

**KWAME NKRUMAH UNIVERSITY OF SCIENCE AND
TECHNOLOGY**

ELECTROLYTE DEGUMMING OF CRUDE VEGETABLE OIL

By
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DECLARATION

I hereby declare that this submission is my own work towards the MSc. Food Science and Technology programme. To the best of my knowledge, it contains no material previously published by another person or material which has been accepted for the award of any other degree of the university, except where due acknowledgment has been made in the text.

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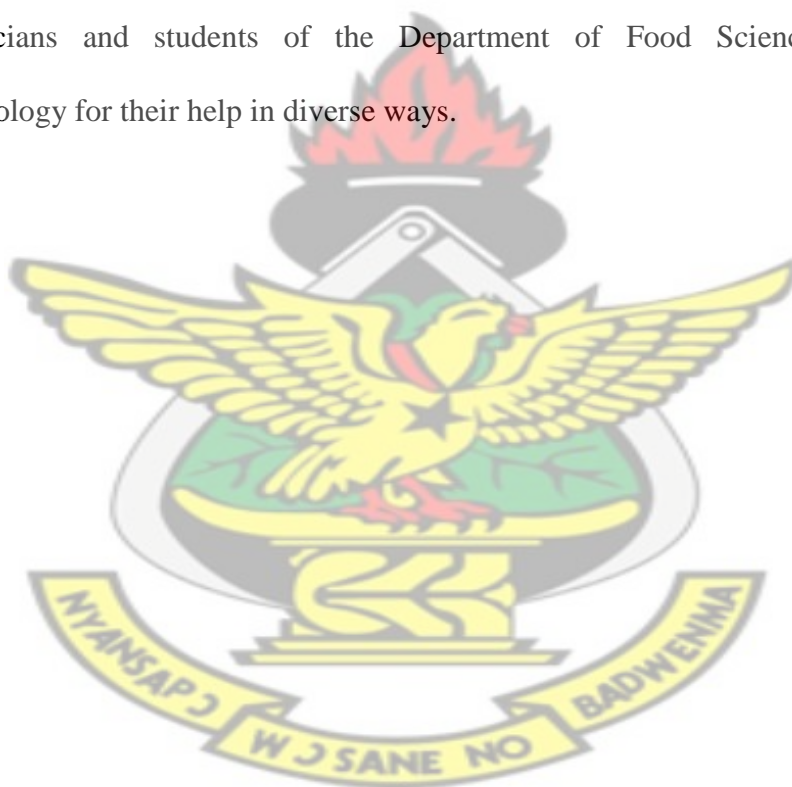
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ABSTRACT

Electrolyte degumming is an emerging procedure which employs the use of electrolytes in the removal of phospholipids present in crude vegetable oil. In this research, Crude soya bean oil (CSBO) was degummed using six factors; electrolyte concentration (0.5-3.0 % w/v), electrolyte-oil ratio (1-5 % v/v), temperature of medium (50 -80 °C), reaction time (30-60 min), agitation speed (150-300 rpm) and the electrolyte combination (KCl-CaCl₂; NaCl-MgCl₂). Response surface methodology was used to randomize the factors involved in the process, analyze the responses obtained, predict the best-fit regression model and optimize the degumming experiment. A reduced quartic model was obtained as the best model with $p < 0.05$ and lack of fit of 0.88. Statistical analysis revealed linear, quadratic, cubic and quartic interactional effects respectively. Based on the model, the optimum condition was evaluated using sodium and magnesium chloride mixture at a concentration of 2.4 % w/v and electrolyte-oil ratio of 4 % v/v. The condition which was maintained at 67 °C, agitated at 235 rpm for 37 min produced a degummed soya oil of 4 ppm.

