

REFINING OF SOYA BEAN OIL

BY

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CHE/2007/060**

**DEPARTMENT OF CHEMICAL ENGINEERING
FACULTY OF ENGINEERING
CARITAS UNIVERSITY AMORJI-NIKE
ENUGU STATE**

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TITLE PAGE

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CHE/2007/060**

A PROJECT WORK SUBMITTED TO THE DEPARTMENT

OF

**CHEMICAL ENGINEERING, FACULTY OF ENGINEERING,
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SUPERVISOR: ENGR. (MRS) V.C OTEGBULU

AUGUST, 2012

CERTIFICATION

This is to certify that this project report was submitted by **OKWOR BLESSING TOCHI** with registration number **CHE/2007/060** to the Department of Chemical Engineering, Faculty of Engineering, Caritas University Emene-Enugu state in partial fulfillment of the requirement for the award of Bachelor of Engineering degree (B. ENG.) in Chemical engineering.

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Date

DEDICATION

This work is dedicated to God Almighty who is the author and finisher of my life and to my family for their love, care and support all the way.

ACKNOWLEDGEMENT

Am grateful to God who has been so faithful to me, for his mercies upon me all the days of my life.

I want to say a great thank you to all who contributed to the success of this work.

To my supervisor Engr (Mrs) V.C. Otegbulu who did not relent until the end of this work, you are indeed a mother, God bless you madam.

To my parents Ikenge and Mrs. Canice Okwor and my sibilings Ngozika, Benard, Rita purity, Nonyelum and Okechukwu for their love, care and support all the way.

I will forever remain grateful to my lecturer whom their efforts brought me this far, people like prof. J.I. ume (HOD), Engr Ken Eze, Engr G.O Mbah, Engr (Mrs) N. Odilinye, Engr B. Ugwu and all other lecturers.

I can never forget you Hon. Davidson Aneke my Guidian angel, Mr. Chibueze Ihegazie, one in a million and Mr. Ndubuisi Peter for being there.

To all my friends who have in one way or the other contributed to the success of this work, people like Kenechi, Bright, Onyinyechi, Ukamaka, Ify and others. I am grateful to you all.

ABSTRACT

This project work studied the refining of crude soya bean oil extracted from soya bean seed using alkali /caustic refining method. The work was carried out using phosphoric acid for the degumming /pretreatment process and sodium hydroxide for the neutralization /refining of the oil. Certain tests were carried out on both the crude and refined oil such as saponification value, acidic value, iodine value, specific gravity and viscosity, the results obtained after the tests include 42.075, 164.28, 589.05, 0.8944 and 0.038 respectively for refined oil and 44.88, 109.52, 448.8, 0.8148 and 0.035 respectively for the crude oil.

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CHAPTER ONE

1.1 INTRODUCTION

Refining of vegetable oils is essential to ensure removal of germs, phosphatides and free fatty acids (F.F.A) from the oil, to impact uniform colour by removal of colouring pigments and to get rid of unpleasant smell from the oil by removal of odiferous matter.

Refining is carried out either on batch operation or as continuous operation. With certain oils even physical refining can be carried out instead of chemical.

For processing less than thirty tones of oil per 24 hours, and when oil has F.F.A content of 1 percent or less normally batch process is recommended. Batch process involves low capital investments, simplicity of operation and low maintenance, making refining economically a viable proposition even at capacity as low as 10 tonnes per 24 hours. (According to Dietary fats and oils in Human Nutrition. (Rome 1977)).

Soyabean oil is produced from the seed of the legume called soja max or calyclue max. The seed has an oil content of about 20%, it is the highest volume vegetable oil produced in the world. The crude oil is obtained by pressing or solvent extraction method. The main uses of the oil after refining, bleaching and deodorization and partial hydrogenation are in the manufacture of Magrine and shortening. The unhydrogenated oil is also used in blends with other oil but its tending to revert when exposed to air or higher temperatures limits its use. (Hand book of industrial chemistry, Reigel et al, (2003)).

Soyabean oil is also used extensively in the manufacture of drying oil products.

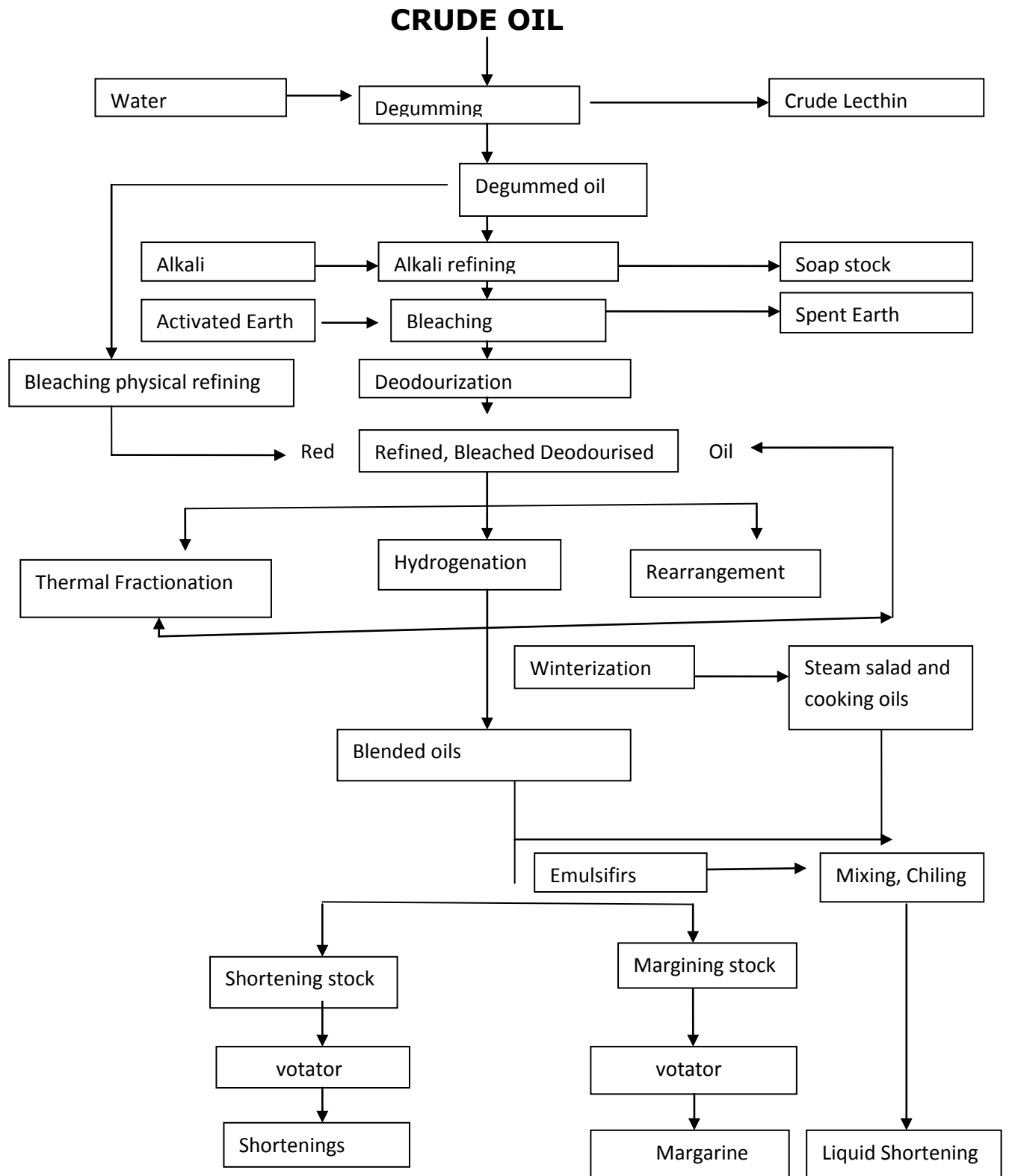
Crude soyabean oil of good quality has a lighter amber colour which upon alkali refining is reduced to the light yellow colour of most vegetable seed oils. Soyabean oil produced from green or immature beans may contain sufficient chlorophyll to have a greenish cast but this is not usually very evident until after the yellow red pigment of the oil have been bleached in hydrogenation (G.S Breck and S.C Bhatia, 2008).

The crude oil particularly that obtained by solvent extraction contains relatively large amount of non-glyceride materials consisting chiefly of phosphatide. They are removed by water washing during refining processes. The phosphatides removed by water washing are converted to soya lecithin. The free fatty acid content of good crude soyabean oil like that many other vegetable oil is slightly in excess of 0.5 percent. (Hand book of Industrial chemistry, Reigel et al (2003)).

1.3 BACKGROUND OF THE STUDY

Crude fats and oils are processed by general scheme shown below with modifications or exceptions for specific species.

Fig 1:1-Generation flow sheet for refining and processing fats and oils (according to G.S Breck and S.C Bhatia, 2008).



