Sudan University of Science and Technology

College of Graduate studies

Comparative Study of Crude and Refined Sunflower and Peanut Edible Oils

دراسة مقارنة للزيت الخام والمكرر من عباد الشمس والفول السوداني

A Thesis submitted in partial fulfillment for the Requirements of M.Sc. in Chemistry

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بسم الله الرحمن الرحيم

قال تعالى (الله نور السماوات والأرض مثل نوره كمشكاة فيها مصباح المصباح في زجاجة الزجاجة كأنها كوكب دري يوقد من شجرة مباركة زيتونة لاشرقية ولاغربية يكاد زيتها يضيء ولو لم تمسسه نار نور على نور يهدي الله لنوره من يشاء ويضرب الأمثال للناس والله بكل شيء عليم)

صدق الله العظيم

DEDICATION

To the soul of my late parents,

My dear husband,

My children,

Ola, Abu baker, Azam and Abdalaziz

Acknowledgments

I express my deepest and sincere gratitude and everlasting appreciation to my Supervisor ***Dr.Elmugdad*** for his kind encouragement and supervision throughout this study. Special thanksare to Arab Sudanese vegetable oil Co. Ltd.For availing their laboratories and providing the samples, specially Miss Nafisa Ahmed Omer. Thanks are also due to ***Dr. AmelZiada*** for her continuous help and support throughout this work.

Above all appraise be to Allah, the compassionate the most merciful for giving me the health and strength to carry out this work.

Abstract

The objective of this study was to compare, crude and refined edible oils of each sunflower and peanut for the physical properties like color, moisture, density and refractive index, and chemical properties like peroxide value, free fatty acid, acid value, saponifiable number and unsaponifiable. For example the moisture content of sunflower oil was reduced from 0.07% to 0.02% after refining and from 0.13% to 0.02% for peanut oil, the peroxide value of sunflower oil was decreased from13.94meq/kg to 2.77 meq/kg and from 3.137meq/kg to 0.2 meq/kg for peanut oil, Gas chromatographic run for sunflower oil Showed that stearic acid area % was decreased from0.5193 to 0.3510 after refining, and for peanut oil was decreased from11.1643 to 1.0281 after refining.

The present study showed marked differences in the physicochemical properties of sunflower and those of peanut. These properties of each crude oil changed significantly when it was subjected to the refining process.

Fatty acid components of both crude and refined were determined by GC.

The study showed marked differences in the crude and refined of sunflower and peanut type.

ملخص البحث

تهدف الدراسة مقارنة الخواصالفيزيائية لزيت الطعام الخام والمكرر لزيتي عباد الشمس والفول السوداني مثل اللون والرطوبة والكثافة ومعامل الإنكسار والخواص الكيميائية مثل قيمة البيروكسيد والأحماض الدهنية الحرة والقيمة الحمضية ورقم التصبن والمواد غير المتصبنهنقص محتوى الرطوبة لزيت عباد الشمس من 0.07% الي 0.02% بعد التكرير ومن 0.13% الي0.02% لزيت الفول ونقصت قيمة البيروكسيد لزيت عباد الشمس من13.94ملي مكافئ/كجم الى2.77 ملي مكافئ/كجم بعد التكرير ومن 3.137 ملي مكافئ/كجم الى 0.2 ملي مكافئ/كجم لزيت الفول. اوضحت قياسات كروماتوغرافيا الغاز لزيت عباد الشمس ان النسبة المئوية للمساحة لحمض الاستياريك نقصت من 0.5193 الي 0.3510 بعد التكرير ولزيت الفول نقصت من 11.1643 الي 1.028 بعد التكرير.اظهرت الدراسة فوارق واضحه في الخواص الفيزيوكيميائية لزيتي عباد الشمس والفول السوداني، عند معالجتها بعد عمليات التكرير.وتم تعييننوعية الأحماض الدهنية الموجودة في العينات باستخدام جهاز كروماتوغرافيا الغاز. وقد أظهرت الدراسة فوارق واضحة في هذه الخواص والنوع.

CONTENTS

List of Tables

List of figures

Chapter One

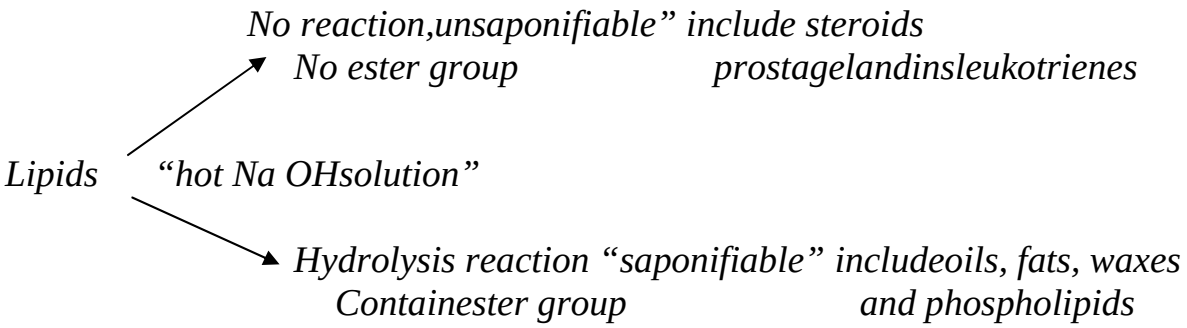
Introduction &literatureReview

1.Introduction and literature review

1.1Introduction

Oils and fats are water insoluble substances of plant or animal origin which consist predominantly of glyceryl esters of fatty acids, or triglycerides. The word fat is ordinary used to refer to triglycerides that are solid or ,more correctly, semi solid at ordinary temperature ,where as the word oil is used for triglycerides that are liquid under the same condition .No clean cut distinction can be made between the two words ,and they are used interchangeably unless the distinction between solid and liquid substances is important .(Bailey1979)

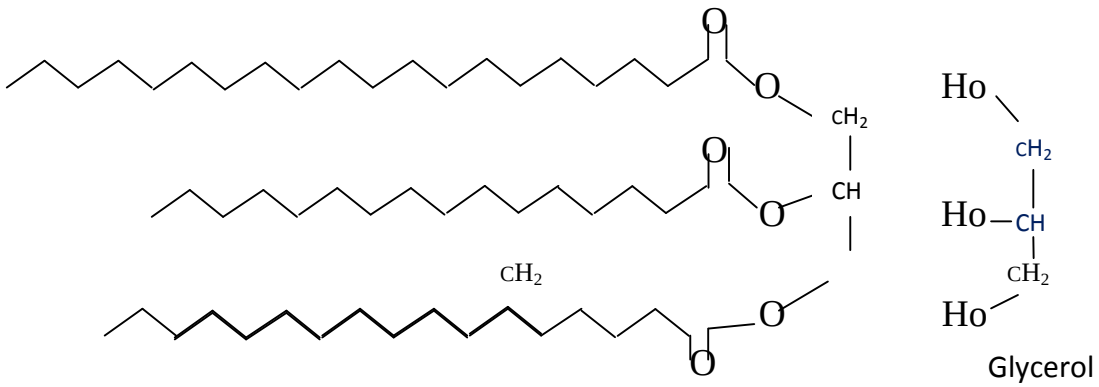
Oils and fats are naturally occurring esters of long straight chain carboxylic acids ;they belong to the saponifiable group of lipids .which are biologically produced materials that relatively insoluble in water but soluble in organic solvents (benzene, chloroform ,acetone, ether, and the like).The saponifiable lipids contain an ester group and react with hot sodium hydroxide solution undergoing hydrolysis (saponification):



Fats and oils are esters glycerol, the simplest triol (tri alcohol),in which each of the three hydroxyl groups has been converted to an ester .the acid portion of ester linkage ((fatty acid)) usually contains an even number of

carbon atom in an un branched chain of 12 to 24 carbon atoms The tri esters of glycerol fats and oils are also known as triglycerides .

Typical fat – triester glycerol



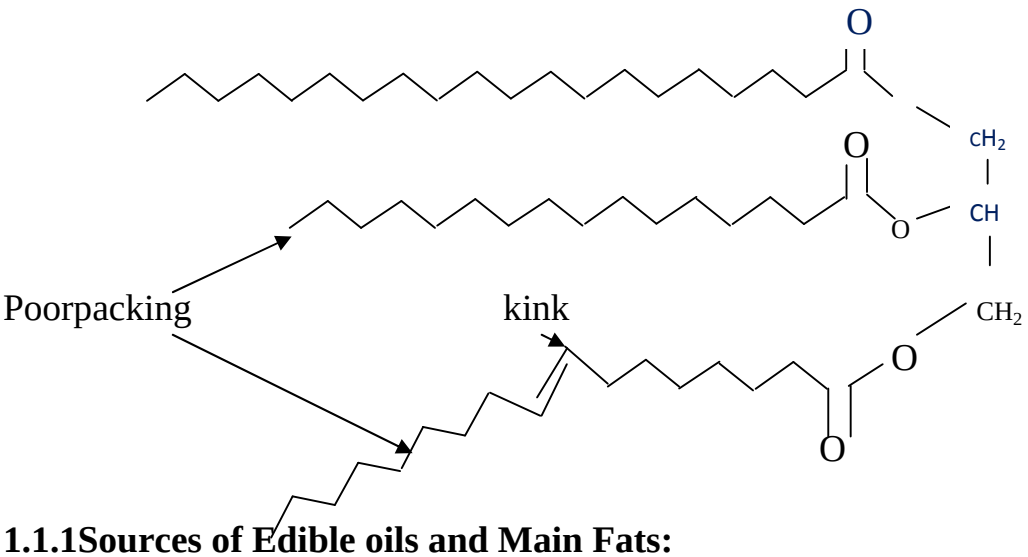
The difference between fats and oils is merely one of melting point :fats are solid at room temperature (20)where as oils are liquids . Both classes of compounds are triglycerides.