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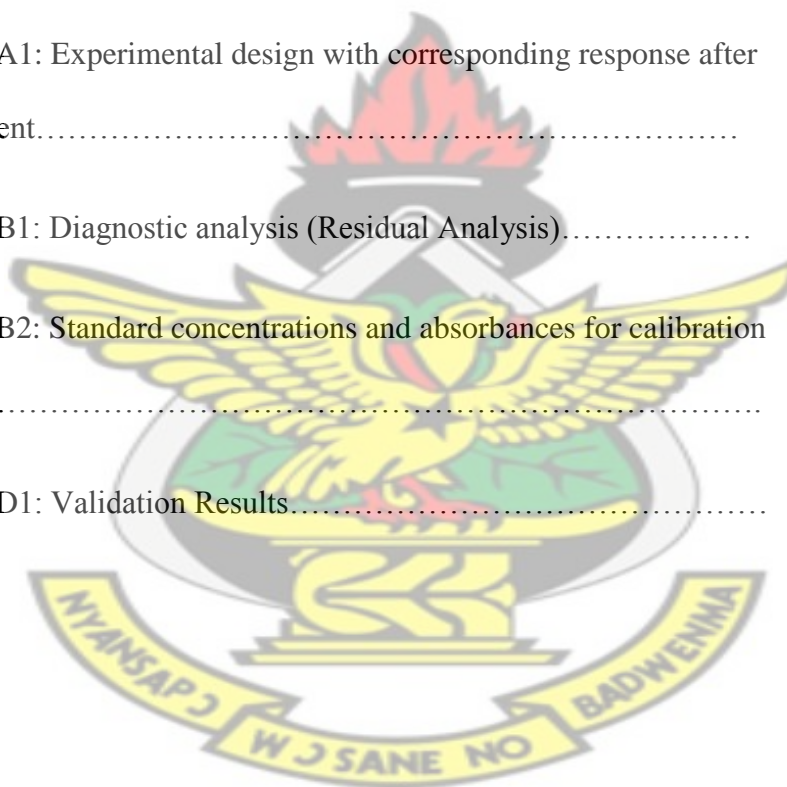
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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background

In the years 2011/2012 160 million tons of vegetable oil was produced throughout the world and 85% of the production was accounted for by four main vegetable oil plant species. One of them is the soya bean plant which produces soya bean oil, this accounted for 26 % of the world vegetable oil production (Michel, 2012).

Soya bean oil is a vegetable oil extracted from the seeds of the soybean (*Glycine soja*), it is one of the most widely consumed cooking oil in the world. It is valued for its affordability, high smoke point and health benefits. It often has a dark yellow or faint green colour, which can be used for baking, cooking and frying (David *et al.*, 2003).

Soya bean oil has high polyunsaturated fatty acids content, containing approximately 50% linoleic acid (ω - 6). This serves as a perfect choice for individuals with heart disease and high bad cholesterol levels (O'Brien, 2009).

Soya bean oil also contains vitamin E and omega - 3- fatty acids, which reduces the risk of cardiovascular disease, slow down the growth of atherosclerotic plaque, and arrhythmias. The oil also naturally contains antioxidants, which remains in the oil after it is extracted from the plant, this aids the prevention of oxidation, thus enhancing the prevention of certain cancers (David *et al.*, 2003).

In the production of soya bean oil, the soybeans are cleaned, cracked, adjusted for moisture content by drying, heated to temperatures between 60 - 80 °C, rolled into flakes and solvent-extracted with hexane. The extracted oil is then refined and blended for different applications (e.g. partial hydrogenation). The residue, soybean meal which is high in protein content is used as animal feed (O'Brien, 2009).

Crude edible vegetable oils are usually known as the unrefined and unprocessed oils extracted from vegetables (seeds or stems), which are rich sources of unsaturated triacylglycerols. Triacylglycerols provide energy, maintain muscle activity, insulates the body, and aid in the absorption of vitamins whilst protecting the organs from injury. Most edible oils are mostly extracted from plants through a combination of mechanical and solvent extraction process (David *et al.*, 2003).

Oil obtained through this process is termed crude oil because it contains a number of impurities, which are categorized into two major parts namely, soluble and insoluble impurities. Visible impurities such as seed fragment, meal fines, fibres etc. are known as insoluble impurities. Impurities such as free fatty acid, ketones, tocopherols, phytosterols, phospholipids, proteins, pigments, resins etc. are soluble impurities (Rohani, 2006 and Marilyn, 2002).

Most of these impurities have unfavourable effects on the flavour, odour, appearance, and shelf life of the vegetable oil. Due to these effects, the removal of these impurities is necessary in order to maintain the nutritional quality and shelf life of the oil.