The phospholipids (Lecithins) must be removed to avoid darkening of the oil during high temperature deodourization and in deep-fat- frying applications. This removal typically is accomplished during the alkali refining process or in a separate water/acidic water degumming step before alkali refining. Crude soyabean oil has an unusually high (2-3.5 percent) phospholipid content among oils and often is degummed in a separate operation to not more than a 300 ppm level (as phosphorus) to avoid precipitation during shipping and storage. Refined soyabean oil contains 10ppm or less phospholipid. Degumming is achieved by mixing crude soyabean oil with water to hydrate the phospholipids and enable their removal by centrifuge. Citric and other acids sometimes are added in a step called super degumming to help remove phospholipids that are not hydrated by water. Degummed soyabean oil or crude oils of other species are neutralized with sodium hydroxide solution to form sodium salts of the fatty acids which are removed as soap stock by a continuous centrifuge. The soapstock also includes remaining phospholipids, some colour and flavor compound. (Hand book of industrial chemistry, Reigel et al (2003)).

The soap stock can be dried if refining is done adjacent to an extraction plant or acidified again to remove fatty acids and sold to the olechemical industry. The oil is then water washed and centrifuge one or two times to remove residual soaps.

According to GS Breck and S.C Bhatia, a total degumming process for removing essentially all the phosphatide from soyabean oil using first an acid and then an alkali and two centrifuges has shown higher yields than conventional refining. This process however, does not remove prooxidant metals efficiently and for this reason has not found commercial acceptance in the united state.

G.S Breck and S.C Bhatia have stated that Dijkstra has described a novel process where the washing water is recycled to the oil feed and use to dilute concentrated alkali. This process does not generate an aqueous effluent and can be used for both acid and alkali refining, thus allowing refiners to change gradually from alkali refining to physical refining. Neutralization of soyabean oil with alkali solution assures elimination of free fatty acids without notable change in the phosphatide content. The phosphatidic

concentration obtained from oil previously neutralized in the miscella was of higher quality than the phosphatidic concentration obtained from the oil of the starting miscella. Aqueous ammonia has the advantage of being safe for the environment because the deacidification agent can be repeated or reused. Oils especially soyabean oil with low degree of oxidation can be fully deacidified only with the help of the ammonia. The same effect can frequently be achieved by a preliminary desliming with 5 percent formic or citric acid. Deodourization at 210⁰c of oils that have been deacidified with ammonia and washed with water yield bland and pale edible oils having good storability (G.S Breck and S.C Bhatia).

List and Erickson state that of all the unit processing operations, refining has the most significant effect on oil quality measured by colour, oxidative stability and storage properties.

If soyabean oil is not properly refined, subsequent processing operation such as bleaching, hydrogenation and deodourization will be impaired so that finished products will not fail to meet quality standards. Also, poor refining will reduce the yield of

natural oil, thereby lowering manufacturing profits. (JAOCS, Vol. 60).

According to G.S Breck and S.C Bhatia, caustic refining removes free fatty acid to 0.01-0.03percent level and remove virtually all the phosphatides. Crude soyabean oil contains trace amount (several part per million (ppm)) of prooxidant metals such as iron and copper. Caustic refining usually removes 90-95 percent of these metals. However, it should be emphasized that even though caustic refining reduces metallic contamination to low levels, residual iron and copper still remain strong prooxidants in refined oils and must be taken in to account during storage and handling. At a constant percentage of water, the total amount of caustic used influences colour removal ie the more caustic used, the lower the colour of the refined oil.

List and Erickson reported that plots of residual iron versus residual phosphorus content of deodourized oil showed that iron increases at phosphorus content below about 1ppm, reaches a constant value of about 2-20ppm phosphorus, then beings to increase. Thus, the decreased oxidative to stability at phosphorus

content above 20ppm can be explained by the sufficiently high iron content (ie greater than 0.2 ppm) which exerts a strong prooxidant effect. Similarly, decreased stability at phosphorus content below 2ppm can also be explained because of the increased iron content. At the same time, it should also be pointed out that the traditional method for calculating the amount of refining lye is based on the free fatty acid content and therefore gives no indication of conditions leading to optimum phosphorus removal. Phosphatide content generally exceed that free fatty acids in crude soyabean oil by a factor of about 6. In refining process control, crude oil is usually evaluated for refining cost by the American oil chemist's society (AOCS) chromatographic method. (JAOCS, vol 60).

1.3 STATEMENT OF THE PROBLEM

In the market today, most vegetable oils solidify at a low temperature of less than 25⁰c. This work is to process and refine edible and quality soyabean oil that will not undergo solidification at a low temperature.

1.4 OBJECTIVES OF THE STUDY

The objective of refining and processing fats and oils include:

- Removal of free fatty acids, phospholipids (gums) colour and off-flavour/odour compounds and toxic substances to produce light- coloured and bland products with long shelf lives.
- Obtaining a mixture of the triacyl-glycerols with the desired solid content profiles over the range of product use.
- Preparation and storage of semi-solid products with desired textures.

1.5 SCOPE OF THE STUDY

The crude oil extracted from soyabean needs further treatment to convert it to a bland, stable, nutrition products that is used to manufacture margarine, shortening, salad and cooking oil, mayonaise, food products, Olechemicals.

This study entails the process of producing good quality oil through caustic/alkali refining process which is going to be

compared with other good quality products in the market like grand product etc.

CHAPTER TWO

2.0 LITERATURE REVIEW

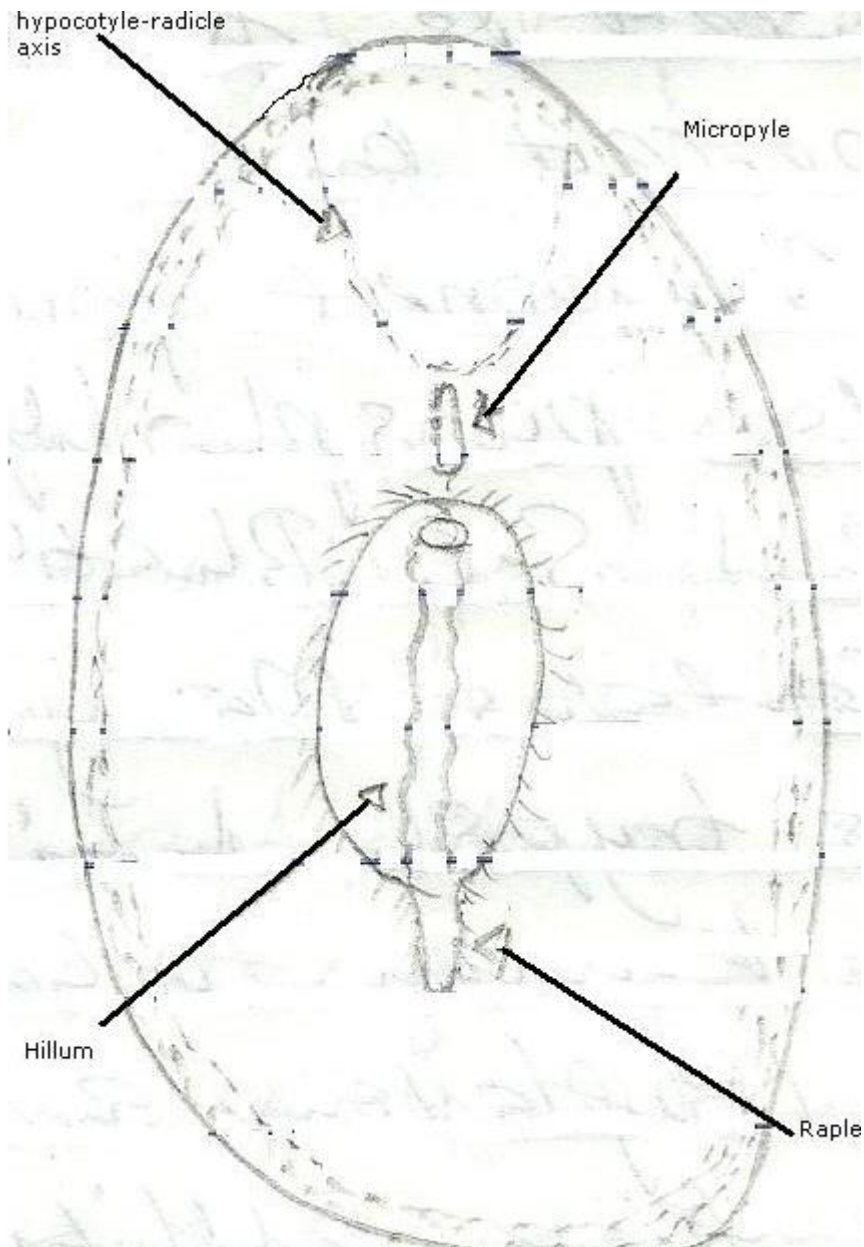
2.1 HISTORICAL BACKGROUND OF SOYABEANS

For centuries, soyabean has been used by Chinese, Japanese, Korean and south-east Asian, people in various forms as one of the most important sources of dietary protein and oil.