As glycerol is common to all fats and oils ,whether animal or vegetable ,it is the fatty acid part of the fat oil that is of interest .The differences among triglycerides (fats and oils) are because of the length of hydrocarbon chains of the acid and the number of position of double bonds (unsaturation).

The hydrocarbon chains of the fatty acid may be completely saturated (saturated fat) or may contain one or more double bonds .The geometric configuration of the double bond in fats and oils is normally cis .If the chain include more than one double bond , the fat is called poly un saturated . The presence of adouble bond puts akink in the regular zigzag arrangement characteristic of saturated carbons . Because of this kink in the chains ,the molecules cannot form aneat , compact lattice and tend to coil ,

so un saturated triglycerides often melt below room temperature and are thus classified as oils .

Unsaturatedfat(oil)– includescis double bond



1.1.1Sources of Edible oils and Main Fats:

The sources of edible oils and main fats are several hundred plants and animals which produce fats and oils in sufficient quantities to warrant processing into edible oils and however only afew sources are numerically significant .Table (1.1) below summarizes the major sources in the world and the method of processing .

Table (1.1)Major edible fats and oils in the world and method of processing :

Source	Oil content %	Prevalent method recovery
Coconut (dried copra)	66	Hard pressing
Cotton seed	19	Hard pressing or progressing direct solvent extraction
Sun flower	40	Prepress solvent extraction
Peanut (shelled)	47	Hard pressing or prepress solvent extraction

1.1.2 Oils and fats: processing and refining:

Crude fats and oils consist primarily of glycerides .However they also contain many other lipids in minor quantities .

All crude oils and fats obtained after rendering , crushing ,or solvent extraction ,inevitably contain variable amounts of non glycerides constituents like fatty acids ,partial glycerides (mono and diglycerides) , phosphatides ,sterols tocopherols , hydrocarbons , pigments (gossypol ,chlorophyll) ,vitamins (carotene) , sterol glucosides , protein fragments as well as resinous and mucilaginous materials , traces of pesticides and heavy metals .

Some of these materials are highly un desirable and must be removed to provide satisfactory processing characteristics and to provide desirable , color, odor , flavor and keeping quantities in the finished products . These objection able co constituents are removed during the refining process in such away that the glyceride yield and the desirable constituents in the oil are not affected .

Figure (1.1) shows the different stages in seed preparation and classical chemical refining process . the general methods employed to produce edible oils suitable for human consumption consist of (a) seed preparation (b) extraction , (c) degumming (d) neutralization (e) bleaching (f) deodorization (g) hydrogenation and sometimes winterization.

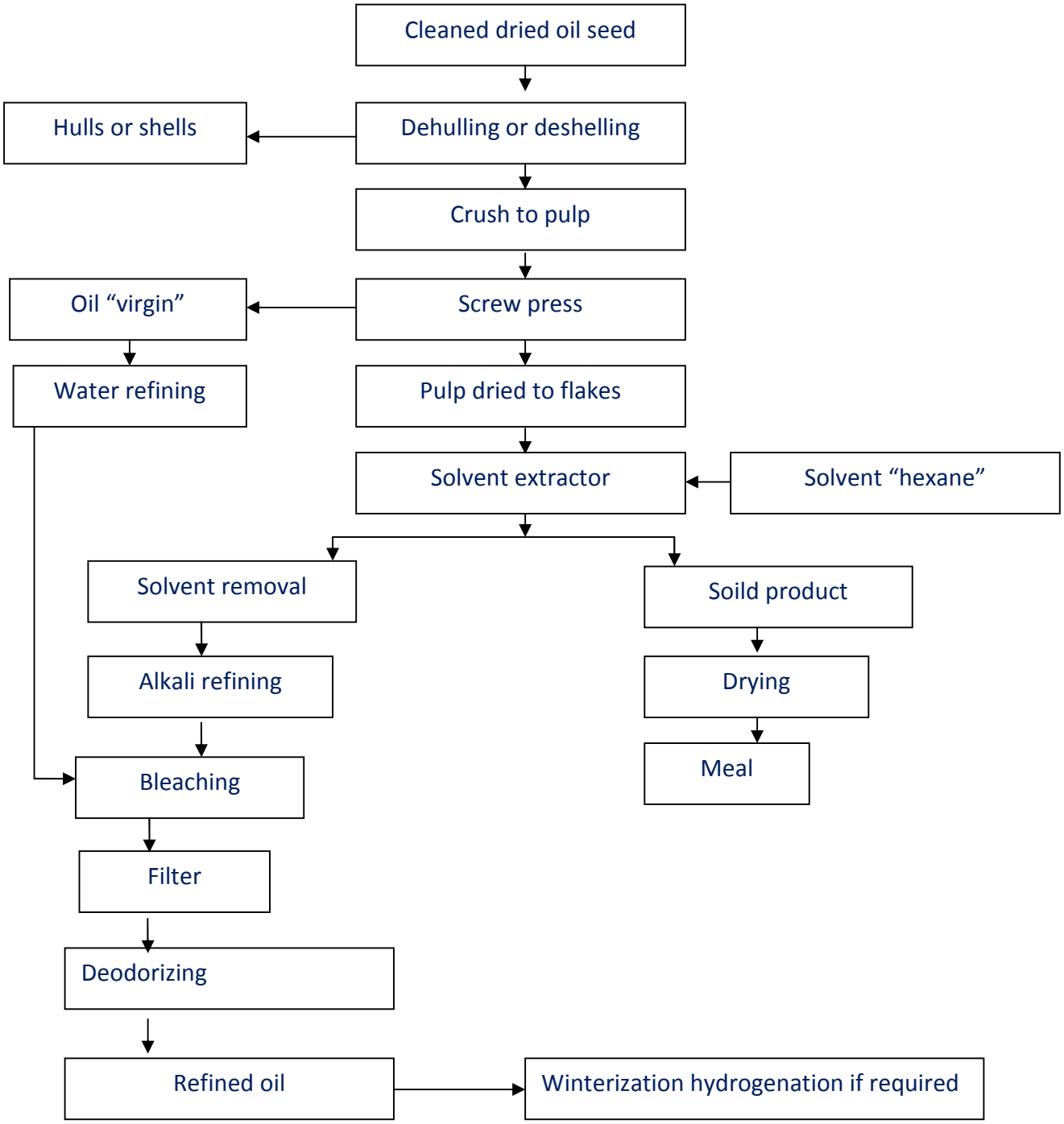


Figure (1.1) Essential steps in the extraction and refining of edible oil from oil Seeds. (Ali,Alali&Steight JG 2005)

1.1.3 Production of Oils

1.1.3.1 Seed Arrival

Seeds can reach aplant in lots ranging from just a few bags to 60000 tone shiploads . It is essential for the plant to control the weight and the quality of each load of seed that is reaching the plant . Weighing and sampling protect the interests of both the buyer and the seller and provide the basis for any necessary price adjustment .

1.1.3.2 Seed Weighing

Weighbridges are usually used for trucks rail cars and various types of inline scale are used in other cases .

The reliability and precision of the weighing equipment is of utmost importance and must be controlled , preferably by specialized body , at regular intervals . The precision sought must be less than one in athousand . Equally important is the use of well proven , well defined and fixed procedures to weigh the incoming seeds in order to prevent any fraud .

1.1.3.3Sampling

As the seed arrives at the plant , it must be sampled .the quality of the raw material determines how it will have to be processed and hence at what cost , how it will be stored and implied in the specifications of the seed purchase contract , must be checked carefully and un ambiguously .

Parameters that are controlled include, as aminimum:

Moisture,

Foreign material,

Proteincontent,

Oil content.(Hamm, Hamilton&Calliauw 2013)

1.1.3.4Seed Preparation

When oil seed are received at the oil mill they still contain plant residues , damaged seeds , dust , sand , wood , pieces of metal , and foreign seeds . The oil seeds are carefully cleaned of these materials using magnets , screens , and aspirator systems . The cleaned seeds are dried to remove moisture. Next , the dried oil seeds are usually decorticated to remove the hull that surrounds the oil seed meat before being further processed . Hulls always contain much less oil than kernels or meats. Further , removal of the hull reduces the amount of material that must be handled , extracted , and refined . De hulling is normally preformed very carefully , to ensure that the meat is not broken into too many small pieces .

Corrugated roller mills or bar mills are used to cut or break the hull to free the meat . The hulls are separated by screening and air clarification. Hulls may be blended back with meat to control protein level , sold as a cattle feed , or burned in boilers to generate steam and electrical energy .

1.1.4 Extraction

The purpose of oil extraction is twofold : first , extracting the maximum amount of good quality oil and then getting maximum value from the residual press cake or meal . The following three methods with varying degrees of mechanical simplicity are used :

1- Rendering

2- Pressing with mechanical presses

3 - Extracting with a volatile solvent.

1.1.4.1 Rendering

The rendering process is applied on a large scale to the production of animal fats , such as tallow ,lard , bone fat , and whale oil . The fatty tissues are chopped into small pieces and are boiled in steam digesters . The fat is gradually liberated from the cells and floats to the surface of the water , where it is collected by skimming . A similar method is used in the extraction of palm oil from fresh palm fruits .

1.1.4.2 Pressing

Oil seeds don't have fat cells like those of animals for storing fats . Instead oil is stored in microscopic globules through the cells in these cases , rendering will not liberate the oil from the cellular structures and the cell walls are broken only by grinding , rolling or pressing under high pressures to liberate the oil . The general sequence of modern operation in pressing oil seeds and nuts is as follows :

(1) Preparation of the seed to remove stray bits of metals and removal of hull .