Most of the impurities are removed from crude vegetable oil by chemical, mechanical and physical refining processes respectively. In order to convert the crude vegetable oil to refined oil, it must undergo further processing to take it from its crude form to a refined edible state. Such processes include degumming, bleaching, deodorisation and fractionation (Marilyn, 2002 and O'Brien, 2009).

Quality and stability are the major important factors in the production, acceptance and marketing of vegetable oil products. Based on the impurities present in crude oils, these properties depend mainly on the seed quality, seed treatment prior to extraction, processing conditions during extraction and refinery of crude vegetable oil (Borner *et al.*, 2003).

Phospholipids which are also called phosphatides or gums are one of major soluble impurities, which pose many problems to the processing and storage of crude vegetable oils. The presence of phospholipid in oils beyond 30ppm causes higher oil loss during neutralization, inefficient bleaching and darkening of the oil at high temperature during deodorization (O'Brien, 2009).

Phospholipids are removed from oil during refining by a process known as degumming. Degumming is an important preliminary step in oil refining process, because it involves the removal of phospholipids along with other unwanted minor compounds without destroying the beneficial ones (Brekke, 1975).

Gums tend to produce high refining losses, foaming, settling during storage and discoloration of the oil during processing(Eickhoff, 2000). Degumming plays a critical role in the refining process of crude vegetable oil, in that any inefficiency during degumming would severely affects other refinery processes. Example includes, increased oil loss at the neutralization section, reduce absorption of the bleaching agent at the bleaching section and darkening of oil at high temperature during deodorisation (O'Brien, 2009). In degumming if such inefficiencies are not corrected immediately, the quality, shelf-life and storage of the finished product would eventually be affected badly(Oyebek *et al.*, 2008).

1.2 Problem Statement

From the refiners' point of view, the degumming and bleaching processes of the refinery are recognized as critical areas in refining crude vegetable oils. These stages must be carefully monitored because any inefficiency during these processes will extremely affect the later stages of the refinery badly.

These stages are also identified as the major contributors to the total operating cost of the plant. This is due to the cost of utilities such as phosphoric acid and bleaching earth used in these processes. It is estimated that about 30% of total operating cost is due to bleaching and degumming process of the refinery (Rohani, 2006 and Borner *et al.*, 2003).

Currently the price of bleaching earth ranges between \$250 to \$400 per MT whilst phosphoric acid ranges between \$1500 to \$2000 per MT(Prayon Technologies, 2012).

In comparing both stages of the refinery, the degumming stage of the refinery is more critical compared to the bleaching stage of the refinery, this is due to the effect any inefficiency during degumming can cause on the preliminary processes involved in refining crude vegetable oil. Such effects includes, reduced absorption of bleaching earth, darkening of the oil during deodorization, and increased oxidative instability leading to reduced shelf life of the finished product (Campos *et al.*, 2009).

In order to achieve a bland, tasteless, odourless and light golden yellow coloured refined vegetable oil, the maximum phosphorus content of the degummed oil should not exceed 10 ppm (Prabhakaran *et al.*, 2009 and Copeland *et al.*, 2001).Several degumming methods have been introduced in recent years to achieve the required low phosphorus content for physical refinery of crude vegetable oils.

These methods include among others, water degumming, super degumming, Totaal Ontsliming Processes (TOP degumming), ultra-filtration degumming and acid degumming. Unfortunately, these degumming methods cannot guarantee the achievement of low phosphorus content degummed oil required for the physical refinery. Furthermore, these processes are not always suitable for all types of crude vegetable oils owing to the high content of non-hydratable

phospholipids present in some crude vegetable oils (Copeland *et al.*, 2001).

The high oil yield loss, equipment requirement and energy expenditure of these processes also leads to high operating costs, hence making the aforementioned degumming methods unsuitable for industrial processes (Yang *et al.*, 2006).

