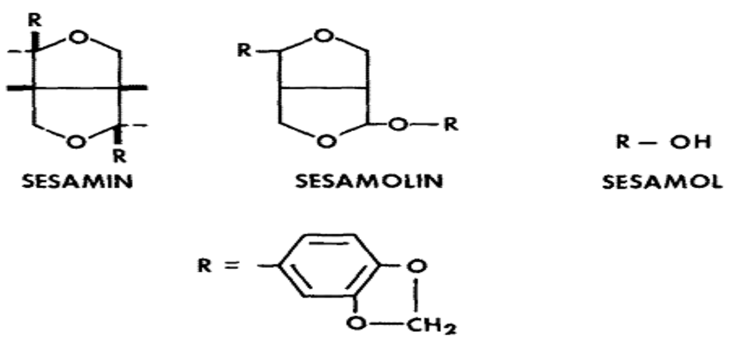


Figure 2.3 Structures of Sesamin, Sesamolin and Sesamol.

It was more effective than butylated hydroxytotuene (BHT), butylated hydroxyanisole (BHA) and several other antioxi- dants and was surpassed only by propyl gallate. In a more recent study of antioxidants (21), the induction period for autoxidation of methyl octadecadienoates, in the presence of 0.01% of various antioxidants, was estimated by measuring the time for pickup of a certain weight of oxygen. In the natural cis, cislinoleate, sesamol was more effective than BHT or BHA and was surpassed only by propyl gallate. Sesamol was not effective in the Trans, trans-ester. In the conjugated ester, BHT, BHA and sesamol were equally effective and were more effective than propyl gallate.

Sesame oil may be readily detected in mixtures with other oils by tests for sesamol, sesamolin and sesamin. For this reason it has been used in some countries as a marker in margarine and other hydrogenated oils. The analyses by the Villavecchia and other color tests, by UV spectrometry and by thin layer chromatography have been thoroughly reviewed (16).

The sterols and triterpenes in sesame oil have been separated and identified using thin layer and gas liquid chromatography (18). The 0.19% of sterols are the usual plant sterols and are, in order of decreasing concentration, fl-sitosterol, campesterol and stigmasterol. The 0.03% of triterpenes includes at least six compounds, three of which were identified as cycloartenol, 24-methylenecycloartanol and c~-amyrin (23).

2.10 Benefits of sesame seed processing

Sesame seeds are widely considered to be healthful foods. They are high in energy but contain many health benefiting nutrients, minerals, antioxidants and vitamins that are essential for wellness and have positive effects on human health (www.nutrition-and-you, Borchani et al. 2010). They are very good sources of B-complex vitamins such as niacin, folic acid, thiamin (vitamin B1), pyridoxine (vitamin B6) and riboflavin. The seeds are incredibly rich sources of many essential minerals which include Calcium, iron, manganese, zinc, magnesium, selenium and copper in concentrated amounts. Many of these minerals have vital role in bone mineralization, red blood cell production, enzyme synthesis, hormone production as well as regulation of cardiac and skeletal muscle activities (www.nutrition-and-you). The seeds are especially rich in mono-unsaturated fatty acid oleic acid which comprise up to 50% fatty acids in them. Oleic acid helps to lower LDL or "bad cholesterol" and increase HDL or "good cholesterol" in the blood. The seeds are also very good source of dietary proteins with fine quality amino acids that are essential for growth, especially in children. In addition, sesame seeds contain many health benefiting compounds such as sesamol and sesaminol which are phenolic anti-oxidants and help stave off harmful free radicals from the body. As a result of the benefits of sesame, seed exportation offer highest potential for farmers due to the demand for the seed, but Sesame’s potential for commercial processing in Nigeria is also great (Nigeria’s Harvest, 2009). This is because sesame can be processed into various forms which include oil, meal, paste, confections, and bakery products.

Sesame oil has a high percentage of polyunsaturated fatty acids (omega-6-fay acids) but keeps at room temperature uniquely due to the presence of sesamol and sesamin, two naturally-occurring preservatives. The presence of high polyunsaturated fatty acids content make it possible to use sesame oil for cooking in place of other edible oils and to help reduce high blood pressure and lower the amount of medication needed to control hypertension. Sesame oil can also be used for medicinal purposes which include reduction of cholesterol levels, anti-bacterial effects, and even slowing down certain types of cancer (www.nutrition-and-you). Apart from these various constituents present in the sesame oil have anti-oxidant and anti-depressant properties. Sesame oil enhances many dishes including salads, pasta, sauces and other hot and cold food as well as sweet dishes, the manufacture of cosmetics such as soap, hair conditioners, and bath oil. The cake obtained after expression can be used in the preparation of livestock feed. The oil has a neutral taste and significantly improves nutritional value (www.nutrition-and-you).

2.11 Use of byproduct

All oilseed cakes can be used as livestock feed ingredients with suitable pre-treatments where required. Often, when low-temperatures are used for oil extraction, the cake can be incorporated into food. However, all oil extraction businesses need to identify markets for their by-products to maximize income. Quality assurance the main quality checks concern raw materials, processing conditions, product quality (conformity to the standards applicable in the market into which the oil is sold) and packaging and storage conditions. Raw materials should be checked to ensure that there is no mould growth, and that they are correctly dried, cleaned and sorted so no other seeds or contaminants are present. During processing, the temperature and time of conditioning, the moisture content of the raw material, and the yield of oil should be routinely checked.

Chapter Three

Material and Methods

3.1 Materials needed to experiments

Sesame seed sourced from Hummera harvested in 2016, which bought at Merkato Addis Ababa Ethiopia.

Solvent n-Hexane were used for extraction process, which bought from Neway Chemicals sale and distruster. Activated carbon used for the purpose of for color absorbent, which bought from Neway Chemicals sale and distruster. Caustic soda, which bought at Neway Chemical distruster.

Addis Ababa Instate of Technology provide such equipment Soxhlat extractor were used for extraction, Beaker to measure chemical and water, jar, water bath, Thermostatic water bath. Balance simple electronic mass balance from AAiT. Oven which owned by AAiT works about 120 ^{o}c. Measuring cylinder, etc.

Sample analysis

20g, 30g, and 40g of the cleaned sample was weighed and dried in an oven at 100°C and the weight was measured every 2hrs. The procedure was repeated until a constant weight was obtained. The percentage moisture in the kernel was calculated using the following:

$$\text{moisture content} = \frac{(w1-w2)*100}{w2*100} \quad (3.1)$$

Where: W1 = Original weight of the sample before drying;

W2 = Weight of the sample after drying.

3.2 Methods

Extraction using solvent has several advantages. It give higher yield and less turbid oil than mechanical extraction, and relative low operating cost compared with supercritical fluid extraction.

The laboratory procedure that can be followed for the extraction sesame oil using soxhlate extractor was as follows.

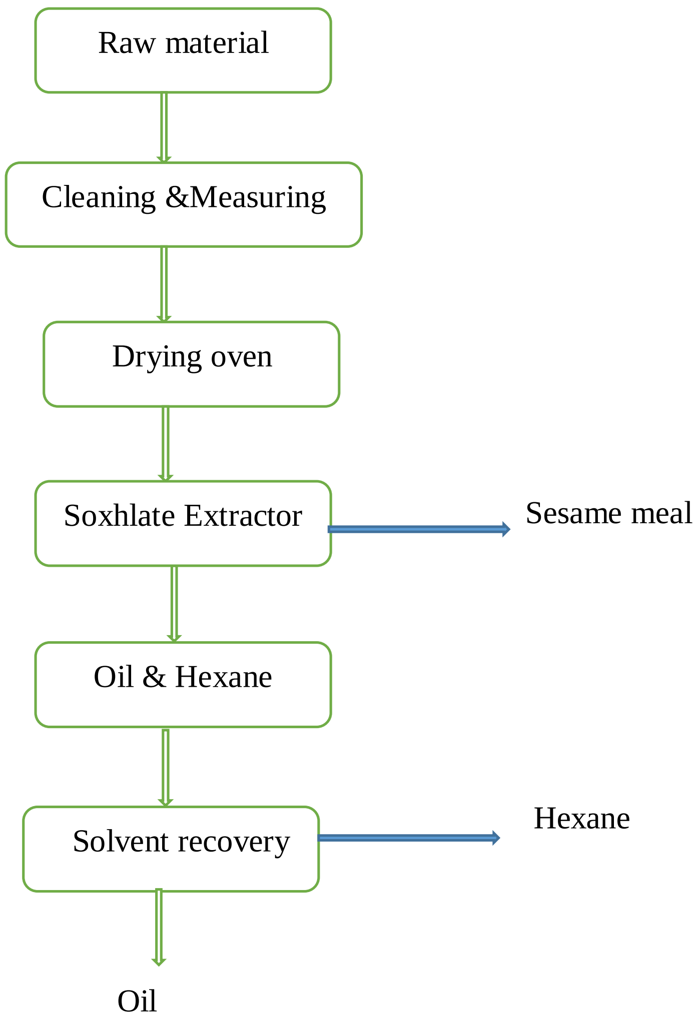


Figure 3.1 process flow sheet

The raw material was bought from Merkato Addis ababa Ethiopia, after had the raw material further cleaning sesame seed to remove foreign matter and measuring using electronic balance. Drying seed using oven to have 6-10 % moisture content and put the sample put in desiccator. Crushing the seed to lower the size Oil extraction using soxhlate and cold press machine.