(2)Reduction of particle size ofthe kernels (meats) by grinding and

(3) Cooking and pressing in hydraulic or screw presses.

Efficient oil extraction by a mechanical press highly dependent on having the correct preparation before pressing . The extraction stage itself is carried out using a screw press .

The press is fed by means of variable speed conveyor , within the feeder unit . The feeder regulates the flow of material into the press and there by controls the loading on the press main motor . Oil released along the length of the cage is allowed to drain into the base of the press where it is collected . The solid material (press cake) remaining within the press is finally discharged into conveyors to be removed for subsequent processing or storage .

Oil pressed without heating contains the least amount of impurities and is often of edible qualitywithout refining or further processing such oils are known as cold-drawn , cold pressed , or virgin oils . The expressed oil from cooked seeds contains greater quantities of non glyceride impurities such as phospholipids ,color bodies ,and un saponifiable matter such oils are highly colored and are not suitable for edible use .

1.1.4.3 Solventextraction

The press cake emerging from a screw press still retains 3 to 15 percent of residual oil . More complete extraction is done by solvent extraction of the residues obtained from mechanical pressing .The common solvent for edible oil is commercial hexane or heptanes , commonly known as petroleum ethers boiling in the range 146 to 156 F (63.3 to 68.9 °C) . After extraction ,maximum solvent recovery is necessary for economical operation The solvent is recovered by distillation and is reused. The extraction oil is mixed with prepress oil for refining . The extracted meals contain less than 1 percent of residual oil . In large scale operation , solvent extraction is a more economical means of recovering oil than mechanical pressing .

1.1.5 Refining (degumming and neutralization)

The usual refining of vegetable oils involves degumming and alkali refining. Degumming mainly reduces the phosphatides and metal content of the crude oil by mixing it with an acid and water . The phosphatides are present in free hydratable form (HP) or in non hydratable form (NHP) , mostly in combination with some Ca^{++} , Mg^{++} , or Fe^{++} . In alkali refining , the NHP that remains behind in the oil after acid treatment , and the free fatty acids formed during the hydrolysis (lipolysis) of the HP , are further removed by neutralization .

Degumming consists of treating the oil with a small amount (0.05 percent) of concentrated phosphoric acid and water , followed by centrifugal separation of coagulated material (lecithin) . Refining with alkali removes free fatty acid that are formed during the lipolysis of the fat or oil before rendering or extraction .

The oil is treated with an excess of 0.1 percent caustic soda solution and the mixture is heated to about 75°C to break any emulsion formed . The mixture is allowed to settle. The settling , called (foots) are collected and sold as (soap stock) . In the continuous system , the emulsion is separated with centrifuges . After the oil has been refined , it is usually washed with water to remove traces of alkali and soap stock . After water washing. The oil may be dried by heating in a vacuum or by filtering through a dry filter and material .

In all degumming processes the efficiency of the degumming treatment depends to a great extent on the quality of the crude oil . A good quality fresh oil is easier to degum than adulterated oil . This is a direct

consequence of the NHP to HP ratio : the lower the NHP content , the easier the degumming procedure and the better the degummed oil yield .Figure 1.2 Summarize the different steps of degumming processes.

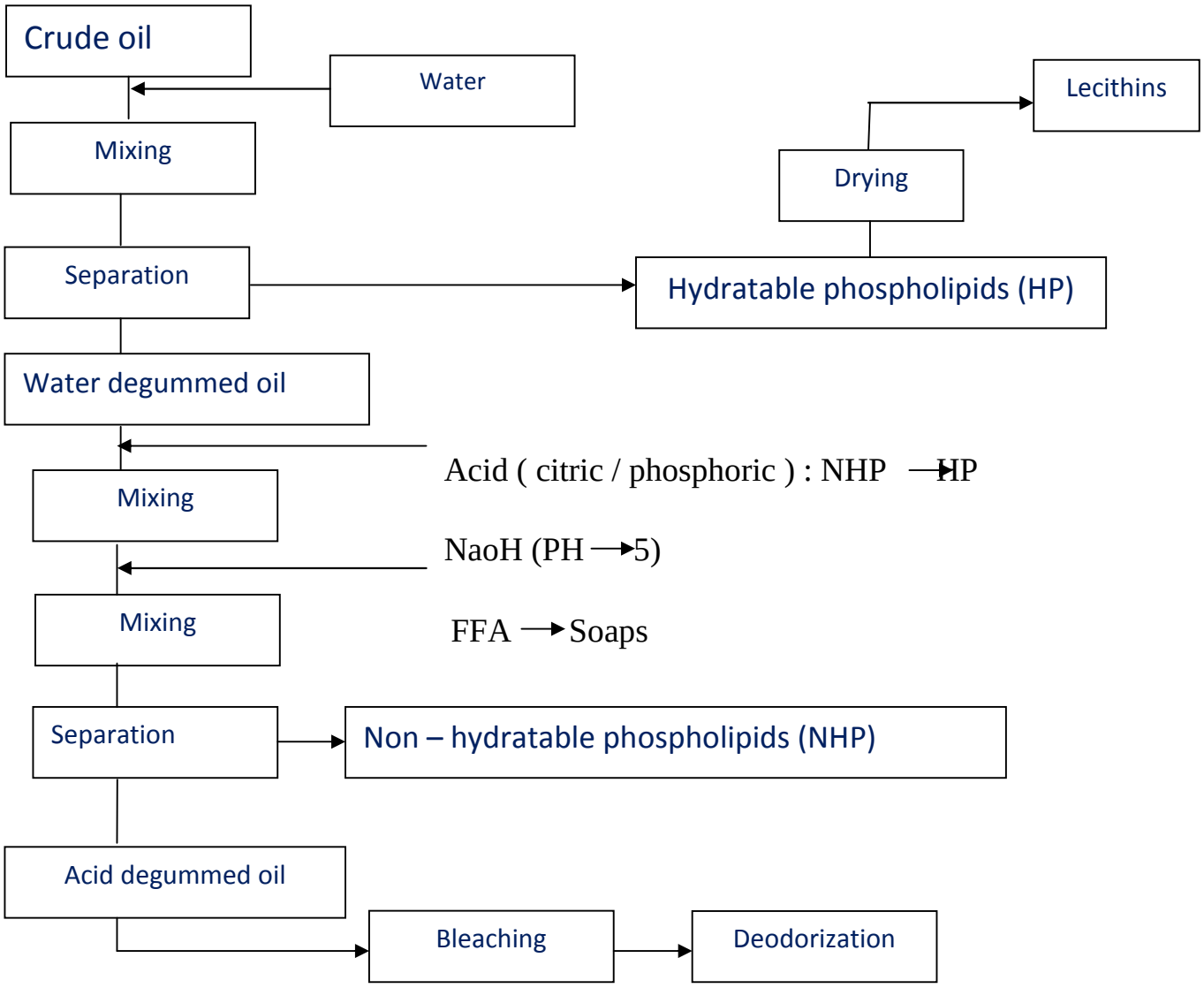


Figure 1.2 Generalized flow sheet for degumming process

1.1.6 Bleaching

The refined oils are usually dark in color owing to presence of some pigmented materials such as chlorophyll or carotenoids and minor impurities like residual phosphatides , soaps , metals and oxidation product . Bleaching reduces the color by absorbing these colorants on bleaching earth (bentonite clays) or activated charcoal ,or both . In addition to decolorization , bleaching clay also absorbs suspended matter and other minor impurities .

The bleaching process comprises three stages

1- Initial mixing of oil with bleaching earth.

2- Oil heating under a vacuum with sparge steam to ensure complete contact between the oil and the earth .

3- Filtration on hermetic leaf filters , followed by polishing filtration.

The spent cake is dried by steam blowing and the recovered oil is recycled . Natural bleaching clays are aluminum silicates (bentonite , attapulgite , and montmorillonite) , containing relatively high amount of Mg^{++} , Ca^{++} , or Fe^{++} . The clays are generally activated by heat treatment. The high metal content , however , limits the adsorptive activity of these clays . The metals can be removed from the reactive spots by means of acid treatment , yielding clays with much higher adsorptive capacity , in some cases , active carbon is added in the course of bleaching to improve the removal of blue and green pigments as well as poly cyclic aromatic hydrocarbons.

1.1.7 Deodorization

Most oils and fats even after refining have characteristic flavors and odors owing to the presence of minor amount of free fatty acids , aldehydes , ketones and other compounds . The concentration of these undesirable

substances , found in most oils , is generally low , between 0.2 and 0.5 percent .

Deodorization is usually conducted at a temperature between 220 and 260 °C at a pressure between 2 and 4 m bar , and under injection of 0.5 to 3 percent steam in a stainless steel vessel .

The different stages in the deodorization process are as follows :

-Deaeration.

-Heating.

-Deodorization or steam stripping.

-Heat recovery or cooling.

-Final cooling.

The volatiles evaporated during deodorization , are condensed and are usually recovered in a direct condenser or scrubber for fatty substances . It is generally the best choice for modern integrated refineries . About 0.01 percent of citric acid is commonly added to deodorized oil to inactivate trace metal contaminants such as soluble iron or copper compounds that would otherwise promote oxidation and the development of rancidity .