1.3 Justification

In the past, TOP degumming, water degumming and acid degumming, were used industrially in refining crude vegetable oils. Water degumming is an ineffective method when used in the production of lecithin, but unfortunately does not enhance the removal of non-hydratable phosphatides. TOP degumming is highly efficient but involves the use of a base and a concentrated acid which leads to high operating cost. This is due to the cost of chemicals and disposal of the soap formed during TOP degumming. The use of alkali chemicals such as sodium hydroxide also makes the method unsuitable for crude vegetable oils containing high free fatty acid (FFA) content (O'Brien, 2009 and Oybek *et al.*, 2008).

Even though acid degumming is an efficient method when used for degumming low phosphate crude vegetable oils. Crude vegetable oils containing high amount of phosphatides are degummed in combination with water degumming (List *et al.*, 1981). In degumming crude vegetable oils with high non-hydratable phosphatides, an increase in the dosage of the phosphoric acid dosage is necessary in order to achieve

the required limit for physical refinery. This leads to high production cost since the food grade phosphoric acid is costly (Borner *et al.*, 2003).

Presently, other degumming methods such as soft degumming, enzymatic degumming, membrane degumming, super critical carbondioxide degumming and electrolyte degumming have emerged. All the degumming methods mentioned above are efficient in producing degummed vegetable oil suitable for physical refinery, but in most cases involves the use of high cost equipment or degumming agent in the process (Borner *et al.*, 2003). This contributes to the high operating cost in degumming leading to high product cost.

Enzymatic degumming requires a long reaction time to reduce the phosphorus content of the degummed oil to the allowable limit (Yang *et al.*, 2006). Electrolyte degumming involves the use of electrolytes to degum the crude vegetable oil within a low reaction time (Nasirullah, 2005). The future aims at degumming at high efficiency at a reduced utility cost whilst producing a degummed oil of negligible residual phosphorus content.

Due to high oil loss, expensive equipment procurement, high energy requirements and inefficiency of the traditional degumming processes, there is a great need to degum using a different degumming method. The method must yield high efficiency at reduced utility cost in order to avert the above challenge. A novel method that can be used in the process is electrolyte degumming.

Electrolyte degumming is an emerging new degumming method which employs the use of electrolytes in degumming of crude vegetable oil. It is an inexpensive method due to the low utility cost, low energy requirements, low contact time and operating cost(Nasirullah, 2005). Electrolyte degumming is suitable for crude vegetable oils with both low and high phosphorus content (gum) when compared to other degumming processes such as membrane degumming, acid degumming etc.

In order to evaluate the optimum condition suitable for degumming, there is a need to identify the relevant factors that affect degumming and note the range within which these factors influence the process. Listet *al.*,(1981) and Pan *et al.*, (2001) during a degumming experiment identified factors such as concentration of the degumming agent, degumming agent-crude vegetable oil ratio, and reaction time. The temperature of the medium and agitation speed (degree of dispersion) were also identified as relevant factors since they have significant effect on the experiment.

1.4.1 Aim

The aim of this experiment was to identify the optimum conditions suitable to efficiently degum crude soya bean oil below the allowable limit set for physical refinery.

1.4.1 Specific Objective

The specific objective of this project was to establish the conditions to optimally degum crude soya bean oil in terms of electrolyte combinations, electrolyte concentration, electrolyte-oil ratio, temperature, contact time and agitation speed.

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CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Chemistry of Degumming

Phospholipids are mostly referred to as phosphoglycerides, which are better known to oil processors as phosphatides and in most cases referred to as gums. Phosphatides are soluble impurities known to cause adverse effects on product quality and oil yield of refined vegetable oil (Segers *et al.*, 1990 and O'Brien, 2009).

Phospholipids are emulsifiers that hinder the separation of oil and water phases in the chemical refining process. They interfere with effective bleaching, act as oxidation catalyst during storage, shorten the shelf-life of finished product and foul equipment surfaces hence reducing the efficiency of the processing equipment (O'Brien, 2009).

The structure of the phospholipid molecule consists of a hydrophilic head and a hydrophobic tails. The hydrophilic head contains the negatively charged phosphate group and hydrophobic tail usually consists of long fatty acid hydrocarbon chains.

The structure of phospholipids (PLs) is composed of glycerol backbone and fatty acids substrate which is usually on the sn-1 and 2 positions. The structures also consist of a phosphate, and usually an organic base or polyhydroxy compound, which is mostly on the sn-3 position (Belitz *et al.*, 2009 and Marilyn, 2002).