3.2.1 Soxhlet extraction

The experimental procedure was done in AAIT Chemical Engineering Laboratory. Each of the experimental run was designed with the aid of Design Expert using the central composite design of the response surface methodology. The sesame seed (20 g per sample) was packed inside a thimble bag (see Figure 3) and placed inside the thimble chamber of the 100 ml Soxhlet extractor shown in Figure 4. The extractor itself was placed inside a thermostatic water bath (shown in Figure 5). A round bottom flask containing n-hexane as well as a condenser was fixed to the extractor. The flask was heated to a temperature above the boiling point of the solvent as indicated in the design matrix. The solvent then vaporized and passed through the prepared seed to remove its oil. The mixture obtained (solvent and oil) moved directly into the round bottom flask. The process was allowed to continue for the specified time, as obtained from the experimental design. Thereafter, the oil extracted was recovered by distilling the solvent using the same apparatus.

Figure 3.2 Thimble bag

Figure 3.3 Soxhlet Extractor

Figure 3.4 Thermostatic with water bath

Figure 3.5 laboratory setup of Soxhlet extraction

Determination of the yield of sesame oil extracted

At the end of each experiment, the yield of the oil was obtained using the following relationship.

$$\text{oil yeild} = \left(\frac{\text{w3} - \text{w1}}{\text{w2}}\right)\text{x100}$$

Where

W1is the weight of flask

W2 is the weight of sample

W3is the weight of flask and extracted oil

3.3 Characterization of sesame oil

3.3.1 Determination iodine value

Add 40ml of cyclohexane and 40ml of acetic acid glacial. Weight the required amount of 0.3646 gm. of oil

Add 15 ml of mixture of cyclohexane and acetic acid glacial and Add 25 ml of weji's solution keep 1 hr. of reaction to dark place. Add 20 ml of potassium iodide to the solution. Add 150 ml of distilled water and titrate by using sodium thiosulphate.

$$\text{IV} = \frac{1.269(\text{Vblank} - \text{Vsamle})}{\text{mass of oil}}$$

V blank= 40 ml

V sample=10 ml

Mass of oil = 0.4636 gm.

$$\text{IV} = \frac{1.269(40 - 10)}{0.4636}$$

$$\text{IV} = 82.11\text{ml/gm}$$

3.3.2 Determination Acid value

Standard alcoholic potassium hydroxide solution (0.1 N) was prepared by dissolving KOH (pellet) with 95% ethanol. The solution was filtered and stored in brown bottle for five days. A phenolphthalein 1g per 100ml of 95% v/v ethanol was used us an indicator. Furthermore, a mixiture of 1 to 1 ratio (v/v) 95% ethanol and diethyl ether was prepared by mixing 250 ml diethyl ether and 250 ml of ethanol.

A weighted quantity of the oil sample was dissolved in 25 ml of 1 to 1 mixture of ethanol and diethyl ether. The solution was titrated with 0.1 N KOH solution in presence of 5 drops of phenolphthalein as indicator until the end point (colorless to pink) is recognized. The volume of 0.1 N ethanolic KOH (v) for the sample titration was noted. The total acidity (Acid number) in mg KOH/ gm was calculated using the following equation.

$$\text{Acid value} = \frac{\text{V X N X 56.1}}{\text{m}}$$

Where V is the volume expressed in milliliter f 0.1 N solution of ethanolic KOH

m is mass in gram of the test portion

N is concentration of ethanolic KOH

Acid value = 0.42 mg KOH/g

3.3.3 Gas Chromatography mass spectroscopy (GC/MS) result analysis

The Free fatty Acid value was determined from GC/MS analysis using the area covered under the peak graph as follow.

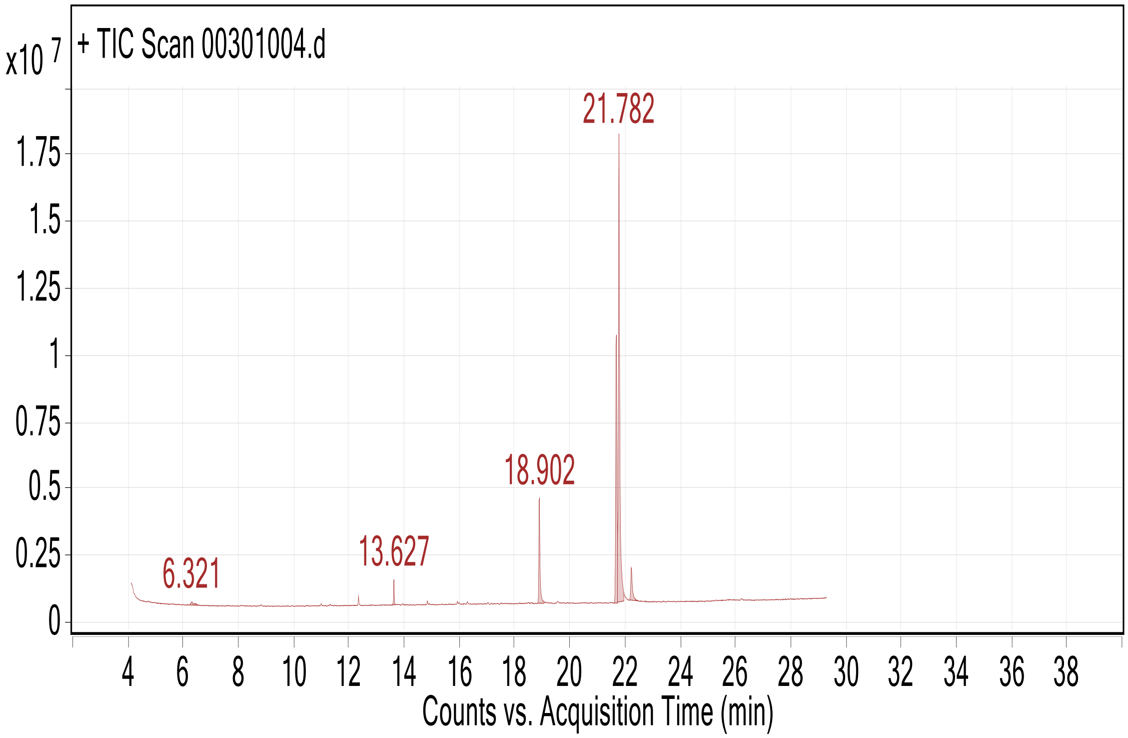


Figure 3.6 graph count vs. acquisition time

Table 3.1 Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
3	18.835	18.902	19.077	3930321.6	12035238.93	10.7914
4	21.607	21.688	21.728	10036843.67	30225554.84	27.1019
5	21.728	21.782	21.957	17526154.9	64191073.12	57.5572
6	22.167	22.226	22.468	1,211,052.07	5073897.81	4.5495

From table 3.1 the composition of sesame oil obtained from GC/MS analysis were found 10.79% palmic acid methyl ester, 57.55% linoleic acid, 27.101 oleic acid and 4.55% stearic acid methyl ester.

3.3.4 Determination of density

Measuring of the density of oil was carried out using picnometer that 50cm ^{3}

$$\text{density} = \frac{\text{mass of oil}}{\text{volume of oil}}$$

$$\text{density} = \frac{45.6 \text{ gm}}{50 \text{ cm3}}$$

$$\text{density} = 0.912 \text{ gm/cm3}$$

3.4 study design

After the sesame seed was obtained and prepared, the experiments to be carried out were designed with the aid of Design Expert 7.0.0 (Stat-Ease, 2005) using the central composite design of the response surface methodology. In designing the experiments, the factors considered were the moisture of the seed, the temperature of extraction and the time used for the extraction while the response of each of the experiments was chosen to be the yield of the oil obtained from the seed. The maximum and the minimum levels used for the factors considered in this work are as given in Table 3.2. Using the levels of the three factors given in the Table 3.2 and an alpha value of the design of 0.100, twenty (20) experiments were designed and carried out using Soxhlet extractor.