1.1.8Hydrogenation

Hydrogenation is used to convert liquid fats to plastic fats thereby making them suitable for the manufacture of margarine or shortening . Hydrogenated oils and fats also exhibit improved oxidative stability and color . In the hydrogenation process , the hydrogen is added directly to double bonds of fatty acids . The most unsaturated fatty acid groups are most easily

hydrogenated and thus react first with the hydrogen if condition are somewhat selective , that is to add hydrogen to the linolenic (three double bonds) and linoleic (two double bonds) acid radicals before adding to the oleic (one double bonds) acid radicals . The reaction may be generalized:

Raney nickel type and copper containing catalysts are normally used. Variables affecting hydrogenation include the catalyst , temperature , hydrogen pressure, and amount of agitation . If very hard fats with low amount of un saturation are derived and selectivity is un important , higher temperatures and pressures are employed to shorten the reaction time and to use the partially spent catalyst that would otherwise be wasted . The catalytic hydrogenation is frequently accompanied by isomerization with a significant increase of melting point , caused , for example , by oleic (cis) isomerizing to olaidic (trans) acid . The trans isomers are much higher melting than natural cis forms . After hydrogenation , hot oil is filtered to remove the metallic catalyst.

1.1.9 Winterization

Winterization is an operation that consists in removing from certain oils the components that solidify at low temperature and therefore are a source of turbidity or setting in the bottle . The process consists in filtering cooled oil under strict control .Winterizing is not practiced so widely in hot countries and its application is restricted mainly to sunflower, maize , cotton , olive , rice bran , and partially hydrogenated soybean oils .Normally a winterized oil should remain clear for at least 24 h at 0 °C . This corresponds to a wax content of below 50 ppm. (Ali,Alali&Steight JG 2005).

1.2 Preparation and pressing of sunflower seed

The high oil content of sunflower seed requires a twostepProcess – mechanical pressing followed by solvent extraction – to fullyrecover the oil.

The seed is composed of a kernel protected by a shell, and it is customaryin many markets to dehull the seed to a degree before extracting the oil.

Sunflower oilseed originates from Argentina, where it is still the secondlargest oilseed crop. Its main area of production is EasternEurope, including Russia. It is also the major oilseed grown in the southernhalf of France, Spain and Turkey.

The sunflower is well known (as suggested by its name) by its heliotropism.

The stem is tall, up to 4m high, has a diameter of 2–8 cm and supports very wide leaves and a heavy flower (15–40 cm wide). The flower is surrounded by orange leaves and carries a quantity of seeds corresponding to 50–60% of its weight. Individual seeds have an elongated shape, with a length of 7–19 mm, are covered by a ligneous shell, which is very hard, abrasive and waxy (3%wax) and are generally of black color.

The exact composition of sunflower seed varies according to its origin. Theun-decorticated seed may contain from 42 to 48% oil. The shells represent25–30% of the total weight.

Sunflower seeds contain:

• 70–75% pure kernels, containing 55–65% oil, 5–10% moisture, 52–57%

Protein (on an oil- and water-free basis) or 16–20% as such, the balancebeing carbohydrates and ash.

• 25–30% pure hulls, containing 1.5–3.0% oil, 7–11% moisture, 4–6%protein, 50–60% fiber, 5–7% ash and carbohydrates.

When arriving at the plant, sunflower seeds are generally dried to 7–8%moisture – adequate for their storage and for their later processing. As withrapeseed, the

extraction of the oil is carried out in a two-stage processinvolving mechanical pressing followed by solvent extraction.

If processed without separating the shell, the deoiled sunflower meal hasa protein content of around 26–28% and fiber content of around 24–26%.

This composition makes it suitable for feeding ruminants, but less so forfeeding poultry and other monogastric animals. It is customary in regionswhere sunflower seed is a major source of seed oil to separate about 15%of hulls (on a seed basis) and produce feed meal with about 34% proteincontent, or to separate up to 22% of hulls and produce high-protein mealwith 37–39% protein and 14–15% fiber. The dehulling consists in most casesin opening the seeds by impact, then separating hulls and meats by screeningand aspiration. A two-step dehulling process is generally used to producehigh-protein meal, in order to avoid entraining more than 1–2% oil with the hulls.

The hulls are in most cases conveyed to a specially designed boiler, wherethey are burnt to produce steam for the process. Akilogram of hulls produces between 3.5 and 4.5 kWh of heating energy. In large plants, the boiler canbe coupled with a turbine to produce electricity. A sunflower seed crush andrefining plant producing high-protein meal produces enough hulls to generateall the steam and electric power it needs to run.

A minimum of 8% of hulls are left with the meats. Without this, therewould not be enough grip for the press to efficiently extract the oil, and thecake would lack the structure required to stay firm in the extractor.

After dehulling, the meats are flaked to 0.4mm thickness, cooked andmechanically pressed to 18–22% oil, then sent to the solvent extractor.

When processed unhulled, seeds are cracked in a double-pair crackingmill, cooked and pressed before solvent extraction. Flaking the cracks beforecooking improves

deoiling, but this has to be balanced against the wearcaused by the hulls to the flaking rolls. Processing un-decorticated seeds willalso substantially increase the power consumed by the press per ton ofproduct entering, and accelerate wear in the press.

Typical prepressed cake from sunflower seed has an oil content of between 18 and 20%, 5–7% moisture and a bulk density of 0.4–0.5 tons/m^3.Sunflower seed press cake is hard and must be broken into pieces. Theaverage size of cake pieces going to the extractor should be around 6 mm,and the proportion of fines limited to 10%.

1.3 Peanut (groundnut)

Groundnut or peanut is a herbaceous annual plant of the leguminous branch,originating from tropical America. The most important countries producinggroundnut are India, the USA, Argentina, China and some tropical Africancountries. The fruit is made of an external shell (21–29%) and the nut(79–71%), consisting of:

• A thin hull surrounding the nut (2–3%);

• The nut (69–73%);

• The germ (2.0–3.5%).

The groundnut contains 40–55% oil, 30% protein and 12% hydrocarbonmatter; its high vitamin B content makes the groundnut an essential part of abalanced diet in tropical countries.

A large amount of peanuts produced are locally consumed; they are eatencrude or grilled, slightly roasted, and are used as appetizers and in theconfectionery industry.

‘Peanut butter’ is made from a preparation of crushed groundnuts, roastedand mixed with 5–7% groundnut oil and salt.

Peanuts are available unshelled or decorticated. Once decorticated, however,it is difficult to store them, as the oil acidifies rapidly. The decortication allows the transport volume to be reduced to a considerable extent. Generally,peanuts are not decorticated except when they are to be used in theproduction of industrial nonfood oil.

Decortication is done either by corrugated rolls, by pounding or by centrifugation;the shells are then separated from the nuts by ventilation and thegroundnuts are dehulled from their fine husks.

To obtain the oil from decorticated groundnuts, they are cleaned andcracked before pre-expelling, and the cakes are cracked, heated and flakedbefore solvent extraction.

Depending on whether they are dehulled or not, groundnut cakes arecalled ‘white cake’ or ‘brown cake’. The cakes are called ‘shelled’ when theystill contain a certain amount of shell. Shelled cakes are used as fertilizer.Decorticated, they are used for animal feed.

The bran made by the hulls is an excellent animal feed as it contains14–19% oil and 22% nitrogen-containing matter.

The extracted meal is still very rich: 41–50% protein content. Shells andhulls are used as fertilizers or as combustible feed for the boiler.

Refined groundnut oil is excellent food-grade oil.

Aspergillusflavus, a mould present in the soil, mostly in tropical countries,frequently contaminates peanuts. The mould generates toxic products, themost dangerous being aflatoxin B1. It grows particularly well on materialsrich in carbohydrates. To develop, it needs a relative humidity of 80% anda temperature between 30 and 40 ◦C. It is especially active on seeds with15–30% moisture and does not develop on seeds with moisture below 8%.

For groundnuts, the most critical time is harvesting: any defect in drying beforeensiling in tropical conditions and any increase in moisture afterwards mayhave extremely bad consequences. Therefore, drying to 8%or less is required.Once present in a contaminated nut, aflatoxine is not eliminated in processingand will end up in the meal, where it represents a danger to animalhealth. This toxin is resistant to high temperature and is practically insolublein hexane; basic, acidic and oxidizing agents are the most appropriate meansof breaking it down. (Hamm, Hamilton&Calliauw 2013).

1.4 Quality control tests

Anumber of analytical methods are used for assessing the quality and deterioration of oils or fat during refining and subsequent storage condition . These methods are summarized below:

1.4.1 Physical tests

1.4.1.1 Color

The color of oils is usually compared in lovibondtintometer using a 1 inch or 5 inch cell . Crude vegetable oils will have a color varying from red to green viayellow and brown. The main contributors to this color are carotenoids(red) and chlorophyll (green). Color is mainly removed during bleachingby adsorption or by interaction with the chemical active sites on thesurface of the bleaching earth. Carotenoids are also decomposed duringhigh-temperature deodorization.

Color measurements are based on comparison with standard colorglasses. Several color standards are used in the oils and fats industry:

Lovibond 1 and 51⁄4 inch cell, Gardner, FAC and iodine scale.

In general, refined oils and fats should be more or less colorless. The‘natural’ oil or fat color does not always match the desired color of the foodproduct containing

this oil or fat. In addition, some color particles promoteoil deterioration or contribute to off-flavours.

1.4.1.2 Moisture

Moisture and dirt in crude oils and fats can be caused by oil crop residuesremaining in the oil after the extraction process or else can originate fromthe supply chain (dirty tanks, condensation of moisture in air, steam blowingetc.). Moisture and dirt are removed in the first refining step: neutralization inchemical refining or bleaching in physical refining. Bleaching earth residuesor spots of polymerized material from a fouled deodorizer can also be thecause of dirt in fully refined oils and fats.

The moisture (and volatile matter) content is measured by determiningthe loss of weight during heating at 105 ◦C on a hot plate or in an oven.