The soyabean (botanical name: *Glycine Max (L) Merrill*) is believed to have originated in China and one of the oldest crops of the far east. (G.S. Breck and S.C. Bhatia, 2008). After flowering, the first stage in the formation of fruit is followed by the development of a pod containing from 1-4 seeds.

Like other leguminous crops, the soyabean seed is essentially composed of a smooth envelop (hull) and an embryo. Generally, its weight (depending on the variety) is between 50 and 40mg, the shape of the seed varies with the cultivars

Fig 2:1 Front View of Soyabean Seed



(Hand Book of Industrial Fats and Oil, vol 2 G.S Breck And S.C Bhatia, 2008)

The amount of protein produced by soyabeans per unit land area is higher than that of any other crop.

According to G.S Breck and S.C Bhatia, 2008. This little bean has been called "meat of the field", "nature's miracle protein" and "yellow jewel". In recent decades soyabeans has generated great interest as a weapon against hunger, a protein of the future and recently as a possible weapon against chronic disease.

2.1.1 COMPOSITION OF SOYABEAN

Among cereals and other legume species soyabean has the highest protein content (around 40 percent), other legumes have a protein content between 20-30 percent where as cereals have a protein content in the range of 8-15 percent oil. The soyabean also contains about 20 percent oil, the second highest content among all food legumes. (the highest oil content is found in peanut which is about 45percent on the dry matter basis) other valuable component found in soyabean include minerals, phospholipids and vitamins. (G.S Breck and S.C Bhatia, 2008). Soyabean contains many other minor substances such as trypsin inhibitors, phytates and oil gosacherides known to be biologically active. Others such as isoflavones are beginning to be recognized for their ability to prevent human cancer and other diseases.

During processing, components extracted from soyabean by organic solvent such as hexane are called crude oil. Major components of crude oil are trigly cerides, minor components includes phospholipids, unsaponifiable material consisting of tocopherol, phytosterols and hydrocarbons. The concentrations of these minor compounds are reduced after processing the oil in the visual way (G.S Breck and S.C Bhatia, 2008).

2.1.2 SOYA CHEMISTRY

Despite its possible usefulness, According to G.S Breck and S.C Bhatia, 2008, this wonderful bean suffers an image problem for its unfamiliar flavor and taste. Only a small protein of annual soyabean production is processed into traditional soya foods. Most of the remaining protein is crushed into oil and defatted meal. Although the oil is used manly for human consumption, only a small protein of protein rich meal is further processed into protein products for food ingredients and the remaining large protein goes as animal feed.

However, the use as value- added protein food ingredients, as industrial paints to table oils and spreads, is increasing. This

has resulted to the development of a new type of soyabean crop with wide region of adoptability, herbicide tolerance, post resistance and altered chemical compositions. Soyabean has emerged for limited regional cultivation to expanded world wide production (G.S Breck and S.C Bhatia, 2008)

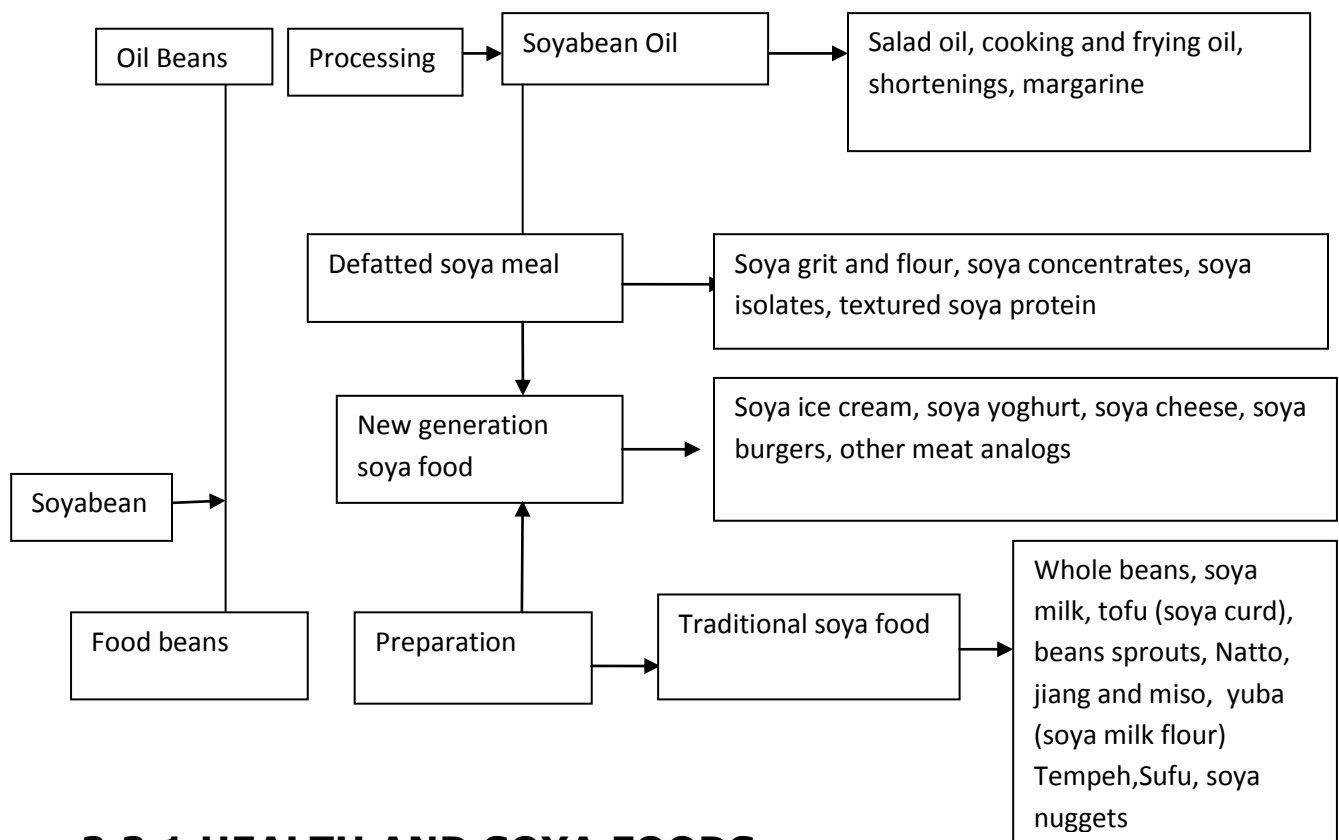
2.2. FOOD BEANS AND OIL BEANS

In the Far East, traditionally, soyabeans are made into various foods for human consumption including tofu. Soyamilk, soya sprouts, misonatto and tempeh. In the west, most soyabeans are extracted for oil and defatted meal. Although soya oil is wholly for human consumption, the cake is mainly used as animal feed. Only a small protein as mentioned earlier is processed into soya protein ingredients like soya flour, concentrates, isolates and textured soya protein. These ingredients in their turn have functional and nutritional application in various types of bakery, dairy and meat products infant formulas and so-called new generation of soya food. (G.S Breck and S.C Bhatia, 2008).

Because of this difference in soyabean use, two different types of soyabean have emerged: food beans and oil beans. Oil beans include all the commonly produced soyabeans.

Food beans on the other hand have been selected and bred over the past several decades for direct food consumption. They do not differ fundamentally from field varieties (oil beans) except that they generally have lighter seed coat, clear hillum, higher protein content, lower oil and yield (G.S Breck and S.C Bhatia, 2008).

Fig 2.2 a general outline of the soyabean food use based on classification of oil and food Bean (G.S Breck and S.C Bhatia, 2008)



2.2.1 HEALTH AND SOYA FOODS

Though soyabeans and soya foods have been primarily identified with their protein contents, during the past decades there has been much interest among researchers in the potential role of soya foods in preventing and treating chronic disease. This recent upsurge in interest in soyabean is due to the presence of isoflavones in soyabean. Isoflavones have been shown to inhibit the growth of cancer cells and bone resorption and to lower cholesterol. Recent understanding of biological activities of

isoflavones in soya foods has established the relationship between them and the risk and treatment of chronic disease like cancer, heart and kidney disease and osteoporosis. (G.S Breck and S.C Bhatia, 2008).

Soyabean oil is the most important vegetable oil because of its high quality and low cost. According to G.S Breck and S.C Bhatia, 2008; soyabean oil has many advantages but also some disadvantage compared to other vegetable oils.

The advantages include:

- ❖ A high level of unsaturation is present
- ❖ The oil remains liquid over a relatively wide temperature range.
- ❖ It can be hydrogenated selectively for bleaching with semisolid or liquid oils.
- ❖ When partially hydrogenated, it can be used as a pomable semisolid oil.
- ❖ Phosphatides, trace metals and soap in soyabean oil can be removed without much difficulty to obtain a high quality product.

- ❖ The presence of naturally occurring antioxidants (tocopherols) which are not completely removed during processing also contribute to its stability.