Table 3.2 soxhlate extraction Factors and their levels for central composite design

Variable	Levels	
	Minimum(-1)	Maximum(+1)
Moisture content (%)	6	10
Temperature (^{o} C)	73	83
Extraction Time (Hr.)	5	7

3.5 statistical analysis and optimization

The software design expert will be employed experimental design, data analysis, and the model building. Statistical significance of the model and model variable will determine at 5% probability level ($p<0.05$) analysis of variance (ANOVA) will be used.

After obtaining the responses (oil yield) from each of the experiments, the values (the responses) were entered into the appropriate column in Design Expert, and the response together with the factors considered were analyzed, and a quadratic and a cubic models relating the percentage oil yield to the factors considered were developed for the extraction. In order to get a model that would best fit the experimental results and take care of effects of all the factors, the developed models were modified. The modifications carried out resulted in improvements in the performances of the models. Using the quadratic models, a numerical optimization was carried out and the optimum conditions of the three factors investigated for the extraction were obtained. For the quadratic model, the goals for the factors were in a range while that of the response was “maximize”. In maximizing the response using cubic model, the goal of each of the factors was a target of moisture content 6%, extraction temperature of 83 ^{o} C and extraction time of 7 hr were found to be the optimum operating conditions, which led to optimum oil yield of 50.17 % for quadratic models.

Chapter four

Result and discussion

4.1 soxhlate extraction yield data

The experiments were carried as per the experimental design. The data generated from the experiment are given in the Table 4.1.

As shown in table 4.1, the result optaind showed that moisture content and extraction time affected the oil yield most. For instance, looking at run 13 and run 9 in which the moisture content increased from 6% to 10% oil yield was noted to decrease from 50.2 % to 47.43%. However, comparing. Run 1 with Run 13, similar variation in extraction time led to increase in oil yield from 45.83 to 50.2%. Although, comparing the extraction temperature run 14 and run 1 in this manner also oil yield incresed from 43.19 to 45.83%.

After the results of the experiments were obtained and considered, they were, first, used to develop a quadratic model equation for the extraction. The developed quadratic model equation is as shown in Equation (2) while the analysis of variance (ANOVA) of the model is given in Table 3.

$$\text{Yield of Oil} = +45.71 + 1.58 * A + 1.64 * B - 1.04 * C + 0.38 * A * B - 0.14 * A * C - 0.16 * B * C + 0.17 * A - 0.63 * C^2 \quad (4.1)$$

Table 4.1 The central composite design matrix and responses

Run	Temperature(oC) A	Time (Hr.) B	Moisture cntent (%) C	Yield of oil %
1	83.00	5.00	6.00	45.83
2	78.00	6.00	8.00	45.85
3	78.00	6.00	8.00	45.5
4	78.00	6.00	11.36	42.28
5	78.00	6.00	8.00	45.81
6	78.00	7.68	8.00	48.5
7	73.00	7.00	10.00	43.83
8	86.41	6.00	8.00	48.9
9	83.00	7.00	10.00	47.43
10	73.00	5.00	10.00	41.62
11	78.00	6.00	8.00	45.62
12	78.00	6.00	8.00	45.66
13	83.00	7.00	6.00	50.2
14	73.00	5.00	6.00	43.19
15	69.59	6.00	8.00	43.5
16	78.00	6.00	4.64	45.58
17	83.00	5.00	10.00	43.72
18	78.00	6.00	8.00	45.72
19	73.00	7.00	6.00	46.04
20	78.00	4.32	8.00	43.031

The coefficients in the model are determined using quadratic regression using ANOVA analysis software. The *coefficients* obtained is inserted in the equation. The ANOVA is used to deterime sum of squares, mean square, F-value, p-value, and other relevant parameters. The result obtained from the analysis is presented in Table 4.2.

The Model F-value is 963.18 which implies the model is significant. According to the F-value, there is only a 0.01% 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 A, B, C, AB, AC, BC, A^2, C^2 are significant model terms. According to litertaure, 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.

The "Lack of Fit F-value" of 0.51 implies the Lack of Fit is not significant relative to the pure error. There is a 78.09% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good we want the model to fit.

Std. Dev.	0.11	R-Squared	0.9986
Mean	45.39	Adj R-Squared	0.9975
C.V. %	0.24	Pred R-Squared	0.9955
PRESS	0.42	Adeq Precision	115.185

Table 4.2 ANOVA for Response Surface Reduced Quadratic Model

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	93.53	8	11.69	963.18	< 0.0001	significant
A-Temperature	34.11	1	34.11	2809.81	< 0.0001	
B-Time	36.54	1	36.54	3010.11	< 0.0001	
C-mositure content	14.79	1	14.79	1218.12	< 0.0001	
AB	1.14	1	1.14	93.92	< 0.0001	
AC	0.15	1	0.15	12.46	0.0047	
BC	0.21	1	0.21	17.40	0.0016	
A^2	0.41	1	0.41	34.09	0.0001	
C^2	5.85	1	5.85	481.93	< 0.0001	
Residual	0.13	11	0.012			
Lack of Fit	0.051	6	8.464E-003	0.51	0.7809	not significant
Pure Error	0.083	5	0.017			
Cor Total	93.66	19				

4.1.1 Effect of temperature on oil yield

The quantity of oil can be affected by temperature. From the data generated in the expereiment, the effect of temeperature on oil yieald is ploted in Figure 10. . The plot shows thatincreasing temperature raised oil yield. Based on the findings, the yield was found to be enhanced with increasing temperature. This was because of rising the temperature enhancedboth the diffusion coefficient and the solubility of the oil in to both solvents, Thus heat treatment improves the extraction sesame oil. The higher extraction temperatures the easier to break the molecule inside the seed; as a result, the yield also gets high.

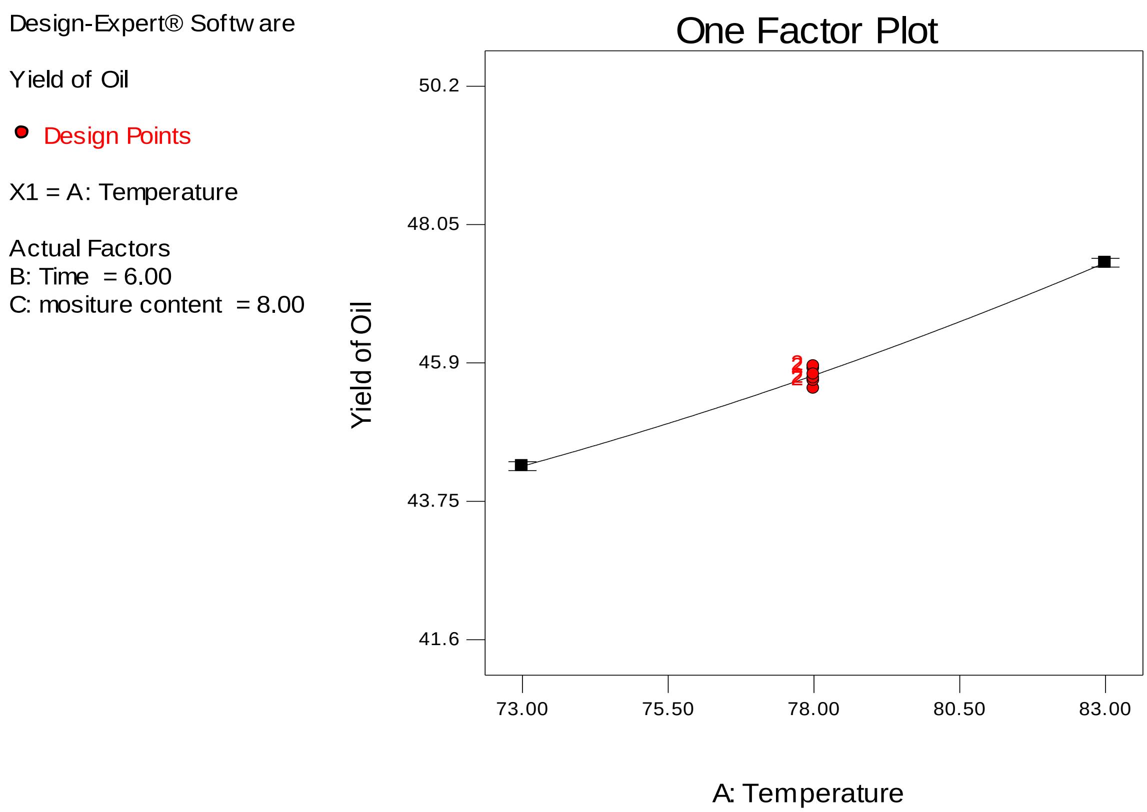


Figure 4.1 Plot of Temperature vs oil yield

4.1.2 Effects of Extraction Time on oil yield

Extraction time is one of the factors that affects the percent yield of sesame oil. . The experiemntal result shows extraction time influences greatly the percentage yield of sesame oil using n-hexane as a solvent. This is shown Figures 11. The plot shows the increase in the contact time increased the oil yield till transfer of oil from the kernel powder to the solvent attains zero. In other word, when the maximum amount of extractable oil is obtained, the oil yield level remains invariable even by extending the extraction time. As shown in Figure 4.2, extracting the oil beyond six hours is wasting time because using n-hexane as a solvent can find maximum yield at this time.