There should be no presence of visible dirt or moisture in a fully refined oilor fat. Dirt should be absent for quality reasons, while free moisture can bethe cause of microbiological contamination.(A.O.C.S1951)

1.4.1.3Density

The density of oil was determinedbypycnometer (50 ml). The pycnometerwas first dried and weighed empty , then it was weighed filled with sample and waterto determine the weight of the sample and water by difference and hence the volume of the sample to calculate the density of the oil sample and at the end calculated the density by equation below

D=W (sample)/V (sample)

Where D=Density of oil

W (sample) =weight of sample .g.

V (sample) =volume of sample.(A.O.C.S 1951)

1.4.1.4Refractive index

The refractive index of a substance is the ratio of the speed of light in a vacuum to the speed of light in the substance .

1.4.2 Chemical tests

1.4.2.1 Peroxide value

The peroxide value is a measure of the peroxides contained in the oil . During storage peroxide formation is slow at first during an induction period which may vary from a few weeks to several months according to the particular oil or fat , the temperature ,etc., and this must be borne in mind when interpreting quantitative results .

1.4.2.2 Acid value

The acid value of an oil or fat is defined as the number of mg of potassium hydroxide required to neutralize the free acid in 1 g of the sample . The result is often expressed as the percentage of free acidity .

The acid value is measure of the extent to which the glycerides in the oil have been decomposed by lipase action . The decomposition is accelerated by heat and light.

1.4.2.3 Saponifiable number

The saponifiablenumber of an oil is defined as the number of mg of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1 g of the sample .

Soap is formed during the saponification for example:

The esters of the fatty acids of low molecular weight require the most alkali for saponification , so that the saponifiablenumber is inversely proportional to the mean of the molecular weights of the fatty acids in the glycerides present .

1.4.2.4 Unsaponifiable matter

Can be defined as the material present in oils and fats which after saponification of the oil or fat by caustic alkali and extraction by a suitable solvent remains non-volatile on drying at 80 °C.

The unsaponifiable matter includes hydrocarbons , higher alcohols and sterols . Most oils and fats of normal purity contain less than 2% of unsaponifiable matter .(Pearson 1970)

1.5 Fatty acid composition

The fatty acid composition of sunflower oil makes it very important oil to be used for cooking. The fatty acid composition of refined sunflower oil as reported by AOAC, (1984)., Mar ,(2005), includes myirstic acid , palmitic acid , palmtoleic acid , stearic acid , Oleic acid , Linoleic acid , linolenic acid , and Arachidic acid .

It is considered a highly polyunsaturates oil due to its high linoleic acid content (48.3 to 74.0%) and its moderate oleic acid content (14.0 to 39.4%) and low level of saturated fatty acid content (12%) on average(Gunstone2002). These fatty acids are essential fatty acid to the body because it cannot be synthesized by the body. (Abitogun 2008).

In the peanut oil myristic, palmitic, palmitoleic, stearic, oleic, linoleic, linolenic, arachidic,gadoleicandbehenic acids were identified mainly by gas chromatography. The major fatty acids of peanut seeds were oleic, linoleic and palmitic acids. (Ozcan&seven2003)

Thetemperature used for cooking should be higher than 80°C to deactivate enzymes, but at temperatures higher than 100°C changes in aroma and taste of the resulting oil arise. In addition, higher cooking temperatures result in an increase of the amount of phosphorus and free fatty acids in the oil. (Matthaus 2012)

1.6 Gas Chromatography

Gas Chromatography is one of the most widely used techniques in analyticalchemistry. It is a sensitive technique for separating a complex mixture ofrelatively volatile components.

In gas-liquid chromatography (GC) the stationary phase is a liquid which is chemically coatedover an inert support. The sample of a mixture of compounds is introduced intothe mobile gas phase stream and is carried onto the column. Once on the column,separation of components, as in all chromatography, is a result of the difference inthe various forces by which the stationary phase tends to retain each of thecomponents. Whatever the nature of this retention, be it by partitioning (solubility), chemical bonding, polarity or molecular filtration, the column will hold back some components longer than others. In GC the important columnretention factors are partition of compounds between the stationary phase and the

mobile gas phase, and solubility of the compounds in the stationary phase,although some of the others mentioned do also play a smaller, incidental part.

Thus, the component compounds move down the column at a rate determined by many factors, but mainly by their "strength" of retention. Here, this refers to both their degree of solubility in the liquid stationary phase and also to their volatility. Bearing in mind that different compounds will have different solubilities in the liquid phase and different volatilities, they will progress down the column atvarying rates and consequently, assuming the column is long enough, the solute components of a mixture will emerge separately at the outlet of the column. Inother words, all components pass through the column at different speeds andemerge in the inverse order of their retention by column materials.

Emerging from the gas chromatographic column, the separated gaseouscomponents enter a detecting device immediately attached to the column and thissenses the presence of a compound. Here the individual components register aseries of signals which are amplified, fed to a chart recorder, an electronic integrator or computer, and a trace is produced. The separated components appear as a succession of peaks above the base line. This is known as thechromatogram. (Bruce R.DA'rcy 2003)

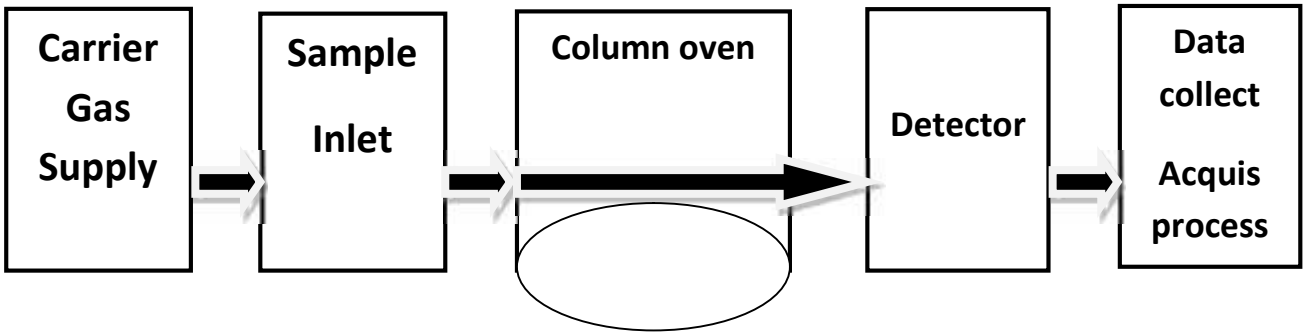


Figure (1.3) Scheme of a gas chromatograph

(Kenndler E 2004)

CHAPTER TWO

Materials &Methods

2.1Sampling and Preparation

The samples were collected from Arab Sudanese vegetable oil Co according to the standard method of sampling to insure that they are representative samples.

2.2Materials

2.2.1 Chemicals

- Distilled water.
- Glacial acetic acid + chloroform 3:2.
- Potassium iodide.
- 0.01 M sodium thiosulfate.
- Starch indicator.
- Neutral alcohol (ethanol 95%).
-0.1M Sodium hydroxide .
- Diethyl ether.
- Phenolphthalein solution (1%).
- Potassium hydroxide.
- Alcohol (95 %).
- Alcoholic potassium hydroxide.
- Aqueous 3 M potassium hydroxide solution.
- 0.5 M hydrochloric acid.

- 0.5 M potassium hydroxide.

2.2.2 Apparatus

- Porcelain dish.
- Hot plate.
- Thermometer.
- Test tubes.
- Beakers.
- Ice bath.
- Pycnometer (50 cm^3).
- Refractometer.(Abbe)
- A reflux condenser.

2.2.3 Instruments

- Digital LovibondTintometerModel F.
- Digital LovibondTintometer PFX 995.
- Digital Mettler Toledo.
- GC. (SHIMADZU),coloumnDB1,Diamter 0.25ml,Detector:FID.

2.3Methods

2.3.1 Color

This test was done by Digital LovibondTintometer,cell 4 inch for crude oil, and DigitalLovibond PFX995 Tintometer, cell 5.25 inch for refined oil. The glass cell was cleaned ,dried,filled with the oil and placed in the tintometer.The color of the oil was matched with the standard slides red,yellow,and blue colors.The results were recorded.

2.3.2 Moisture:

This test was done by Digital Mettle Toledo and 4 g of the sample were taken .

2.3.3Density: The density of oil was determined by pycnometer (50 ml). The pycnometer was first dried and weighed empty , then it was weighed filled with sample and water to determine the weight of the sample and water by difference and hence the volume of the sample to calculate the density of the oil sample and at the end calculated the density by equation below

D=W (sample)/V (sample)

Where D=Density of oil

W (sample) =weight of sample .g.

V (sample) =volume of sample. (A.O.C.S, 1951)

2.3.4Refractive index

The refractive index was determined using refractometer.Adrop of oil was placed on surface of the lower prism. The prisms were closed and the mirror and light were adjusted until a dark border line was observed on the cross wire. The refractive index was determined.

2.3.5Peroxide value

5.o g of the sample were weighed in aflask,to which 25 cm^3 of glacial acetic acid and chloroform(3:2) and 0.5 cm^3 of potassium iodidewere added. The flask was closed shaked and left to stand in the dark for 1 minute , preferably at 15-25 C°. About 30 cm^3 of water were addedand titrated against 0.01 M sod thiosulphate with vigorous stirring using starch indicator to detect end point when the color of the solution changed from blue to colorless .

2.3.6Free Fatty Acid

7 g of the sample were weighed to which 50 cm^3 of neutral alcohol (ethanol 95%) and 2 cm^3 of phenolphthalein were added .The solution was titrated against 0.1M sodium hydroxide until its colour changed to pink to indicate the end point.

2.3.7Acid value

25 cm^3 diethyl ether were mixed with 25 cm^3 alcohol and 1 ml of phenolphthalein solution (1%) and carefully neutralized with 0.1 M alkali . 1-10 g of the oil was dissolved and titrated with aqueous 0.1 M sodium hydroxide while shaking constantly until a pink color which persisted for 15 sec was obtained .