The parent structure of the phospholipid is a derivative of phosphatidic acid (sn-1, 2-diacylglycerol-3-phosphate). In table 2.1 are the chemical structures of phospholipids mostly found in crude vegetable oils (Wassef, 1996 and Marilyn, 2002). Degumming is a unit operation which involves purification of seed oils, which normally contain impurities in the colloidal state or dissolved in the crude vegetable oil (Bernardini, 1985).

Phospholipids are categorised into two types, based on their ability to precipitate in the presence of water, either fast at elevated temperature or slowly at low temperature. These categories include hydratable and non-hydratable phosphatides. Examples of hydratable phosphatides, are phosphatidylcholine and phosphatidylinositol; they usually form precipitates when contacted with water (water degumming). Non-hydratable phosphatides such as phosphatidylethanolamine (cephalin), phosphatidic acid, lysophosphatidic acid, calcium and magnesium salts of the acids remain in the oil after water degumming. Non-hydratable phospholipids are removed by methods such as acid or TOP degumming (O'Brien, 2009).

2.2 Hydratability of Phospholipids

The extent of degumming depends on the amphipathic character of the phospholipid, in other words this refers to hydrophilic and hydrophobic characters of the phospholipid. The hydrophilic property is the ability of the phospholipid molecule to attract water and the hydrophobic character is the ability of the phospholipid to repel water. Both the

hydrophilic and hydrophobic properties of the lipids depend on the chemical structure of the phospholipid (Pan *et al.*, 2000).

The hydrophilic properties of phospholipids determine the hydratability or the extent to which a phosphatides present in the crude oil are removed during degumming. (Dijkstra, 2011). Table 2.1 shows the chemical structure of phospholipids found in crude vegetable oil. The chemical structure of phosphatidylinositol has five free hydroxyl groups on the inositol moiety, hence making phosphatidylinositol strongly hydrophilic. Phosphatidylinositol present in crude oil is hydratable, hence after water degumming the phosphatidylinositol content of properly water-degummed oil is negligible.

Similarly, the positive charge of the trimethylamino group in phosphatidylcholine makes it hydrophilic. The hydrophilicity of phosphatidylcholine does not depend on the pH of the water used to degum the oil. At $\text{pH} > 5$, the phosphate group in the phosphatidylcholine is dissociated and therefore carries a negative charge, it does not form an internal salt with the quaternary amino group for steric reasons. Consequently, the positive quaternary amino group remains isolated at all pH values and causes phosphatidylcholine to be hydrophilic at all pH values (Dijkstra, 2011).

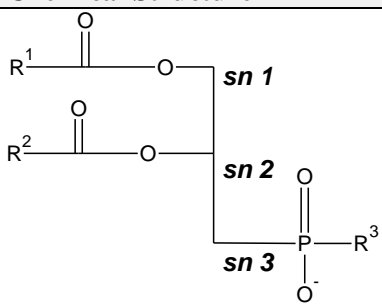
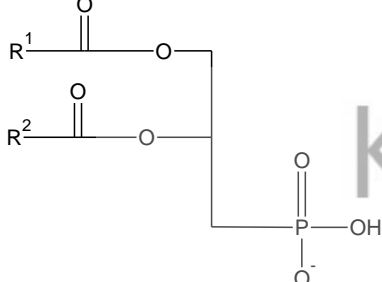
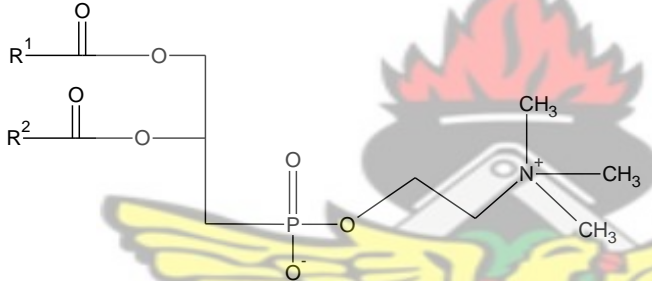
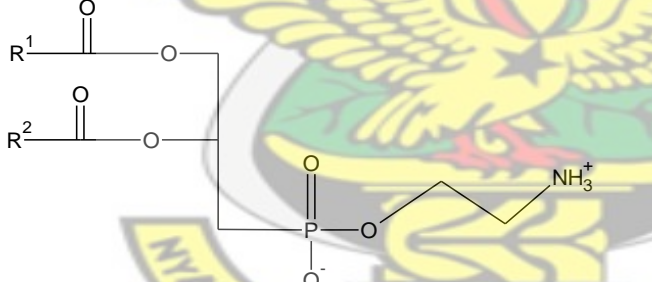
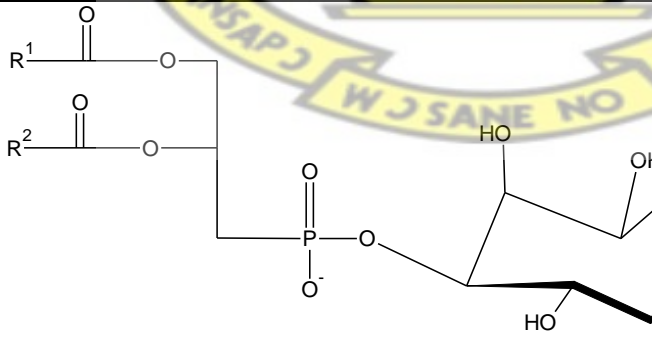
During water degumming, phosphate groups of phosphatidylethanolamine (PE) dissociate and a zwitterion molecule is formed. In this case the dissociated positive amino group forms an internal salt with the negative phosphate group.