The Figure also shows the extraction rate is fast at the beginning of the extraction but gets slow gradually. The reason is that when the kernel powder is exposed to the fresh solvent, the free oil on the surface of seeds is solubilized and oil gets extracted quickly inducing a fast increase in the extraction rate. Furthermore, since the oil concentration is low in the solvent at the beginning of the extraction process, the oil diffuses quickly from the kernel to the liquid phase due to the difference in concentration (driving force) of the oil. As the time passing by, the concentration of oil increases in the solvent resulting in a decrease in the diffusion rate.

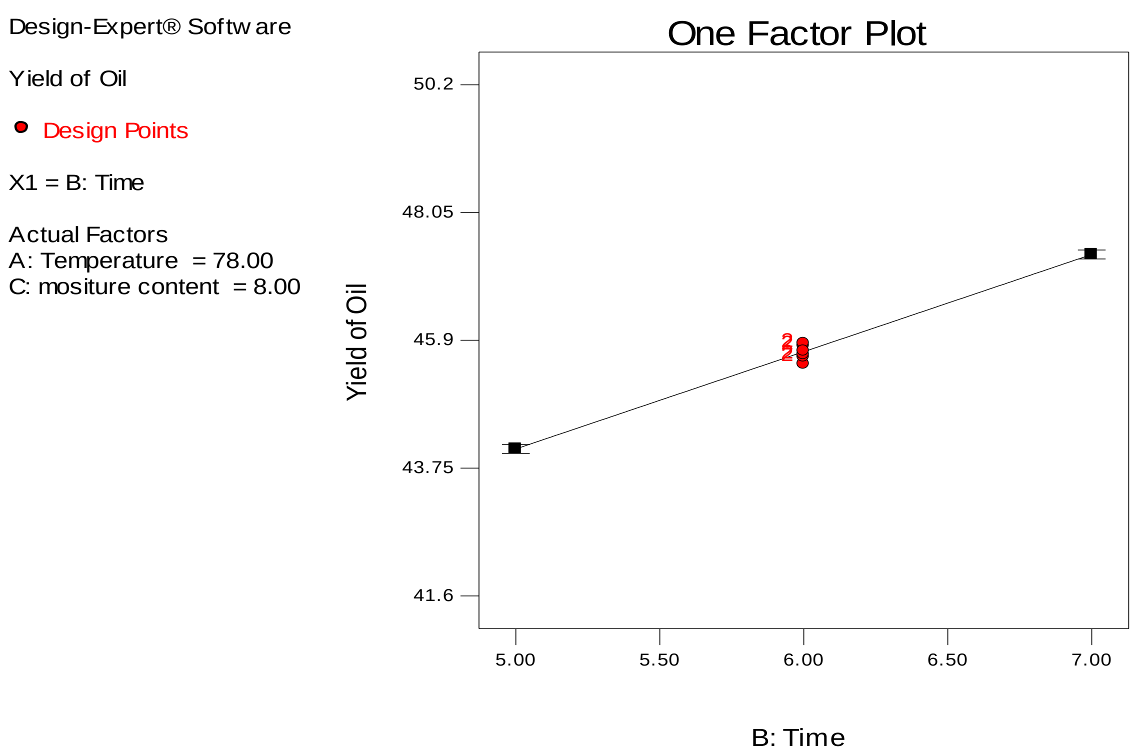


Figure 4.2 Plot of extraction time vs oil yield

4.1.3 Effects of moisture content on oil yield

The effect of moisture content is shown in Figure 4.3. The plot shows that as the moisture content decrease from 12% to 6%, the oil yield increased from 42% to 47%. In order to enhance the downstream unit operation of flaking, oleaginous materials are typically heated in the range of 90 –110 ^{o}C temperature. By heating and softening the oleaginous materials, it enables the oleaginous material to stretch and flatten in the flaking operation with a minimum of fragmented particles being created.

For oleaginous materials being prepressed prior to solvent extraction, they are typically heated in the range of 90 –110 ^{o}C temperature to decrease the viscosity of the oil and allow the prepress to expel the oil and make a good quality cake