2.3.8Saponifiable number

2 g of the oil was weighed into a conical flask and 25 cm^3 of the alcoholic potassium hydroxide solution was exactly added during which it was shaked frequently a reflux condenser was attached to the flask which was heated in boiling water for 1 hr . 1 ml of phenolphthalein (1%) solution was added and the hot excess alkali was titrated with 0.5 M hydrochloric acid (titration =a ml). Ablank was carried out .Utmost care was taken during the titrationthe titrated liquid in the sample flask was retained to determine the unsaponifiable matter.

2.3.9Determination of unsaponifiable matter

After the titration of the saponifiable number the neutralized liquid was made alkaline again with 1 $cm^3$of aqueous 3 M potassium hydroxide solution , transfered to a separator and washed in with water (50 ml less the volume of 0.5 N hydrochloric acid used). The solution was extracted while still just warm 3 times with 50 cm^3 quantities of diethyl ether . Each ether was extracted into another separator containing 20 cm^3 of water . After the third

extract had been added , the combined ether extracts were shaked with the first 20 $cm^3$of wash water and then vigorously with two further 20 cm^3 quantities . The ether extract was washed twice with 20 cm^3 of aqueous 0.5 M potassium hydroxide solution and at least twice with 20 cm^3 quantities of water until the wash water was no longer alkaline to phenolphthalein . The ether extract was poured into a weighed flask , the solvent was evaporated off , the residue was dried at not more than 80 ◦C and weighed to constant weight .(A.O.C.S 1971).

2.3.10 Fatty acid analysis:

[Injection port SPL1]

Injection Mode: Split.

Temperature: 250°C.

Carrier Gas: N2/Air.

Flow Control mode: Pressure

Pressure: 94.2kpa.

Total Flow: 16.2ml/min.

Column Flow: 1.00ml/min.

Linear Velocity: 28.2cm/sec.

Purge Flow: 3.0ml/min.

[Column Oven]

Initial Temperature: 100°C.

Equilibration Time: 0.0min.

[Column Information]

Column Name: DB-1.

Serial Number: US6554753H.

Film Thickness: 0.25um.

Column Length: 30.0m.

Inner Diameter: 0.25mm ID.

Column Max Temperature: 350°C.

Description: non polar hydrocarbon/poly nuclear/aromatic/steroid.

[Detector channel 1 FID1]

Temperature: 300°C

Signal Acquire: Yes.

Sampling Rate: 40 msec.

Stop Time: 60.0min.

Delay Time:0.00min.

Makeup Gas: N2/Air.

Makeup Flow:30.0ml/min.

H_2 Flow:40.0ml/min.

Air Flow:400.0ml/min.

Chapter Three

Results, Discussion& Recommendations

3.1Physical Tests

3.1.1Color

Table 3-1:Shows the lovibondtintometercolour of crude and refined oils of sunflower and peanut

The oil	Red	Blue	Yellow
Crude sunflower	4.5	0.9	25
Refined sunflower	1.0	0.0	5.0
crude peanut	3.6	0.8	25
Refined peanut	1.0	0.0	4.2

3.1.2 Moisture

Table 3-2: Shows the moisture percentage of crude and refinedoils of sunflower and peanut

The Oil	Moisture
crude sun flower	0.07%
Refined sun flower	0.02%
crude peanut	0.13%
Refined peanut	0.02%

3.1.3 Density

3.1.3.1Calculation:

Crude sunflower:

Weigh of empty pycnometer=17.79g

Weigh of pycnometer+water=45.0g

Weigh of pycnometer+oil=42.74g

$$Density = \frac{w \quad o\ o}{w \quad o\ W} = \frac{4\ .7\ -1\ .7}{4\ .0-1\ .7} = \frac{2\ .9}{2\ .2} = 0.9169\ gcm^{-3}$$

Refined sunflower:

Weigh of empty pycnometer=17.79g

Weigh of pycnometer +water=45.0g

Weigh of pycnometer+oil=42.76g

$$Density = \frac{4\ .7\ -1\ .7}{4\ .0-1\ .7} = \frac{2\ .9}{2\ .2} = 0.9177 gcm^{-3}$$

Crude peanut:

Weigh of empty pycnometer=25.86g

Weigh of pycnometer+water=74.43g

Weigh of pycnometer+oil=70.03g

$$Density = \frac{70.03-25.86}{74.43-25.86} = \frac{44.17}{48.57} = 0.9094 gcm^{-3}$$

Refined peanut:

Weigh of empty pycnometer=25.86g

Weigh ofpycnometer +water=74.43g

Weigh of pycnometer +oil = 70.59g

$$Density = \frac{70.59-25.86}{74.43-25.86} = \frac{44.73}{48.57} = 0.9209\ gcm^{-3}$$

Table 3.3: Shows the density of crude and refined oils of sunflower and peanut

The Oil	Density
crude sun flower	0.9169 gcm^{-3}
Refined sun flower	0.9177 gcm^{-3}
crude peanut	0.9094 gcm^{-3}
Refined peanut	0.9209 gcm^{-3}

3.1.4 Refractive index

Table 3.4:Shows therefractive index of crude and refined oils of sunflower and peanut

The oil	Reactive index
crude sun flower	1.468
Refined sun flower	1.467
crude peanut	1.468
Refined peanut	1.466

3.2Chemical tests

3.2.1Peroxide value: P.V

3.2.1.1Calculation:

P.V=V×N×1000/W

Where:

V=the volume of thiosulphate.

N=normality of sodium thiosulphate.

W=weigh of the sample.

For Sunflower Oil:

Crude:P.V=7.1×0.01×1000/5.09=13.94 meq.

Refined: P.V=1.5×0.01×1000/5.41=2.77 meq.

For Peanut Oil:

Crude:p.v = $\frac{1.6\times0.0\ \times1}{5.1} = 3.137\ m$

Refined:

p.v = $\frac{0.1\times0.0\ \times1}{5.0} = 0.2\ m$

Table 3.2.1: shows the peroxide values of crude and refined oils of sunflower and peanut

The oil	Peroxide value
crude sun flower	13.94 meq/kg
Refined sun flower	2.77 meq/kg
crude peanut	3.137 meq/kg
Refined peanut	0.2 meq/kg

3.2.2Free Fatty Acid:(F.F.A)

3.2.2.1Calculation:

F.F.A=V×N×28.2/W (sample).

Where:

V= volume of sodium hydroxide.

N=normality of sodium hydroxide.

28.2=factor.

W (sample) =weigh of sample.

For sunflower Oil:

Crude: F.F.A=3.3×0.1×28.2/7.28=1.27 %.

Refined: F.F.A=0.1×0.1×28.2/7.07=0.039 %.

For peanut Oil:

Crude: F.F.A=0.9×0.1×28.2/7.02=0.3615 %.

Refined:

F.F.A=0.2×0.1×28.2/7.04= 0.080 %

Table 3.2.2: Shows the free fatty acid of crude and refined oils of sunflower and peanut

The Oil	Free Fatty acid
crude sun flower	1.27%
Refined sun flower	0.04%
crude peanut	0.3615%
Refined peanut	0.08%

3.2.3Acid value

3.2.3.1Calculation:

Acid value=V×N×56.1/W (sample).

Where:

V=volume of sodium hydroxide.

N=normality of sodium hydroxide.

56.1=molecular weight of potassium hydroxide.

W (sample) =weight of sample.

For Sunflower Oil:

crude:1×0.1×56.1/2.27=2.4713 g/mol.

Refined: 0.1×0.1×56.1/2.73=0.2054 g/mol.

For Peanut Oil:

crude:1.2×0.1×56.1/3.55=1.8963 g/mol.

Refined: 0.1×0.1×56.1/2.81=0.1996 g/mol.

Table3.2.3: Shows the acid values of crude and refined oils of sunflower and peanut

The oil	Acid value
crude sun flower	2.4713 g/mol
Refined sun flower	0.2054 g/mol
crude peanut	1.8963 g/mol
Refined peanut	0.1996 g/mol

3.2.4Saponifiable number

3.2.4.1 Calculation:

Sponifiable number =V×N×56.1/W (sample).

Where:

V=volume of hydrochloric acid.

N=normality of hydrochloric acid.

56.1=molecular weight of potassium hydroxide.

W (sample) =weight of sample.

For Sunflower Oil:

crude:13.7×0.5×56.1/2=192.1425.

Refined: 13.3×0.5×56.1 /2=186.532.

For Peanut Oil:

crude:13.6×0.5×56.1/2=190.74.

Refined: 13.5×0.5×56.1/2=189.3375.

Table 3.2.4:Shows the saponifiable numbers of crude and refined oils of sunflower and peanut

The Oil	Saponifiable number
crude sun flower	192.1425
Refined sun flower	186.5325
crude peanut	190.74
Refined peanut	189.3375

3.2.5Unsaponifiable matter

Weight of crude sunflower ppt=0.16 g.

Weight of refined sunflower ppt=0.03 g.

Weight of crude peanut oil ppt=0.09 g.

Weight of refined peanut oil ppt=0.01 g.

3.2.5.1Calculation:

Unsaponifiable matter=w (ppt)/w (total sample)*100 .

For Sunflower Oil:

Crude: 0.16/2×100=8.

Refined: 0.03/2×100=1.5.

For Peanut Oil:

crude:0.09/2×100=4.5.

Refined: 0.01/2×100=1.