The disadvantages includes

- ❖ Phosphatides are present in relatively large amount (about 2 percent) and must be removed by processing.
- ❖ Soyabean oil contain relatively high level (7-8 percent) of linolenic acid which is responsible for its flavor and odour.

Some of these disadvantages are offset, however, in that the recovered “gums” are the source of commercial lecithin and by partial and selective hydrogenation of the oil the linolenic acid concentration can be lowered to below 3 percent with the result of greatly improved stability. (G.S Breck and S.C Bhatia, 2008).

2.3. COMPOSITION OF SOYABEAN OIL

The soyabean oil has the following approximate composition

Table 2.1 composition of soyabean oil. (According to G.S Breck and S.C Bhatia, 2008).

Composition	percentage
Protein	40percent
Lipid	20percent
Cellulose and hemi cellulose	17percent
Sugar	7percent
Crude fibre	5percent
Ash (dry weight basis)	6percent

Average compositions of crude and refined soyabean oil are given in table below. The refining processes does not affect acid composition of the glyceride, it only removes most of the free fatty acids and colour bodies and lowers the levels of some of the lesser constituents such as the tocopherols by 31-47 percent, the sterol by 25-35 percent and squalene by 15-37 percent.

Table 2.2: average composition for crude and refined soyabean oil (courtesy of G.S Breck and S.C Bhatia, 2008).

Component (%)	Crude oil	Refined oil
Triglyceride	95-97	>99
Phosphatides	1.5-2.5	0.003-0.045 ^a
Unsaponifiable matter	1.6	0. 3
Plant sterol	0.33	0.13
Hydrocarbon squalene	0.014	0.01
Tocopherols	0.15-0.21	0.11-0.18
Free fatty acids	0.3-0.7	<0.05
Trace metals		

Iron (ppm)	1.3	0.1-0.3
Copper (ppm)	0.03-0.05	0.02-0.06

^aCorresponding to 1-15 ppm of phosphorus

Triglycerides constitute more than 95 percent of the lipidic compounds of the soyabean seed its other compounds being phospholipids and diacylglycerol.

2.3.1 TRIGLYCERIC STRUCTURE

Because of the high unsaturated fatty acid content of soyabean oil, nearly all the glyceride molecules contain at least two unsaturated fatty acid and di and tri-saturated glycerides are essentially absent. The fatty acids are not randomly distributed in a single glyceride molecule and the glyceride is biosynthesis with a definite configuration. Each hydroxylgroup of glycerol is unique and has been assigned a stereospecifically numbered (sn) position. (G.S Breck and S.C Bhatia, 2008).

2.3.2 PHYSICAL PROPERTIES OF SOYABEAN OIL

Physical properties of soyabean oil depend on climate and variety. Processing the oil has an influence on the presences and level of the many minor constituents. Important physical

properties of soyabean oil are summarized in table 2.3 below. The values reported are not absolute because the compositions of many of the properties depend on the degrees of unsaturation and other factors. The densities of soyabean oil at several temperatures are given in table 2.4 below. The densities of oils depend on temperature; they have inverse relationship with molecular weight and a direct one with the degree of unsaturation. (G.S Breck and S.C Bhatia, 2008)

Lund has developed an equation based saponification and iodine values of vegetable oils to predict their specific gravity as follows: $\text{specific gravity} = 0.8475 + 0.00030 (\text{saponification value}) + 0.00014 (\text{iodine value})$.

Table 2.3 selected physical properties of soyabean oil (according to G.S Breck and S.C Bhatia, 200)

Property	Value
Specific gravity, 25 ⁰ c	0.9175
Refractive index, n ²⁵ _D	1.4728
Specific refractive, r ²⁰	0.3054
Viscosity, centipoises at 25 ⁰ c	50.9
Solidification point (°c)	-10 to -16
Specific heat (cal/g)	9478
Smoke point (°c °f)	234 (453)
Flash point (°c °f)	328 (685)
Fire point (°c °f)	363 (685)

Table 2.4 density of soyabean at several temperatures (By G.S Breck and S.C Bhatia, 2008).

Temperature °c	Density (g/ml)^a
-10.0	0.9410
0.0	0.9347
10.0	0.9276
25.0	0.9175
37.8	-
40.0	0.9075
50.0	-

^acommercial edible soyabean oil having an iodine value of 132.6 and containing 0.10percent free fatty acid.

G.S Breck and S.C Bhatia, 2008. Also state that smoke, flash and fire points to a major extent are functions of the free fatty acid content in soyabean oil because the fatty acids are much more volatile than the glycerides. The temperatures for these properties are generally similar for all vegetable oils having comparable fatty acid composition and low free fatty acid content.

Table 2.5 smoke, flash and fire points' temperatures of soyabean oil (courtesy of G.S Breck and S.C Bhatia, 2008).

SOYABEAN OIL

Description	Free Fatty Acid Content (%)	Smoke point (°c (°f))	Flash point(°c (°f))	Fire point (°c (°f))
Refined and bleached	0.010	234(453)	328 (623)	363(685)
Refined and bleached	0.010	228 (453)	329 (625)	363 (685)
Crude expeller pressed	0.51	185 (365)	296 (565)	349 (660)

2.4. RECOVERY OF OIL FROM SOYABEAN

After pretreatment, the oil is extracted from the soyabean. The three most common procedures are:

Hydraulic pressing, expeller pressing and solvent extraction. The most prevalent extraction method today in modern processing facilities is solvent extraction. There are four parts to the preparation of extraction process:

- a) Storage and preparation of beans
- b) Extraction of oil from bean

c) Reclamation of solvent from oil and meal

d) Oil storage (By G.S Breck and S.C Bhatia, 2008).

2.4.1 STORAGE AND PREPARATION OF BEANS

Before they are ready for processing, the beans are cleaned dried to about 10percent moisture and preferably conditioned by storage for about 10days to facilitate dehulling. For optimum operation of the solvent extraction process, 9.5-10percent moistures is described and preparatory flaking is accomplished most satisfactory at 74-79^ac (according to G.S Breck and S.C Bhatia, 2008)

2.4.2 SOLVENT EXTRACTION

Soyabean oil is miscible with many organic solvent, particularly those that are apolar and aprotic i.e. those that do not form hydrogen bonds.

According to G.S Breck and S.C Bhatia, 2008. hydrocarbons, esters, ethers, ketones and chlorinated solvents are miscible with soyabean oil. Ethanol, isopropyl and isobutyl-alcohols,

ethylenedichloride, trichloroethylene, carbontetrachloride and hexane have been examined in the laboratory as potential extraction solvents. Hexane is the preferred solvent for the commercial extraction of oil from soyabean flakes.

Furfural and soyabean oil are not miscible at normal temperatures and their mixtures separate into two phases: the solvent- predominant phase containing more the unsaturated glyceride and the oil- predominant fraction containing the more saturated glycerides (G.S Breck and S.C Bhatia, 2008).

According to G.S Breck and S.C Bhatia, 2008, trichloroethylene (TCE) Boiling at 86.7°C was applied many years ago as a safe nonflammable solvent for soyabeans; however, serious toxicity problems arose from the extracted oil meal when it was fed to cattle. While acetone and methyl acetone are miscible with soyabean oil at low temperature, the more saturated glyceride crystallise leaving, the more unsaturated glycerides in solution, this makes their use in a commercial operation impractical.

Anhydrous or aqueous ethanol is into a good solvent for soyabean oil at normal temperatures. As the temperature increases,

however, the solubility also increases until the critical solution temperature is reached at which point the components become miscible. Methanol is a poor solvent because of its polarity (G.S Breck and S.C Bhatia, 2008).

2.4.3 OIL STORAGE

With limited access to air, crude soyabean oil can be stored in large tanks for a longtime. It is recommended that the oil be cooled to near ambient temperature before being pumped to storage. Crude oil in storage should be clean and low in moisture in order to prevent hydrolysis. Crude oils high in phosphatides deposit a layer of gumming material upon long standing in tanks or during long shipment in tank cars. (G.S Bleck and S.C Bhatia, 2008).

2.5. EDIBLE OIL PROCESSING

The extracted crude oil need further treatment to convert it to a bland, stable and nutritious product because crude soyabean oil contains small and variable amount of non glyceride impurities impacting on the quality of the oil. These impurities also have an

effect on the processing behavior and yield of the finished product.

The impurities are of two general types:

- Oil- insoluble and
- Oil soluble.

The insoluble impurities consists of seed fragments, excess moisture and a waxy fraction that appears in refrigerated oil, thus making it appear turbid or cloudy. Some of these impurities such as seed fragment and meal fines are normally removed by filtration.

The oil soluble impurities are more difficult to remove. They include free fatty acids, phosphatides, gummy or mucilaginous substances, colour protein or protein fractions, tocopherols, sterols, hydrocarbons, ketones and aldehydes. some of the non glyceride constituents are useful.

The tocopherols for example, help protect the oil from oxidation. The sterols being odourless, tasteless, colourless and heat stable also are detrimental to oil quality. The other impurities however,

should be removed because they adversely affect the flavor, odour, colour, heat stability and storage stability of the oil. (According to G.S Breck and S.C Bhatia, 2008).