The positive and negative charges are so close together that the hydrophilicity of the zwitterion molecule is weak and on water degumming, the hydration of PE is incomplete. In an acidic medium phosphatidylethanolamine (PE) molecules have a positive charge and the charge causes PE to be hydrophilic hence in an acidic medium PE is hydratable. The chemical structure of phosphatidic acid (PA) reveals that in an acid medium, the hydroxyl groups of its phosphate moiety will not dissociate since the pK_a value of the first hydroxyl group is within 2.7-3.0.

This indicates that PA will be poorly hydratable in an acidic medium. During degumming as the pH of the water approaches 5, the PA dissociates hence becoming negatively charged giving it a hydrophilicity that makes it hydratable (Abramson *et al.*, 1964). Considering calcium, magnesium and iron salts of phosphatidic acid (Ca-PA, Mg-PA and Fe-PA), preliminary acidification will dissociate the divalent cation attached to the phosphatidic acid. The dissociated divalent phosphatidic acids are then attached unto the bleaching earth which can be removed by filtration.

Further treatment by raising the pH using a base will dissociate the H^+ ion creating a sodium salt of phosphatidic acid (Na - PA) which can be removed by centrifugation (Dijkstra, 2011 and Deffense *et al.*, 2009). Below in figure 2.1 is an illustration of the process of degumming divalent phosphatidic acids described below;

Table 2.1: Types of phospholipids in crude vegetable oil

Chemical Structure	IUPAC Name
 <p>General Structure of a Phospholipid R^3 = Phospholipid class.</p>	
 <p>Phosphatidic Acid, $R^3 = -OH$.</p>	
 <p>Phosphatidylcholine, $R^3 = -O-CH_2-CH_2-N(CH_3)_3$, 1,2 – diacyl – sn – glycerol (3) phosphocholine.</p>	
 <p>Phosphatidylethanolamine, $R^3 = -O-CH_2-CH_2-NH_3^+$ 1,2 – diacyl – sn – glycerol (3) phosphoethanolamine.</p>	
 <p>Phosphatidylinositol $R^3 = -C_6H_{12}O_6$ 1,2 – diacyl – sn – glycerol (3) phospho – L – myoinositol.</p>	

(Marilyn, 2002)