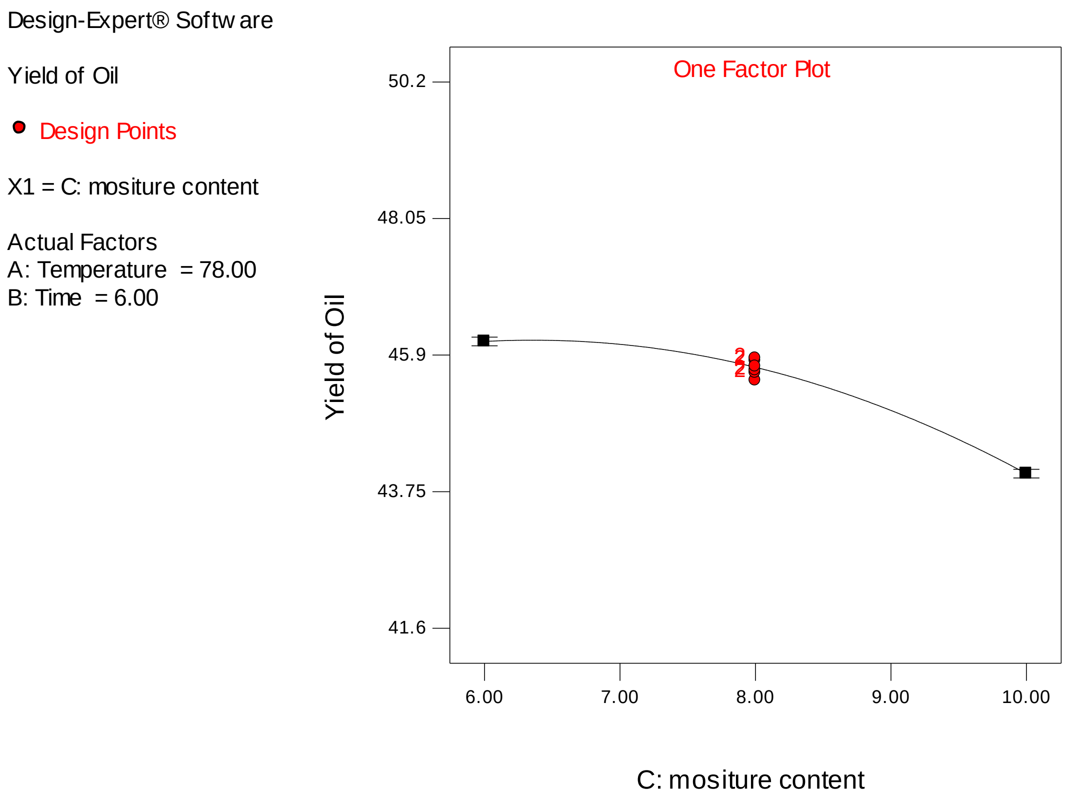


Figure 4.3 moisture content vs yield of oil

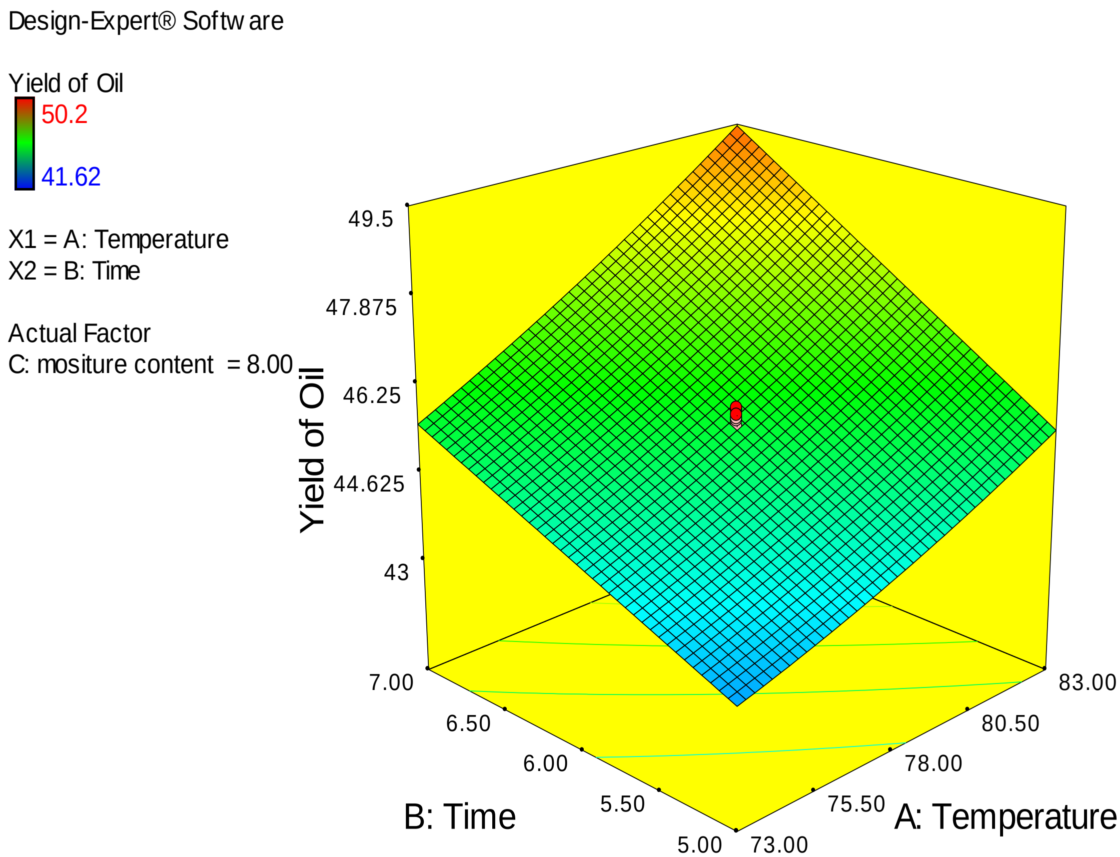


Figure 4.4 3D surface plot for effect of extraction time and extraction temperature on oil yield

As Shown in Figures 4.4, the variations of extraction time and extraction temperature with oil yield for the quadratic models. According to the figures, it was discovered that the variation of ettraction time with temperature affecte oil yield.

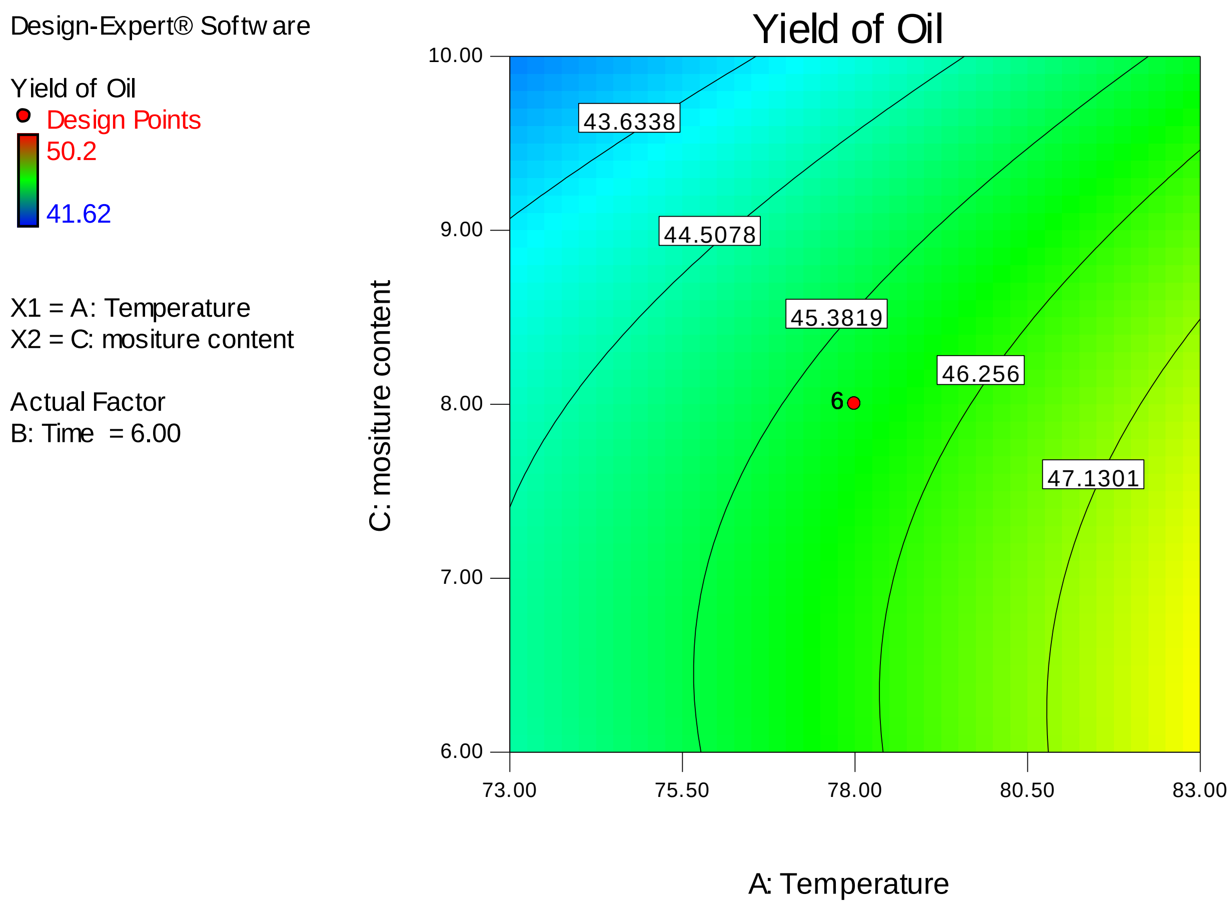


Figure 4.5 Contour plot for effect of moisture content and extraction temperature on oil yield

Looking at Figures 4.5 and 4.6, the effects of the three factors (moisture content, extraction temperature and extraction time) considered on the oil yield can be easily seen. From these figures, it can be seen that the variation of moisture content was found to significantly affect the oil yield obtained from sesame seed as the extraction time was varied. For instance, increasing the moisture content while slightly varying the extraction temperature and extraction time as shown in Figures 4.5 and 4.6 led to decrease in oil yield. That means, the oil yield was not significantly affected by the interaction of temperature and extraction time as the moisture content is increase. So, it seems that the moisture content should be controlled or reduced as much as possible to get the positive effect of temperature and extraction time.

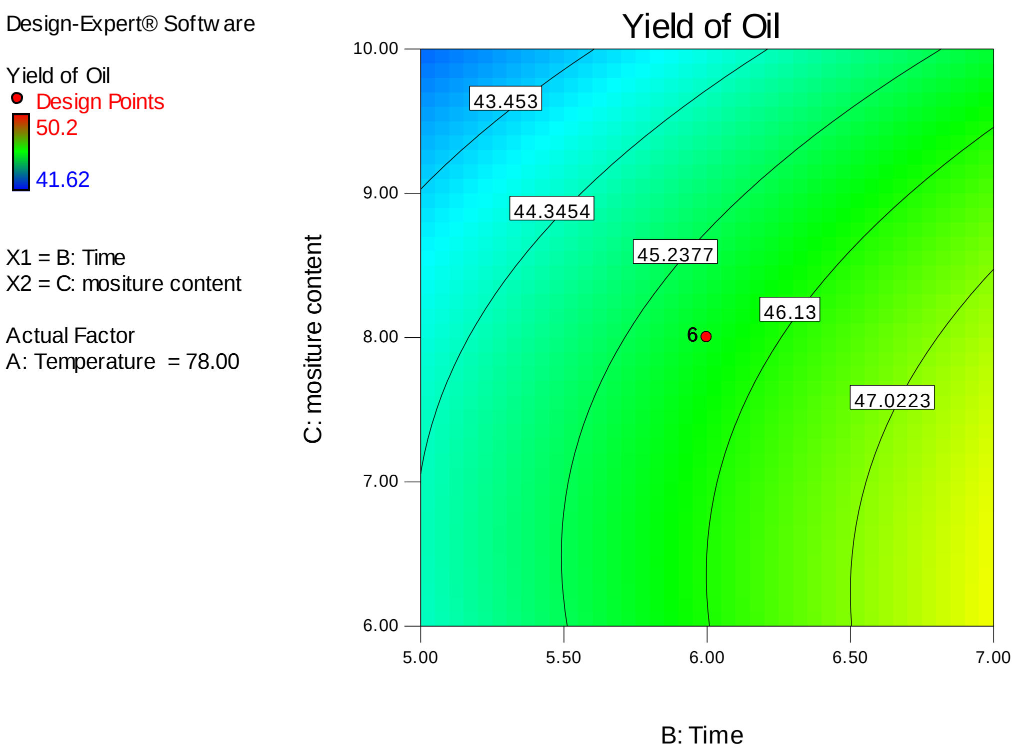


Figure 4.6 Contour plot for effect of Time and moisture content on oil yield based on quadratic model