2.5.1 BASIC PROCESSING OPERATIONS AND PRINCIPLE EDIBLE OIL PRODUCT

The steps followed for basic oil processing include: crude oil storage, degumming and/or caustic refining, bleaching, hydrogenation and or winterization, deodourzation and finished oil storage. (By G.S Breck and S.C Bhatia, 2008).

Each step removes specific components from the oil. The specific components removed from the oil during these steps may be classified as follows:

- 1) Naturally occurring:-gums, phospholipide, prooxidant metals, pigments, colour bodies, tocopherols and free fatty acids.
- 2) New compounds formed during processing: - soaps, oxidation products, hydroperoxides, polymers and their decomposition products, colour bodies, isomers and high melting triglycerides.

3) Processing adjuncts: - hydrogenation catalysts bleaching clay and metal scavengers that precipitate from finished oils ie citric acid.

4) Contaminants introduced by processing: - moisture, trace metals, carbonaceous and oil-insoluble materials.

All soyabean oil processing steps consists of independent operations that are usually not connected with each other by continuous flow. Thus, crude soyabean oil is converted into a quality edible oil by a series of refining operations (G.S Breck and S.C Bhatia, 2008).

2.5.2 OIL DEGUMMING AND SOYABEAN LECITHIN

The degumming step removes the phosphatide and mucilaginous gums that when hydrated becomes insoluble in the oil. Free fatty acids, colour bodies and metallic prooxidants are removed to a varying degree in the alkaline refining step. Although the crude soyabean oil may or may not be degummed prior to caustic refining, the removal of nearly all the phospholipids is essential to produce a finished oil of optimum

colour, flavor oxidative stability. (G.S Breck and S.C Bhatia, 2008).

Water degummed oil from damaged beans may have an abnormally high phosphorus level. The non hydratable phosphatides are believed to be calcium and magnesium salts and may not all be removed by alkali refining either. In a commercial operation, crude soyabean oil is mixed with 1-3 percent of water based on the oil volume and the mixture agitated mechanically. Care being taken to avoid air entrainment for 30-60 minutes at about 70⁰c (158⁰f) to hydrate the phosphatide and other colloidal impurities. The hydrated phosphatides and gums can be separated from the oil by settling, filtering or centrifuging (G.S Breck and S.C Bhatia, 2008).

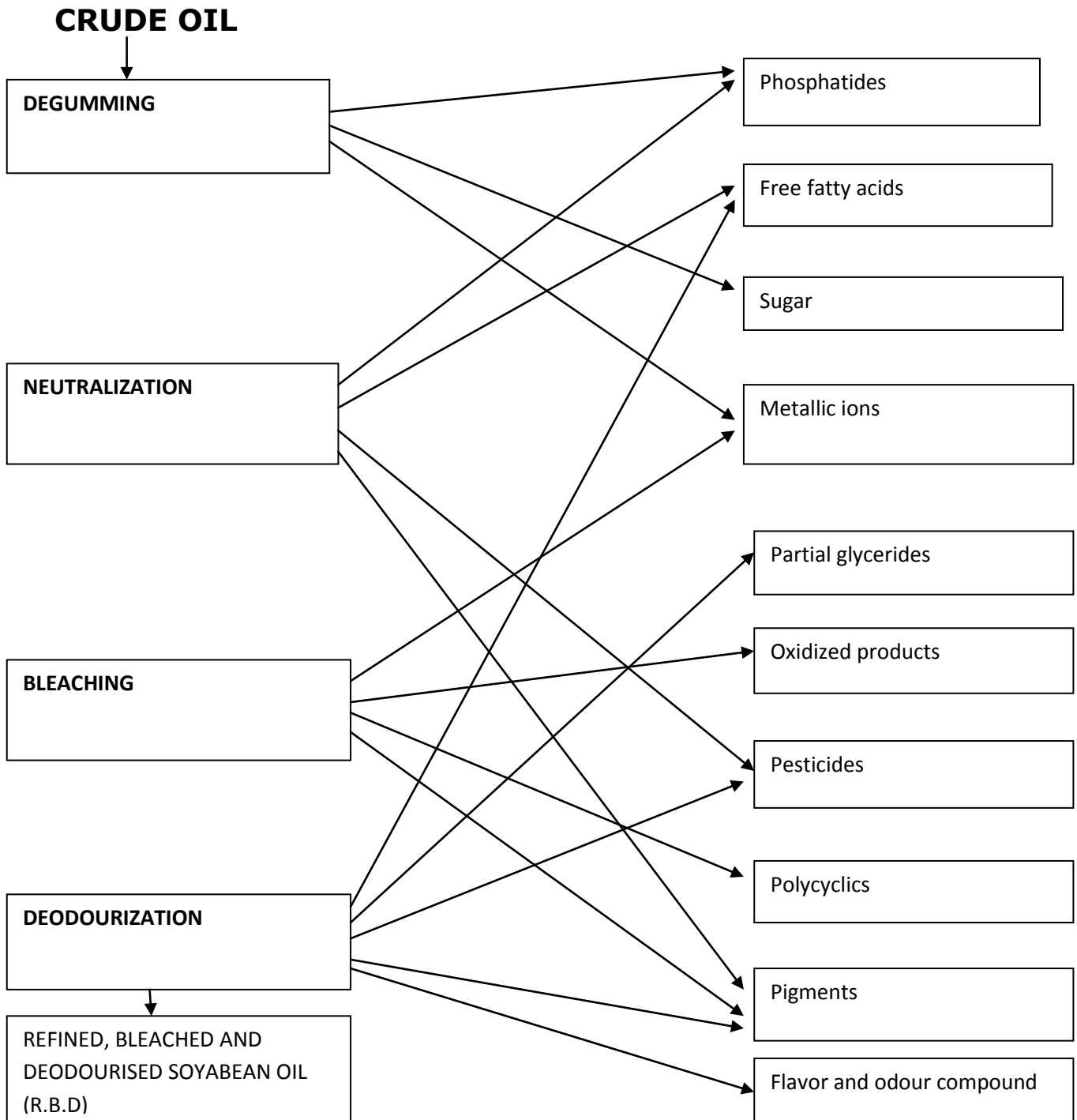
A combined treatment with citric acid, sodium silicate and silica absorbents has been described for processing crude and water degummed oils to over come difficult separation problems normally required a large number of centrifuges to achieve good separation of the hydrated phospholipids from the oil. Using this treatment, oils of good quality are obtained by providing

enhanced phospholipids' removal without the generation of an aqueous effluent. (G.S Breck and S.C Bhatia, 2008).

2.6. REFINING OF SOYABEAN OIL

Almost all edible soyabean oil can be refined with alkali, usually caustic soda, in a continuous processing system. As discussed later, steam refining or physical refining which is similar to steam deodourzation is another method of refining for oils where the impurities are primarily free fatty acids. (G.S Breck and S.C Bhatia, 2008).

Table 2.6 classical refining of soybean oil



(Courtesy of Handbook of Soy Oil Processing and Utilization, E.H Pry de)

2.6.1 CHEMICAL (CAUSTIC) REFINING

Either a batch or continuous process can be used to refine soyabean oil. In a commercial operation, the soyabean oil from the storage tank or directly from the oil dryer in the solvent extraction plant (and in case of degummed oil from the degumming centrifuge) is pumped through a heat exchanger (to adjust the temperature to 38⁰c into a 'day' tank). The oil thoroughly agitated a sample is taken and analyses are made for free fatty acids and neutral oil content (to determine the amount of caustic required for the refining process). The oil is mixed with a proportioned steam of dilute caustic soda solution. (G.S Breck and S.C Bhatia, 2008).

Where the crude oil is refined directly without degumming, it is pretreated for four hours with (75 percent concentration) of phosphoric acid to facilitate removal of the last traces of phosphatides. The caustic concentration can be varied to compensate for changes in the oil feed characteristics.

According to G.S Breck and S.C Bhatia, 2008. Most soyabean oils are refined with 0.10-0.13 percent excess dry air basis. Caustic

strength of 17-18 Be (12-13percent sodium hydroxide content) is used with soyabean oil.

Pressure or hermetic-type centrifuges are used for both degumming and refining of the oil. Change in the position of the zone of separation can be achieved by adjusting the back pressure applied to the light-phase discharge. Complete separation of the two phases is difficult to achieve, however with either system. The subsequent steps in the refining process are as follows:

The caustic refined oil discharge from the centrifuge is heated to 190⁰f (88⁰c) and mixed with 10-12 percent by weight of soft water that has been heated to 93⁰c. The water oil mixed then passes through a high speed shear mixture to obtain intimate contact for maximum soap transfer from the oil to the water phase. The water oil mixture next passes through a second centrifuge to remove residual soaps; the water- washed oil is discharged as the light phase and the soapy water as the heavy phase. The water-washed refined oil is then sprayed into a continuous vaccum dryer operation at 28inch (58mmHg) pressure

where the moisture content is reduced to less than 0.1percent. The refined soyabean oil is next transferred to the bleaching process or alternatively is continuously cooled to 49⁰c (120⁰f) and shipped as once-refined soyabean oil or it is pumped to storage to await further processing into edible products. (G.S Breck and S.C Bhatia, 2008).