Table 3.2.5: Shows theunsaponifiable matter of crude and refined oils of sunflower and peanut

The Oil	Unsaponifiable matter
crude sun flower	8
Refined sun flower	1.5
crude peanut	4.5
Refined peanut	1

3.2.6 Fatty acid analysis :(see appendix)

Table (3.2.6): Shows the Fatty Acid composition of crude Sunflower Oil

Peak #	Cmpd Name	Area%
1	Undecanoicacid	0.3963
2	Tridecanoicacid	2.7035
3	Myristicacid	0.4355
4	Cis-10-Pentadecenoic Acid	20.6702
5	PalmitoleicAcid	67.8046
6	Linolelaidic Acid	0.3610
7	Elaidic Acid	0.9562
8	,Stearic Acid	1.8805
9	Arachidonic Acid	3.5917
10	ErucicAcid	1.0078
11	Tricosanoic acid	0.1927

Table (3.2.7):Shows the Fatty Acid composition of refined Sunflower Oil

Peak#	Cmpd Name	Area%
1	Undecanoic acid ME	0.0779
2	Tri decanoic acid M	0.5825
3	Myristic acid M.E	0.1396
4	Cis-10-pentadeceno	7.9634
5	Palmitoleic acid M.E	0.1993
6	Palmatic acid M.E	89.6607
7	Heptadecanioc acid	0.2822
8	Oleic acid M.E	0.0087
9	Stearic acid M.E	0.3510
10	Cis-8,11,14-Eicosath	0.918
11	Arachidonic acid M	0.5398
12	Erucic acid M.E	0.1030
Total		100.0000

Table (3.2.8):Shows the Fatty Acid composition of crude peanutOil

Peak#	Cmpd Name	Area%
1	Undecanoic acid ME	0.2243
2	Tri decanoic acid M	0.8616
3	Cis-10-pentadeceno	59.3641
4	Palmitoleic acid M.E	0.7531
5	Linolelaidic acid M	1.1275
6	Elaidic acid M.E	2.3556
7	Stearic acid M.E	11.1643
8	Cis-8,11,14-Eicosath	0.2619
9	Cis-11-Eicosath	16.6382
10	Heptadecanioc acid	0.1342
11	Erucic acid M.E	6.1132
12	Tricosanoic acid M.E.	1.0020
Total		100.0000

Table (3.2.9):Shows the Fatty Acid composition of refined peanutOil

Peak#	Cmpd Name	Area%
1	Tricosanoic acid M.E.	0.5157
2	Myristic acid M.E	0.1617
3	Cis-10-pentadecenoic acid M.E	12.3999
4	Palmitoleic acid M.E	0.4664
5	Palmatic acid M.E	83.0663
6	Linolelaidic acid M.E	0.5223
7	Stearic acid M.E	1.0281
8	Cis-11-EicosnoicM.E .Cis-11,14,1-	1.4202
9	Erucic acid M.E	0.4194
Total		100.0000

Discussion

The physicochemical properties of crude and refined oils of sunflower and peanut were studied.

The physical property of crude and refined sunflower oil is as follow:

Bleaching of the colour of the oil of sunflower was clearly noticed when the crude product was refined. The lovibond reading were decreased from 4.5 to 1.0 for red, from 0.9 to 0.0 for blue and from25 to 5 for yellow. Similarly the moisture content was reduced, from 0.07% to 0.02%.Likewise the refractive index, was decreased from1.468 to 1.467.The density, however, was slightly increased from 0.9169gcm^{-3} to0.9177gcm^{-3}.

The physical properties of crude and refined peanut oil is as follow:

Bleaching of the colour of the oil of peanut was clearly noticed when the crude product was refined. The lovibond reading were decreased from 3.6 to 1.0 for red, from 0.8 to 0.0 for blue and from25 to 4.2 for yellow. Similarly the moisture content was reduced, from 0.13% to 0.02%.Likewise the refractive index, was decreased from1.468 to 1.466.The density, however, was high increased from 0.909gcm^{-3} to0.920gcm^{-3}.

The chemical properties of crude and refined sunflower oil:
The peroxide value was decreased from 13.94meq/kg to2.77 meq/kg.Similarly the free fatty acid was decreased from 1.27% to0.04%, the acid value was decreasedfrom2.4713 to0.2054, the saponifiable number was decreased from 192.1425 to 186.5325 and the unsaponifiable matter decreased from 8 to 1.5.

The chemical properties of crude and refined peanut oil:

The peroxide value was decreased from 3.137meq/kg to0.2meq/kg.Similarly the free fatty acid was decreased from 0.3615% to0.08%, the acid value was decreased from 1.8963 to0.1996, the saponifiable number was decreased from 190.74 to 189.3375 and the unsaponifiable matter decreased from 4.5 to 1.

The fatty acid of crude sunflower oil which were detected were palmitolic acid,cis-10-pentadecenoic acid,arachidonicacid,tridecanoicacid,stearicacid,erucicacid,elaidicacid,myristicacid,undecanoicacid,linolelaidic acid and tricosanoic acidand their area%.

The area% were67.8046%,20.6702%,3.5917%,2.7035%,1.8805%,1.0078%,0.9562%,0.4355%,0.3963%,0.3610%,and 0.1927%respectively.

The fatty acid of refined sunflower oil which were detected were palmitolic acid,cis-10-pentadecenoic acid,arachidonicacid,tridecanoicacid,stearicacid,erucic acid, myristicacid,undecanoicacid,palmaticacid,heptadecanoicacid,oleic acid and cis-8,11,14-eicosatic acid and their area%.The area% were 0.4775%,20.1220%,1.6902%,2.0329%,0.7481%,0.3366%,0.3158%,0.2397%,72.1860%,1.29886%,0.2613% and 0.2914% respectively.

In crude sunflower oil; linolic, linolinc and oleic acid were not found, and in refinedonelinolic, linolinc acid were not found.In crude sunflower oil the contribution of palmitolic acid,cis-10-pentadecenoic acid,arachidonicacid,tridecanoicacid,stearicacid,erucicacid,elaidicacid,myristicacid,undecanoicacid,linolelaidic acid and tricosanoic acid were found to be high in comparisonwith refined one.

The fatty acid of crude peanut oil which were detected were palmitolic acid,cis-10-pentadecenoic acid,,tridecanoicacid,stearicacid,erucicacid,elaidic acid, undecanoic acid, linolelaidic acid ,tricosanoic acid,cis-8,11,14-eicosatic acid,cis-11-eicosenoic acid and heneicosanoicacid.The area % were1.6999%,50.0597%,2.3096%,11.3800%,8.9181%,2.5748%,0.4202%,1.0842% ,1.1639%,0.4105%,19.7005%and 0.2784% respectively.

The fatty acid of refined peanut oil which were detected were palmitolic acid,cis-10-pentadecenoic acid,,tridecanoicacid,stearic acid, erucicacid,linoleic acid ,cis-11-eicosenoic acid ,myristicacidandpalmaticacid.The area % were 0.4664%,12.3999%,0.5157%,1.0281%,0.4194%,0.5223%,1.4202%,0.1617% and 83.o663% respectively.

In crude peanut oil myristic, , palmitic, , oleic, linoleic, linolenic, arachidic,gadoleic and behenic acids were not found, in refined one oleic,linolenic, arachidic,gadoleic and behenic acids were not found.

In crude peanut oil the contribution of tridecanoic, cis-10-pentadecenoic, palmitolic, stearic, cis-11-eicosenoic and erucic acid were found to be high in comparison with refined one.

As long as the crude and refined peanut of sample shows no arachidic acid and peanut oil is famous for itsarachidic acid component, the sample may be forgered.

Recommendations

1. Research and Development work to increase the efficiency of the refining process.

2. Establishment of quality control laboratories equipped with essential instruments to analyze vegetable oils and their products.

3. Regular training of engineering, technological, scientific and technical staff.

4. Safetymeasures including adequate air circulation and ventilation in the different process of the plant should be adopted.

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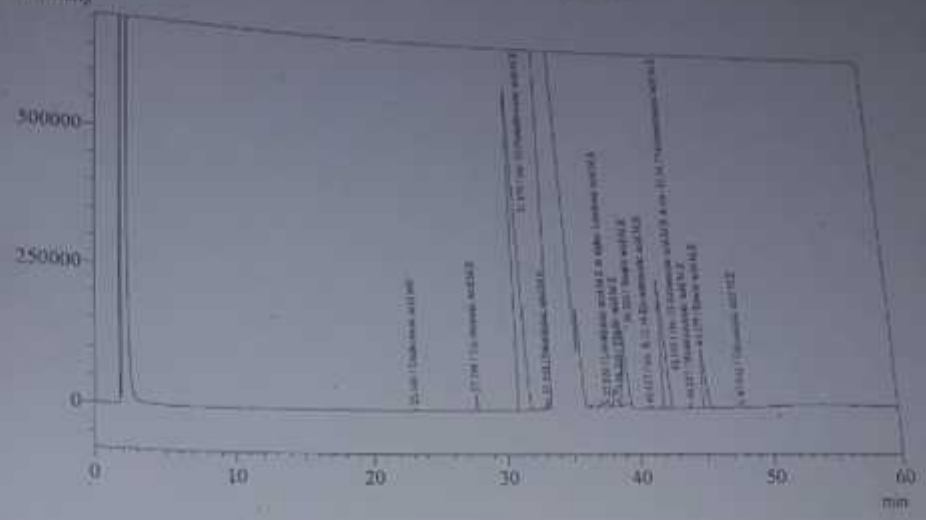
Appendices

Analysis Date & Time: 8/23/2013 2:17:37 PM
User Name: Admin
Vials: 1
Sample Name:
Sample ID:
Sample Type: Unknown
Injection Volume:
ISTD Amount:

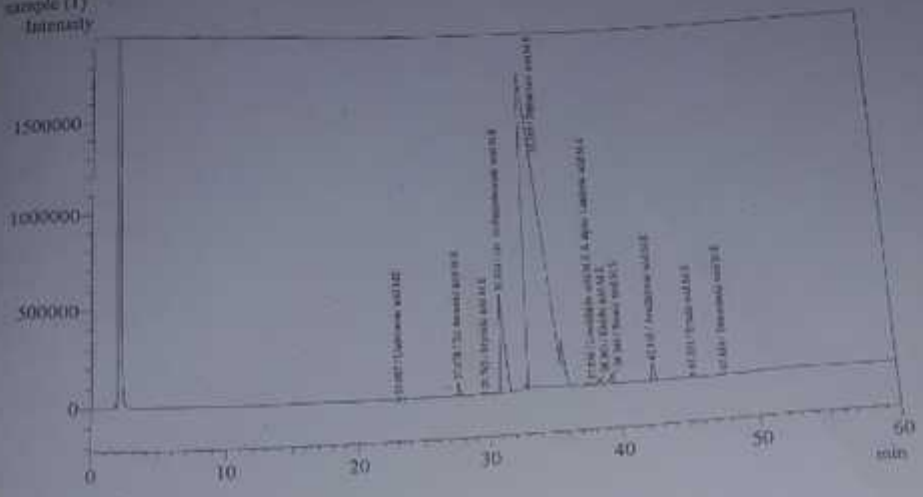
Data Name: D:\fatty acid analysis\Huda\HU1.gcd
Method Name: D:\fatty acid analysis\Huda\fatty acid analysis.gcm
[Description]
sample (3)



Peak#	Cmpd Name	Ret.Time	Area	Height	Area%	Height%
1	Undecanoic acid ME	23.140	64734	5233	0.2243	0.4202
2	Tri decanoic acid M	27.794	248722	28763	0.8616	2.3096
3	cis- 10-Pentadecenc	31.074	17136311	623432	59.3641	50.0597
4	Palmitoleic acid M E	33.259	217387	21170	0.7531	1.6999
5	Linolelaidic acid M	37.820	325466	13503	1.1275	1.0842
6	Elaidic acid M E	38.318	679971	32066	2.3556	2.5748
7	Stearic acid M E	39.226	3222736	141724	11.1643	11.3800
8	cis- 8,11,14-Eicosati	41.017	75615	5112	0.2619	0.4105
9	cis- 11-Eicosenoic a	42.141	4802866	245346	16.6382	19.7005
10	Heneicosanoic acid	44.017	38727	3468	0.1342	0.2784
11	Erucic acid M E	45.177	1764678	111064	6.1122	8.9181
12	Tricosanoic acid M	47.932	289240	14495	1.0020	1.1639
Total			28866453	1245376	100.0000	100.0000

Analysis Date & Time: 8/24/2015 11:58:10 AM
User Name: Admin
Vial#: 1
Sample Name:
Sample ID:
Sample Type: Unknown
Injection Volume:
ISTD Amount:

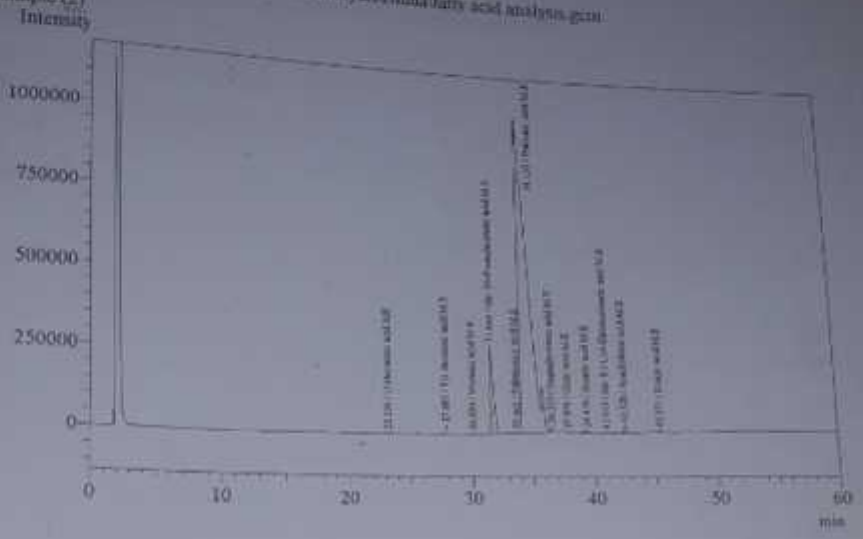
Data Name: D:\fatty acid analysis\Huida\SHT.gcd
Method Name: D:\fatty acid analysis\Huida\fatty acid analysis.gcm
[Description]
sample (1)



Peak#	Cmpd Name	Ret.Time	Area	Height	Area%	Height%
1	Undecanoic acid M.E.	23.087	96860	10066	0.0596	0.3963
2	Tri decanoic acid M.	27.678	702370	68671	0.4321	2.7035
3	Myristic acid M.E.	29.765	220207	11063	0.1355	0.4355
4	cis- 10-Pentadecenoi	31.024	11829502	525041	7.2769	20.6702
5	Palmitoleic acid M.E.	33.109	146209646	1722294	89.9412	67.8046
6	Linolelaidic acid M.	37.856	299854	9169	0.1845	0.3610
7	Elaidic acid M.E.	38.363	529087	24288	0.3255	0.9562
8	Stearic acid M.E.	39.364	844189	47768	0.5193	1.8805
9	Arachidonic acid M.	42.410	1323466	91232	0.8141	3.5917
10	Erucic acid M.E.	45.331	373208	25600	0.2296	1.0078
11	Tricosanoic acid M.	47.660	132910	4894	0.0818	0.1927
Total			162561299	2540086	100.0000	100.0000

Analysis Date & Time: 8/23/2015 1:12:23 PM
User Name: Admin
Vial#: 1
Sample Name:
Sample ID:
Sample Type: Unknown
Injection Volume:
ISTD Amount:

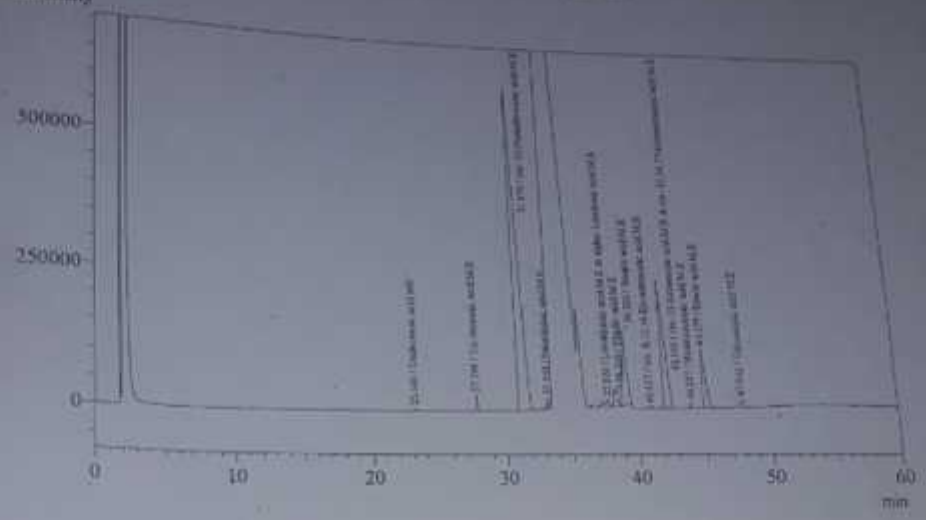
Data Name:
Method Name: D:\fatty acid analysis\Hexdr\SH.gmt
[Description]: D:\fatty acid analysis\Hexda\fatty acid analysis.gcm
sample (2)



Peak#	Cmod Name	Ret Time	Area	Height	Area%	Height%
1	Undecanoic acid ME	23.150	47838	3479	0.0779	0.2397
2	Tri decanoic acid M	27.885	357657	29502	0.5825	2.0329
3	Myristic acid M.E	30.080	85738	4583	0.1396	0.3158
4	cis- 10-Pentadecenc	31.644	4889497	292026	7.9634	20.1220
5	Palmitoleic acid M.I	33.662	122367	6929	0.1993	0.4775
6	Palmatic acid M.E	34.132	55051257	1047618	89.6602	72.1860
7	Heptadecanoic acid	36.525	173239	18846	0.2822	1.2986
8	Oleic acid M.E	37.879	5362	3793	0.0087	0.2613
9	Stearic acid M.E	39.470	215543	10856	0.3510	0.7481
10	cis- 8,11,14-Eicosat	41.013	56353	4229	0.0918	0.2914
11	Arachidonic acid M	42.528	331443	24530	0.5398	1.6902
12	Erucic acid M.E	45.371	63271	4885	0.1030	0.3366
Total			61399565	1451276	100.0000	100.0000

Analysis Date & Time: 8/23/2013 2:17:37 PM
User Name: Admin
Vials: 1
Sample Name:
Sample ID:
Sample Type: Unknown
Injection Volume:
ISTD Amount:

Data Name: D:\fatty acid analysis\Huda\HU1.gcd
Method Name: D:\fatty acid analysis\Huda\fatty acid analysis.gcm
[Description]
sample (3)



Peak#	Cmpd Name	Ret.Time	Area	Height	Area%	Height%
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9	cis- 11-Eicosenoic a	42.141	4802866	245346	16.6382	19.7005
10	Heneicosanoic acid	44.017	38727	3468	0.1342	0.2784
11	Erucic acid M E	45.177	1764678	111064	6.1122	8.9181
12	Tricosanoic acid M	47.932	289240	14495	1.0020	1.1639
Total			28866453	1245376	100.0000	100.0000