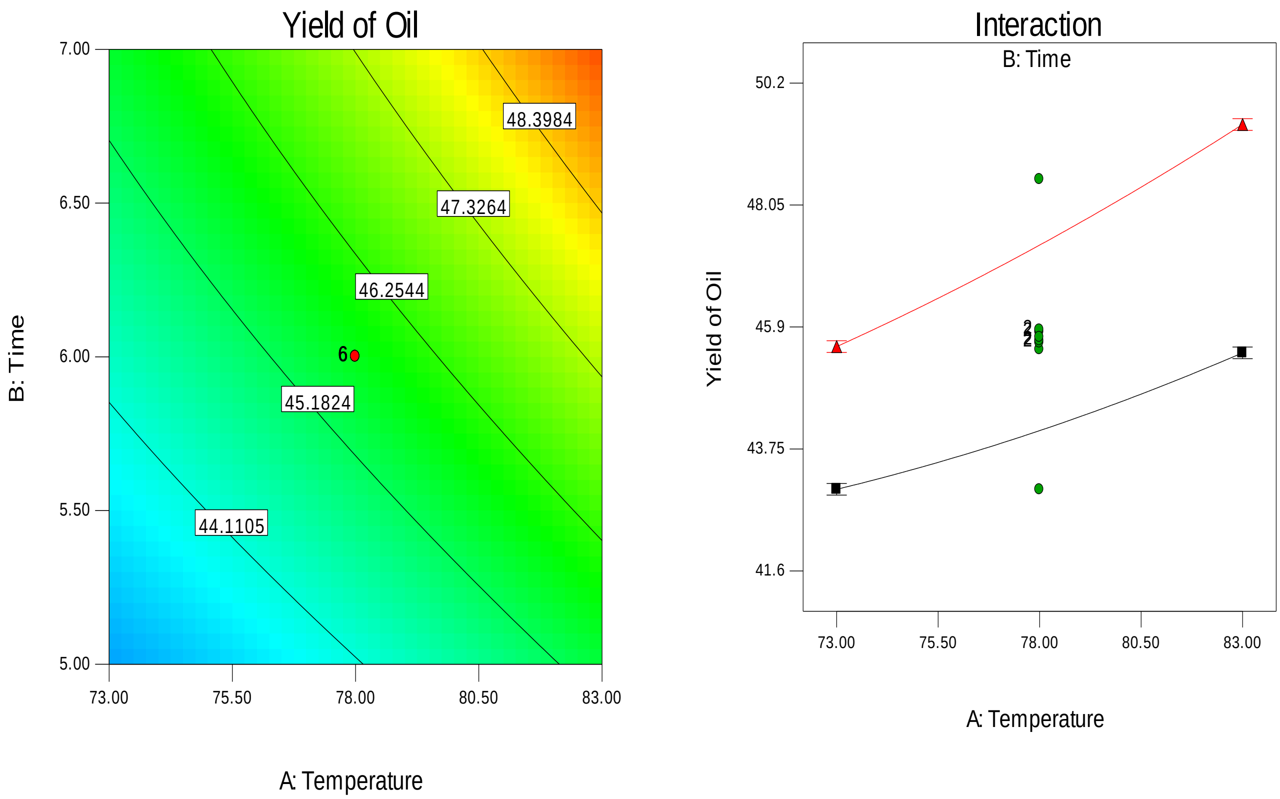


Figure 4.7 Contour plot for effect of temperature and extraction time on oil yield on quadratic model

From the numerical analysis carried out for the quadratic model developed for oil extraction from sesame seed, moisture content 6%, extraction temperature of 83 ^{o} C and extraction time of 7 hr. were found to be the optimum operating conditions, which led to optimum oil yield of 50.17 % for quadratic models. Furthermore, using the optimum parameters obtained from the response surface methodology employed carried out to perform a validation experiment, oil yield of 50.17 % was obtained. The value obtained was found to compare well with the predicted optimum yield. Therefore, the result obtained has revealed that the optimum values of the parameters given by response surface methodology with the aid of Design Expert were valid ones.

Diagnostics

The following figure 4.8. shows the relation between the actual value of the experiment and the value predicted by the model equation developed by the Design Expert software.

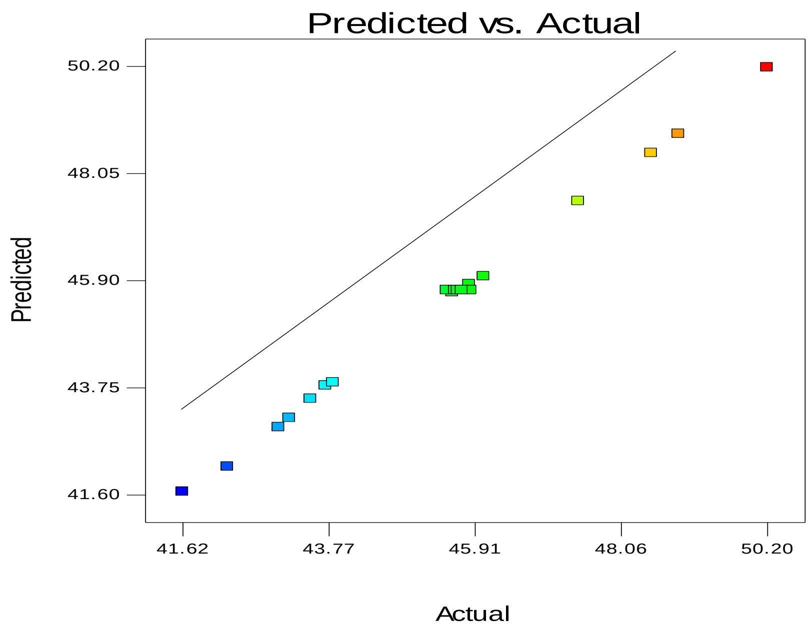


Figure 4.8 predicted vs. actual

Chapter 5

Conclusion and Recommendation

5.1 Conclusion

The sesame seed is a good source of rich in protein, minerals and oil. Sesamum indicum L. seed oil is of unsaturated type and contains mainly the fatty acids 10.79% palmic acid methyl ester, 57.55% linoleic acid, 27.101 oleic acid and 4.55% stearic acid methyl ester were found from GC/MS analysis.

The experimental investigation and modeling using surface response method shows a quadratic model is a good fit for this extraction of oil from sesame seed. The R-squared, the Adj R-squared and the Pred R-squared values of the developed quadratic model were 0.9986, 0.9975 and 0.9955, respectively. Furthermore, the results of the optimization using quadratic model carried out revealed that the optimum values of the seed moisture content, the extraction temperature and the extraction time were 6.%, 83 ^{o}C and 7 hr. respectively. The optimum valued of parameters as validated experimentally. The result showed that there was a good agreement between the optimum oil yields predicted by the statistical method employed were 55% for modified quadratic, while that obtained from the validation experiment was found to be 50.17%. This has demonstrated that the optimum values obtained from the Design Expert were valid ones. Thus, it can be concluded that response surface methodology has been successfully applied to optimize sesame seed oil yield.

5.2 Recommendation

The following recommendations are made on the subject area of sesame seed extraction:

- Here in the experimental work, the solvent to solid ratio and seed particle size has not been studied. This is due to. So, further investigation needs to analize the oil yield
- Further plant design and economics analysis will also needs to be work for feature investigation on the continuation of thesis paper.