2.6.2 PHYSICAL (STEAM) REFINING

The change from water degumming to acid water degumming has made it possible to eliminate alkali refining entirely in favour of what is called physical refining. This eliminates one significant source of water usage and of waste-water, it also require less capital investment in process equipment. One negative aspect is that it some what restricts the type of oil that can be processed at a plant. (G.S Breck and S.C Bhatia, 2008).

In the steam refining process according to G.S Breck and S.C Bhatia, 2008, the phosphatide-free oil from a pretreatment process is dedourized by operations conducted under high vaccum at high temperature. In a modified physical refining (MPR) process, soaps are generated by adding caustic to crude or

degummed oil to facilitate adsorption removal of phospholipids on amorphous silica hydrogenation.

In physical refining, total adsorbent usage can be reduced while improving product quality, also the primary and water wash centrifuge steps and the associated by-product waste streams, which are needed in caustic refining can be eliminated.

Acid treatment however is required when non hydrated phospholipids are present in the feed stock. (G.S Breck and S.C Bhatia, 2008).

2.7. BLEACHING

Treatment of the refined oil with a bleaching adsorbent removes additional pigments, metallic compounds, residual soaps and trace amounts of pro-oxidants that were not removed by caustic refining and improves the oil's taste. These minute quantities of materials, if not removed, can cause problems in subsequent processing. Prooxidant metal ions present in the form of metallic soaps have a detrimental effect on the hydrogenation process as well as on the flavor and stability of the deodourised oil.

Bleaching adsorbent also removes sulphur compounds, decompose peroxides and adsorb the aldehydes and ketones resulting from decomposition of peroxides (G.S Breck and S.C Bhatia, 2008).

When fatty oils are bleached chemically, colour in reduction occurs because of the oxidizing reactions that have undesirable effect in the flavor and oxidative stability of the oil. For this reason, chemical bleaching is not for edible oils. Exposure of soyabean oil to light, particularly ultraviolet rays has a bleaching effect but light also results in a decrease in the oil's flavor stability. For this reason it is recommended to keep light exposure of soyabean oil to a minimum. (G.S Breck and S.C Bhatia, 2008).

According to G.S Breck and S.C Bhatia, 2008, acidic earths are generally used for bleaching soyabean oil to obtain good chlorophyll removal without which the finished oil may have a greenish cast.

In addition to colour removal, bleaching also almost always improves the initial taste, aged flavor and oxidative stability of the deodourised soyabean oil.

Over bleaching may have an adverse effect on oil quality (such as colour reversion) and it is more costly. Bleaching conditions in the plant should be judiciously selected so that the bleaching effect and impurities removal in other process steps will produce a high-quality finished oil. (G.S Breck and S.C Bhatia, 2008).

2.7.1 BLEACHING PROCESS

Bleaching of soyabean oil is generally carried out according to one of the three basic processes:

- a) Batch atmospheric bleaching
- b) Batch vacuum bleaching
- c) Continuous vacuum bleaching

Batch atmospheric bleaching

In this process, the heated oil (71.1⁰c) is pumped into the tank equipped with steam coils or a jacket and a paddle agitator. Bleaching clay is added at the top of the tank with the agitator

running and the oil is heated to the described temperature (typically 100- 104⁰c or less). The clay oil slurry is then re-circulated through a filter press and back to the bleach tank until it is clear, the clear oil is then pumped to storage or fed into the deodouriser. (According to G.S Breck and S.C Bhatia, 2008).

Batch vaccum bleaching

In a typically batch vaccum process, bleaching clay is added to a portion of heated oil (71.1⁰c) in an open slurry tank and then pumped to a vaccum bleach tank containing the balance of the oil. Vaccum bleach tank are usually under 686-711mmHg (27-28 inch) of vaccum and are equipped with steam coils or jacket and an agitator. After 15-20minutes agitation at the bleach temperature, the oil is cooled to approximately (71.1⁰c), the vaccum is broken and the clay oil slurry is filtered and cooled again to 38-66⁰c before exposure to air (According to G.S Breck and S.C Bhatia, 2008).

Continuous vacuum bleaching

In a continuous vacuum bleaching process, the degummed refined oil is pumped in a properly proportioned steam about 54⁰c into the slurry tank at a rate regulated by a flow controller. Bleach clay and filter aid are also continuously fed into the slurry tank at a controlled rate that is adjusted as required by the characteristics of the oil being bleached. The slurry of oil, filter aid and bleaching clay is thoroughly mixed and then pumped or sprayed at a constant rate into the deaerating and dehydrating (upper) section of a vacuum bleacher that is maintained at 381mmHg (15inch) absolute pressure. In the bleacher, the slurry is first deaerated by sprayed through nozzles and then after retention period of about 7minutes is pumped or sprayed into the bleaching (lower) section of the tower through an external heat exchanger to be heated under automatic temperature control to a bleaching temperature of 104-116⁰c. The slurry is mixed thoroughly to provides for maximum contact between the oil and clay and is retained in the bleacher for a definite period of time. The bleached oil-clay slurry is then filtered in a closed press and

cooled and then the vacuum is broken. The bleached oil is then either pumped to storage or sent to a deodouriser. (By G.S Breck and S.C Bhatia, 2008).

2.7.2 TYPES OF ADSORBENTS USED

The most frequently used adsorbents used for bleaching soyabean oil are natural earth, activated earth and activated carbon. They differ in their performance and in a number of characteristics that affect the processor's choice, ranging from natural, inactivated earth that adsorb soap but have little other function to the expensive, highly activated earths. For each type of adsorbents, more than one grade is available.

Activated earths have a higher bleaching efficiency than natural earths and therefore are preferred to bleach soyabean oil particularly when the oil is dark in colour or high in chlorophyll content. (G.S Breck and S.C Bhatia, 2008).

2.8. DEODOURIZATION

In the conventional edible oil refining, deodourization is the last in a series of process steps used to improve the taste, odour,

colour and oxidative stability of the oils by the removal of undesirable substances. There is still natural odour, flavor and colour of soyabean (ie "beany" odour and flavor and greenish cast) left in a refined soya bean oil. Also after bleaching, the oil still can have a musty or earthy odour. Further more, hydrogenation itself also introduces a typical odour and flavor that has to be removed by deodourization in order to convert the oil into a bland-tasting clear and brilliant liquid. (G.S Breck and S.C Bhatia, 2008).

Deodourization is basically a high temperature, high vacuum, steam distillation process that removes residual constituents left in the oil after refining such as free fatty acids, flavor and odour bearing compounds (e.g. aldehydes, ketones, alcohol), pigments, sterols, hydrocarbons and other compounds formed by the heat decomposition of hydroperoxides and destroy any peroxides that may be present. (G.S Breck and S.C Bhatia, 2008).

According to G.S Breck and S.C Bhatia, 2008. The three common methods of deodourization currently in commercial use are (a) batch (b) semi -continuous (c) continuous.

The conditions used commercially for deodourization of fatty oils vary appreciably with the type of equipment used, the type of oil being deodourised and the degree to which the oil needs to be deodourised. Commercially deodourization also vary considerably with respect to operating time and oil residence time (G.S Breck and S.C Bhatia, 2008).

2.8.1 DEODOURIZATION OF SOYABEAN OIL

Batch deodourization can produce good quality soyabean oil but semi continuous and continuous deodouriser have a number of advantages. Some of the advantages are: inherent in the process while others are result of equipment design. Removal of odour and flavor-bearing compounds from edible oils by deodourization in batch system is dependent on both time and temperature. To obtain the best balance between both initial quality and keeping properties of oils, deodourization times,

should be short when temperatures are high and longer when temperatures are low. . (G.S Breck and S.C Bhatia, 2008).

Deodourization time and temperature producing the most stable soyabean oil are measured by organoleptic tests and oxidative stability under schall oven conditions. . (G.S Breck and S.C Bhatia, 2008).

2.8.2 EFFECT OF DEODOURIZATION ON OIL QAULTY

A freshly and properly deodourized soyabean oil has a bland or nearly bland taste and even after monetary heating to 177⁰c has almost no odour. The deodourised oil ultimately should have less than 0.03percent FFA, a peroxide value of zero, lovibond colours of 10 yellow and 0.7 red and a bland taste . (G.S Breck and S.C Bhatia, 2008).

2.9. EVALAUTION OF FINISHED OIL QUALITY

The official specifications for soyabean oil and its products as discussed earlier are only minimum standards. The ultimate measures of quality are the odours and flavor to the products.

Sensory evaluation, therefore, with or without instrumental analysis, is necessary for both product development and quality control . (G.S Breck and S.C Bhatia, 2008).