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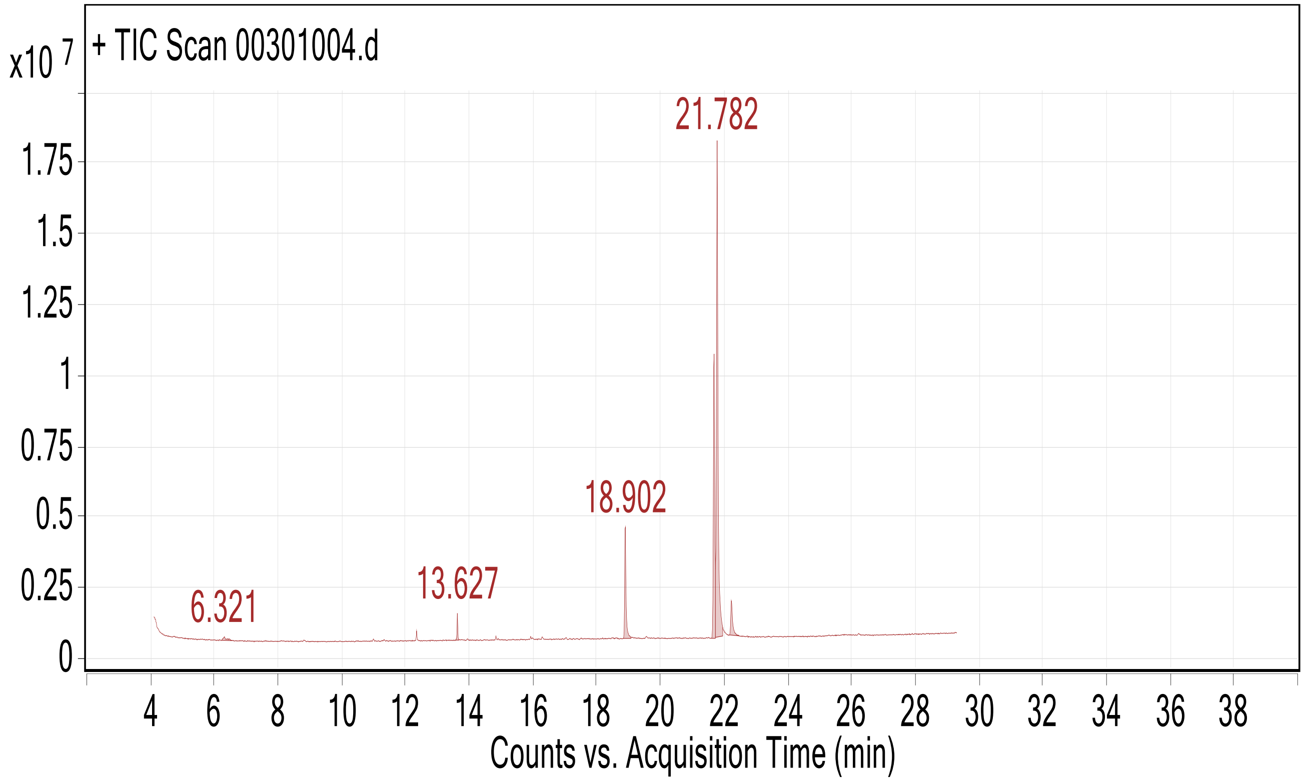
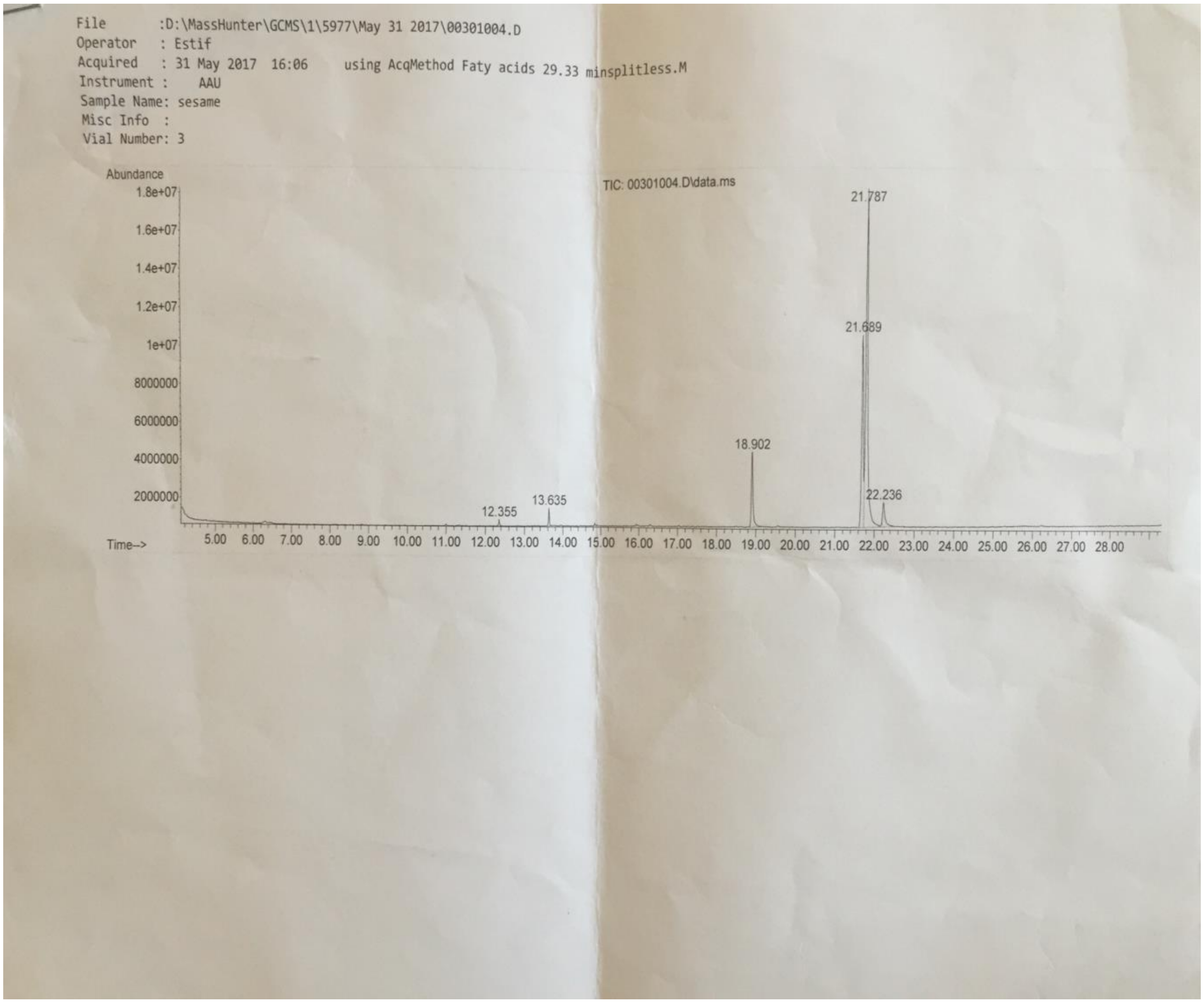
Appendix