Although chemical and physical tests are more reproducible and less time consuming the sensory evaluations, rancidity determinations must always be done by an organoleptic observation. Nevertheless, establishing correlations between organoleptic scores and various chemical tests are possible and frequently used in commercial practice. For example, the spectrometric characteristics of raw oil can predict the sensorial acceptability of the refined deodourised final product. . (G.S Breck and S.C Bhatia, 2008).

2.9.1 STORAGE AND HANDLING

Soybean oil may undergo oxidative, enzymatic and microbiological determination in storage or during and after processing. Production of the highest quality soybean oil product requires close control from harvesting to consumption of the finished products. . (G.S Breck and S.C Bhatia, 2008).

In relation to handling and storage factors that contribute to oxidative deterioration of soyabean oil G.S Breck and S.C Bhatia, 2008. Reported that list and Erikson states that heat accelerates the reaction of atmospheric oxygen with edible oils and therefore localized. Over heating is detrimental to fat quality. To avoid this, all storage tanks with heating devices should be equipped with mechanical agitation.

G.S Breck and S.C Bhatia, 2008. Also stated that Going has reported on the oxidative stability of soyabean oil at various processing steps and related the results to commercial storage conditions. According to this study the oxidative rate doubles for every 11.1°C increase in temperature. The effect of container sizes on the oxidative deterioration of refined, bleached soyabean oil stored at 48.9°C for 5 weeks has also been reported. The results clearly demonstrate that oxidation is not only a function of time and temperature but also of the amount of oxygen available. Oxygen availability is lower if container size increases; this is so because the surface to volume ratio decreases.

CHAPTER THREE

3.0 MATERIALS AND METHODS

This chapter describes the details of the procedures employed in the refining of soyabean oil showing the materials and equipments. It also shows in details the procedures and the reagents used in determining some properties of the oil refined.

3.1 MATERIALS USED

The major material used in this research project is crude soyabean oil which was bought at Phenoma Nig. Ltd. Ngwo, Enugu state.

3.2 EQUIPMENTS USED

- Heating mantle
- Round bottom flask (250ml)
- Reflux condenser
- Conical flask (250ml)
- Beakers-50ml 100ml, 250ml
- Burette
- Weighing balance
- Specific gravity bottle

- Measuring cylinder 50ml
- Stirrer
- Stop watch
- Viscometer
- Electronic oven
- Pipette
- Restort stand
- Volumetric flask
- Constant temperature water bath
- Pipes

3.3 REAGENTS USED

- Alcoholic potassium hydroxide (0.5molar)
- Standard hydrochloric acid (0.5molar)
- Carbontetrachloride
- wij's solution
- Standard sodium thiosulphate (0.1molar)
- Neutral alcohol
- Chloroform
- Standard potassium hydroxide (0.5molar)

- Starch indicator
- Phenolphthalein indicator
- Ethanol
- Phosphoric acid (50%)
- Sodium hydroxide (1normal)

3.4 PREPARATION OF REAGENTS

Some reagents that was prepared for testing some of the properties of the refined oil includes:

- i. 0.5molar Alcoholic potassium hydroxide.

14 grams of potassium hydroxide poured into a volumetric flask and 500ml of ethanol was used to dissolve it.

- ii. 0.5molar potassium hydroxide

28 grams of potassium hydroxide was weighed using a weighing balance and poured into a 1litre volumetric flask and was dissolve with distilled water up to 1 litre

- iii. 0.5molar hydrochloric acid

18 grams of hydrochloride was weighed using a weighing balance and transferred into 1litre volumetric flask and was dissolved with distilled water up to 1 litre

iv. 0.1molar sodium thiosulphate

6.2 grams of sodium thiosulphate was weighed using a weighing balance and poured into a 250ml volumetric flask and dissolved with distilled water up to 250ml.

v. Neutral alcohol.

250ml of ethanol was measured and diluted with 20ml of sodium hydroxide

3.5 PROCEDURES

250ml of the crude soyabean oil was poured into a beaker and 250ml of water was measured and mixed with the crude soyabean oil and the mixture was heated for 30minutes with a heating mantle.

Again 250ml of crude soyabean oil was poured in another beaker and mixed with 250ml of 50% phosphoric acid and heated for 10minutes. The two Samples were allowed to cool and decanted

into separate beakers. The degummed oil with phosphoric acid was neutralized/ refined with sodium hydroxide (NaOH) of different quantities. 50mls each of different samples of the oil was mixed with different quantities of sodium hydroxide and allowed to stand for 12hours before decanting. The table below shows the different quantities of sodium hydroxide used.

Table 3.1 degummed soyabean oil with different quantities of sodium hydroxide (NaOH).

Degummed soyabean oil	Quantity of NaOH (1 normal)
50ml +	2ml
50ml +	4ml
50ml +	6ml
50ml +	8ml
50ml +	10ml

Finally, 250ml of crude soyabean oil was added and poured into a beaker and 250ml of phosphoric acid was measured and the mixture was heated for 10 minutes and allowed to cool before it was decanted and the degummed oil was mixed with 8ml of sodium hydroxide and heated for 20 minutes, it was allowed to cool and then decanted into a beaker.

3.6 TESTING OF OIL

A number of tests were conducted on the refined oil. These tests were as follows

- I. Saponification value
- II. Iodine value
- III. Acidic value
- IV. Specific gravity viscosity

3.6.1 TEST FOR SAPONIFICATION VALUE

5grams of the refined oil was weighed using a weighing balance and transferred into a 250ml round bottom flask. 50mls of 0.5molar alcoholic potassium hydroxide was measured using a measuring cylinder and added to the oil. To this was fixed a reflux condenser and the contents were refluxed for one hour. Porous bite was added to the flask to ensure uniform heating. The content of the flask was cooled and titrated against 0.5molar standard hydrochloric acid. In a similar manner, 50mls of the same alcoholic potassium hydroxide was refluxed in a round bottom flask for one hour and the content was allowed to cool

and titrated against standard hydrochloric acid. Phenolphthalein indicator was used.

3.6.2 TEST FOR IODINE VALUE

0.5grams of the refined oil was weighed using weighing balance and poured into a conical flask. 25mls each of carbon tetrachloride and Wij's solution was measured accurately using a measuring cylinder and poured into the oil and the content of the flask was shaken well to ensure complete mixing. Standard sodium thiosulphate was taken in a burette and titrated against the content of the flask using a starch indicator. A duplicate solution was prepared without the oil and was also titrated using the same sodium thiosulphate.

3.6.3 TEST FOR ACIDIC VALUE

1.0gram of the refined oil was weighed using a weighing balance and transferred into a conical flask. 50ml each of neutral alcohol and chloroform was measured using a measuring cylinder and added to the oil in the flask. The content of the flask was shaken well to dissolve the free fatty acids. This was immediately titrated

with standard potassium hydroxide using phenolphthalein as indicator. The end point was the appearance of a pale pink colour.

3.6.4 TEST FOR SPECIFIC GRAVITY

The specific gravity bottle was dried in an electronic oven and weighed using a weighing balance. Distilled water was poured into the bottle and weighed. In the same manner the refined oil was also poured into the bottle and weighed.

3.6.5 TEST FOR VISCOSITY

Digital display viscometer model NDJ85 was used to test for the viscosity of the oil. The oil in a beaker was placed under the instrument which was gradually lowered into the oil and the rotor gradually dips into the oil until the oil surface mark on the rotor was level. The rotating speed used was 60r/min.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 RESULTS

In this chapter the results of the test carried out on this work will be discussed using table were necessary. The experimental work provides the proportion of materials used in this work.

Table 4.1 Result of the degummed soyabean oil.

Classification	Result
Crude soyabean oil + water +heat	Less oil yield, emulsion formed
Crude soyabean oil + phosphoric acid +heat	No emulsion/high oil yield

Table 4.2 Result of the canstic refining of soyabean oil with different quantities.

Classification	Result
Degummed oil (50ML) + 2ml NaoH	High oil yield (clear oil)
Degummed oil (50ML) + 4ml NaoH	Lowest oil yield (clear oil)
Degummed oil (50ML) + 6ml NaoH	Lowest oil yield (clear oil)
Degummed oil (50ML) + 8ml NaoH	Lowest oil yield (clear oil)
Degummed oil (50ML) + 10ml NaoH	Lowest oil yield (soap stock)
Degummed oil (250ML) + 8ml NaoH + HEAT	High oil yield (oil not very clear)

Table 4.3 Test results of the properties of the refined oil and crude oil.

Classification	Refined Oil	Crude Oil
Saponification value	42.075	44.88
Iodine value	164.28	109.52
Acidic value	589.05	448.8
Specific gravity	0.8944	0.8148
Viscosity	0.038	0.035

4.2 TITRATION READINGS

Table 4.4 Titration reading of saponification value of refined oil.

Saponification value of Refined Oil

Burette reading	Refluxed oil +			Refluxed solution alone		
	1st titre	2nd titre	3rd titre	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00	0.00	0.00	0.00
Final value (ml)	27.50	29.50	30.00	38.00	35.50	36.00
Average titre value = 29ml				Average titre value = 36.5ml		

Table 4. 5 Titration reading of Iodine value of refined oil.

Iodine value of refined oil

Burette reading	Solution + oil			Solution alone		
	1st titre	2nd titre	3rd titre	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00	0.00	0.00	0.00
Final value (ml)	24.50	22.00	25.50	30.50	31.00	28.50
Average titre value =24ml				Average titre value =30ml		

Table 4.6 Titration reading of Acidic value of refined oil.

Acidic value

Burette reading	Oil + solution		
	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00
Final value (ml)	21.00	23.00	19.00
Average titre value = 21ml			

Table 4.7 Titration reading of saponification value of crude oil.

Saponification value of crude oil

Burette reading	Solution + oil			Solution alone		
	1st titre	2nd titre	3rd titre	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00	0.00	0.00	0.00
Final value (ml)	25.00	23.00	24.00	30.00	33.50	32.50
Average titre value =24ml				Average titre value =32ml		

Table 4.8 Titration reading of iodine value of crude oil.

Iodine value of Crude oil

Burette reading	Solution + oil			Solution alone		
	1st titre	2nd titre	3rd titre	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00	0.00	0.00	0.00
Final value (ml)	31.50	29.50	29.00	32.00	34.50	35.50
Average titre value = 30ml				Average titre value = 34ml		

Table 4.9 Titration of acidic value of crude oil.

Acidic value of crude oil

Burette reading	Oil + solution		
	1st titre	2nd titre	3rd titre
Initial value (ml)	0.00	0.00	0.00
Final value (ml)	17.50	14.50	16.00
Average titre value = 16ml			

4.3 DISCUSSION

In this research work, I was able to observe that crude soyabean oil does not undergo solidification at room temperatures.

Table 4.1 shows that crude soyabean oil react with water to form emulsion as a result of its high protein content and yield low quantity of oil at the degumming, while phosphoric acid yields high quantity of oil and does not react with oil to form emulsion.

From table 4.2 it was observe that neutralization of pretreated/degummed soyabean oil requires a cold process (no heat) for a good quality bland of oil and it also shows that 50ml of degummed oil requires only 2ml of sodium hydroxide for neutralization.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Having being privileged to conclude a research on this project work and engaging fully to it practical work I can say that refining of soyabean oil was in indeed an interesting one. I have also arrive at the conclusion that refining of soyabean oil is best carried out using the alkali/ caustic refining method and that degumming process should be done with phosphoric acid while the neutralization with sodium hydroxide should take a cold process for a good quality edible oil to be produce.

5.2 RECOMMENDATION

This work centered on the neutralization/refining of soyabean oil using alkali method, I by recommend that for a good bland quality product to be obtained, the oil should subjected to further refining processes like bleaching and deodourization.

I also encourage my fellow students to carry out their research project themselves with the help of their supervisor in order to help them to grow in knowledge and also be productive so that they will be able to defend themselves and measure up with their colleague in the labour market

REFERENCES

- Akpan U.G, Jimoh A., Mohammed A.D. *Extraction Characterization of oil seed.*
- Breck G.S and Bhatia S.C (2008). *Hand book of industrial fats and oil: Vol 2.*
- Erickson D.R (1983). *Jaocs.* vol 60, Asa Saint Louis (U.S.A).
- Heldman Dennis R. (1975). *Food processing Engineering.* West Port Connecticut. The AVI Publishing Company.
- Kirk. Othmer (1964). *Encyclopedia of chemical Technology: 2nd Edition: Vol. 4.*
- National Occupational Health and Safety Commission (1991). *Draft National Code for spray painterly.* Canberra, Australia.
- Perry, R.H (1988). *Chemical Engineering Handbook: 5th Edition* USA. McGrawHill Book Company

Pryde E.H. *Hand book of soy oil processing and utilization* Usda-peoria.

Reigel (2003). *Hand book of industrial chemistry:vol 4.*

Weise E A. (1983). *Oil seed crops.* Tropical Agricultural series Longman.

Young F.V.K (1981). *Symposium on soya bean processing: 5th Edition (U.S.A).*

APPENDICES

A

Calculation of Saponification Value

Strength of Hcl used = M (molar) = 0.5 molar

Weight of oil used = A(gram) = 5grams

The value of Hcl against

koH+oil after relax = xml = 29ml

the value of Hcl against

koH alone = Yml = 36.5ml

vol of Hcl required to neutralize excess alkali

after saponification = (Y-Xml) = zml = 36.5-29

=7.5ml

Saponification value constant=56.1

$$\text{SAPONIFICATION VALUE} = \frac{Z \times m \times 56.1}{A}$$

$$= \frac{7.5 \times 0.5 \times 56.1}{5} = \frac{210.375}{5}$$

$$= 42.075$$

B

Calculation of Iodine Value

Weight of oil used = A gram = 0.5 gram
Strength of thiosulphate = M molar = 0.1 molar
Amt of thiosulphate required to
Neutralize 25ml of wij's solu = x ml = 30 ml
Amt of thiosulphate required to
Neutralize the solution with oil = y ml = 24 ml
Amt of iodine consumed by
The oil = x - y = Z ml = 30 - 24 = 6 ml
Iodine Value constant = 136.9

$$\begin{aligned}\text{Iodine value} &= \frac{136.9 \times m \times z}{A} \\ &= \frac{136.9 \times 0.1 \times 6}{0.5} = \frac{82.14}{0.5} = 164.28\end{aligned}$$

C

Calculation of Acidic Value

Weight of oil used = A gram = 1 gram
Strength of KOH used = M molar = 0.5 molar
Vol of KOH required to
Neutralize the oil solution = X ml = 21 ml
Acidic constant = 56.1

$$\begin{aligned}\text{Acidic value} &= \frac{x \times 56.1 \times m}{A} \\ &= \frac{21 \times 56.1 \times 0.5}{1} = \frac{589.05}{1} \\ &= 589.05\end{aligned}$$

D

Calculation of Specific Gravity

Weight of specific gravity bottle = 21.45gram

Weight of specific gravity bottle + water = 74.68gr

Weight of specific gravity bottle + oil = 69.06gr

Weight of water = 74.68-21.45=53.23gr

Weight of oil = 69.06-21.45= 47.61gr

Specific gravity of oil = $\frac{\text{weight of oil}}{\text{weight of water}}$

$$= \frac{47.61}{53.23} = 0.8944$$

(E)

Calculation of Saponification Value of Crude Soya Bean Oil

Strength of Hcl used = m (molar) = 0.5molar

Weight of oil used = A (gram) = 5grams

The value of Hcl against

koH + oil after reflux = xml = 24ml

the value of Hcl against

koH alone = yml = 32ml

vol of Hcl required to neutralize excess

alkali after saponification = (y-x ml) = Zml

$$32-24 = 8\text{ml}$$

Saponification constant = 56.1

Saponification value = $\frac{z \times m \times 56.1}{A}$

$$= \frac{8 \times 0.5 \times 56.1}{5} = \frac{224.4}{5} = 44.88$$

(F)

Calculation of Iodine Value of Crude Soya Bean Oil

Weight of oil used = A (gram) = 0.5gram

Strength of thiosulphate = M (molar) = 0.1molar

Amt of thiosulphate required to neutralize

25ml of wips solution = xml = 34ml

Amt of thiosulphate required to neutralize

The solution with oil = Yml = 30ml

Amt of iodine consumed by the oil = (X-Yml) = Zml

$$= 34-30 = 4\text{ml}$$

Iodine value constant = 136.9

$$\begin{aligned} \text{Iodine value} &= \frac{136.9 \times m \times z}{A} = \frac{136.9 \times 0.1 \times 4}{0.5} \\ &= \frac{54.76}{0.5} = \underline{109.52} \end{aligned}$$

(G)

Calculation of Acidic Value of Crude S.B.O

Weight of oil used = A (gram) = 1gram

Strength of KOH used = M (molar) = 0.5molar

Vol of KOH required to Neutralize the

Oil solution = Xml = 16ml

Acidic value constant = 56.1

$$\begin{aligned} \text{Acidic value} &= \frac{x \times 56.1 \times m}{A} = \\ &= \frac{16 \times 56.1 \times 0.5}{1} = \frac{448.8}{1} = \underline{448.8} \end{aligned}$$

(H)

Calculation of Specific Gravity of C.S.B.O

Weight of specific gravity bottle = 21.45gram

Weight of specific gravity bottle + water = 74.68 gram

Weight of specific gravity bottle + C.S.B.O = 64.82 gram

Weight of water = 74.64 – 21.45 = 53.23 gram

Weight of oil = 64.82 – 21.45 = 43.37gram

Specific gravity of C.S.B.O = $\frac{\text{weight of oil}}{\text{weight of water}}$

$$= \frac{43.37}{53.23} = \underline{0.8148}$$