Appendix A Integration Peak Area from Gas chromatography spectrometer



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6.226	6.321	6.547	127773.83	978567.94	1.52
2	13.574	13.627	13.681	946990.31	1540415.18	2.4
3	18.835	18.902	19.077	3930321.6	12035238.93	18.75
4	21.607	21.688	21.728	10036843.67	30225554.84	47.09
5	21.728	21.782	21.957	17526154.9	64191073.12	100
6	22.167	22.226	22.468	1211052.07	5073897.81	7.9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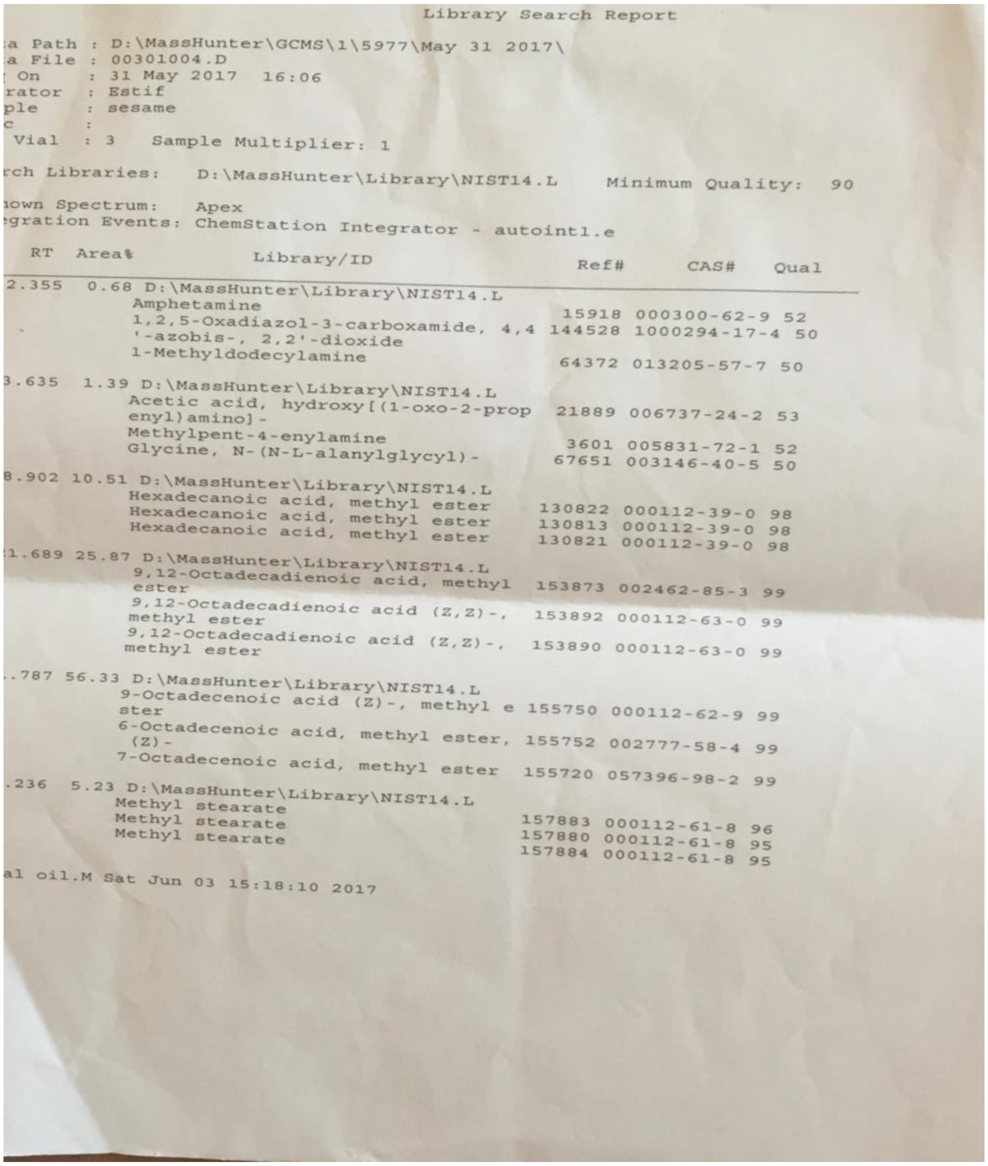
Acquired : 31 May 2017 16:06 using AcqMethod Faty acids 29.33 minsplitless.M
Instrument : AAU
Sample Name: sesame
Misc Info :
Vial Number: 3
Abundance
TIC: 00301004.D\data.ms
21.787
21.689
18.902
22.236
13.635
12.355
Time-->
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00 24.00 25.00 26.00 27.00 28.00



Library Search Report

a Path : D:\MassHunter\GCMS\1\5977\May 31 2017\
a File : 00301004.D
On : 31 May 2017 16:06
rator : Estif
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Vial : 3 Sample Multiplier: 1

rch Libraries: D:\MassHunter\Library\NIST14.L Minimum Quality: 90

nown Spectrum: Apex
egration Events: ChemStation Integrator - autoint1.e

RT	Area%	Library/ID	Ref#	CAS#	Qual
2.355	0.68	D:\MassHunter\Library\NIST14.L			
		Amphetamine	15918	000300-62-9	52
		1,2,5-Oxadiazol-3-carboxamide, 4,4'-azobis-, 2,2'-dioxide	144528	1000294-17-4	50
		1-Methyldodecylamine	64372	013205-57-7	50
3.635	1.39	D:\MassHunter\Library\NIST14.L			
		Acetic acid, hydroxy[(1-oxo-2-propenyl)amino]-	21889	006737-24-2	53
		Methylpent-4-enylamine	3601	005831-72-1	52
		Glycine, N-(N-L-alanylglycyl)-	67651	003146-40-5	50
8.902	10.51	D:\MassHunter\Library\NIST14.L			
		Hexadecanoic acid, methyl ester	130822	000112-39-0	98
		Hexadecanoic acid, methyl ester	130813	000112-39-0	98
		Hexadecanoic acid, methyl ester	130821	000112-39-0	98
1.689	25.87	D:\MassHunter\Library\NIST14.L			
		9,12-Octadecadienoic acid, methyl ester	153873	002462-85-3	99
		9,12-Octadecadienoic acid (Z,Z)-, methyl ester	153892	000112-63-0	99
		9,12-Octadecadienoic acid (Z,Z)-, methyl ester	153890	000112-63-0	99
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		9-Octadecenoic acid (Z)-, methyl ester	155750	000112-62-9	99
		6-Octadecenoic acid, methyl ester, (Z)-	155752	002777-58-4	99
		7-Octadecenoic acid, methyl ester	155720	057396-98-2	99
.236	5.23	D:\MassHunter\Library\NIST14.L			
		Methyl stearate	157883	000112-61-8	96
		Methyl stearate	157880	000112-61-8	95
		Methyl stearate	157884	000112-61-8	95

al oil.M Sat Jun 03 15:18:10 2017

Name	Synonymous
Palmic acid	Hexadecanoic acid
Methyl oleate	9-octadecenoic acid(z) methyl ester
Lineolic acid	9-12 octadecadienoic acid
Stearic acid	Methyl stearate

Oil source	C16:0 (Palmitic)	C18:0 (Stearic)	C18:1 (Oleic)	C18:2 (Linoleic)	C18:3 (Linolenic)
Cocoa butter	26	34	35	-	-
Corn	13	3	31	52	1
Cottonseed	27	2	18	51	Tr
Groundnut	13	3	38	41	Tr
Linseed	6	3	17	14	60
Olive	10	2	78	7	1
Palm	44	4	39	11	Tr
Canola	4	2	56	26	10
Rice bran oil	20	2	42	32	-
Safflower	7	3	14	75	-
Safflower (high oleic)	6	2	74	16	-
Sesame	9	6	41	43	-
Soybean	11	4	22	53	8
Sunflower	6	5	20	60	Tr
Sunflower (high oleic)	4	5	81	8	Tr

Table 1. Fatty acid composition (Gunstone, 2005)

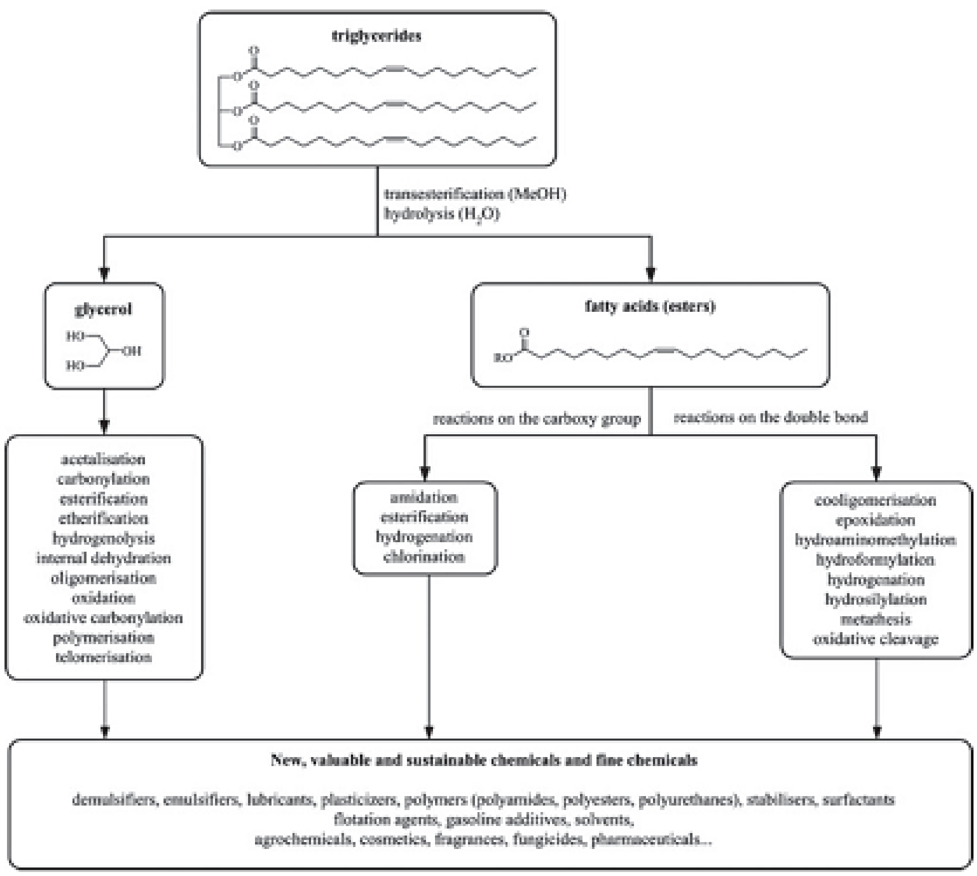

Fig. 1. Overview of possible reactions in oleochemistry (Behr & Gomes, 2010).