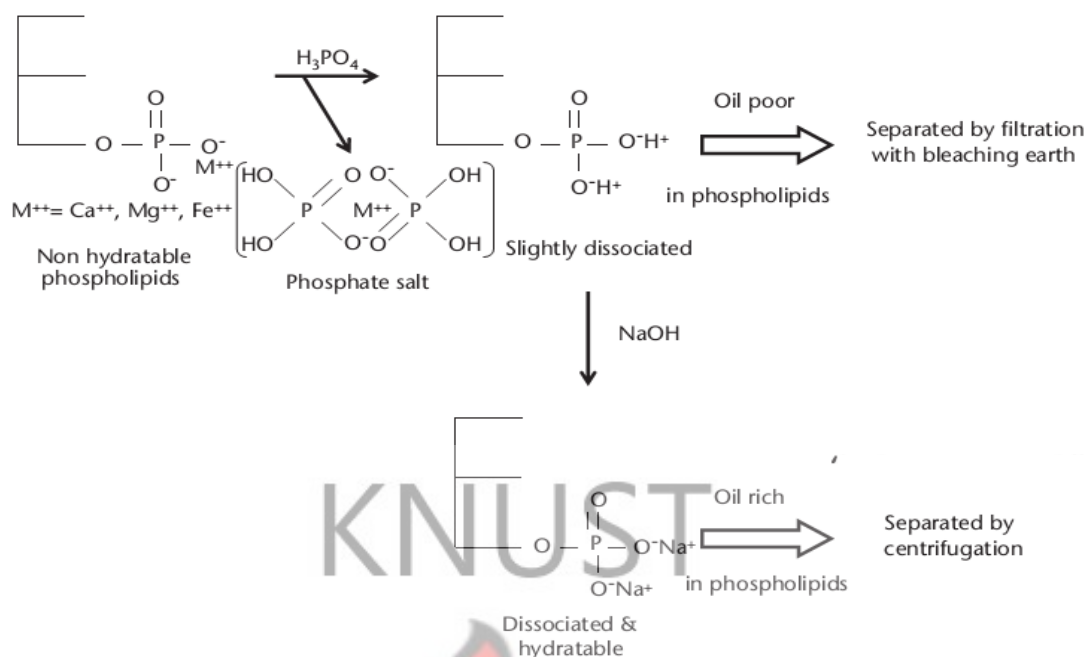


Figure 2.1: Chemical reaction routes illustrating the removal of divalent phosphatic acids (Deffense *et al.*, 2009).

2.3 The Kinetics of Degumming Process

The hydratability of phosphatides revealed that the molecular structure determines the type of phospholipid that remains in the oil phase or move to the water phase when contacted with water. Further illustration shows that the molecular structure indicates the condition of the medium that best precipitate the phospholipids in the oil (pH of the medium). Hence there should be no residues left after subjecting the crude oil to specific conditions as illustrated above. Unfortunately, this is not the case, because after degumming vegetable oils still contains appreciable amounts of phospholipids (Dijkstra *et al.*, 1989).

This indicates that there are other factors involved in the degumming of crude vegetable oil apart from the hydratability properties of the phospholipids. The degummed vegetable oil still containing appreciable

amount of phospholipid indicates the process of degumming has not reached the equilibrium state of the reaction process. This occurs because the process was not given enough time for the reaction to reach its equilibrium state. Due to this fact when the partially degummed vegetable oils are again subjected to a degumming treatment, the residual phosphorus content continues to reduce. However, the reaction time is not the only factor involved but also the interface between the phospholipids in the oil and the water soluble degumming agent. The diffusional distance towards the interface are other factors affecting hydration kinetics of a degumming process (Dijkstra, 2011).

In other words the attribute can be referred to as the degree of dispersion of the degumming agent in the oil. A reasonable dispersion will provide an oil/water interface that hydrates phosphatides and migrate it into the water phase. In the case where the degumming agent is an acid, a much finer dispersion is required since the reaction is instantaneous. During degumming, the situation is further aggravated by the stability of the oil/water dispersion (Dijkstra *et al.*, 1989 and Mag *et al.*, 1980).

In the process of degumming, aqueous acid droplets will coalesce, the interface will decrease, diffusion distances will increase and reaction between the phospholipid and the degumming agent will slow down. Accordingly, the dispersion has to be fine so that the reaction between the acid and the non-hydratable phospholipids (NHP) is instantaneous (Dijkstra, 2011).

The kinetics and hydratability of degumming, forms the basic mechanism behind most degumming methods and separation of phosphatides from crude vegetable oil. Refinery of crude vegetable oil takes into consideration the source, quality and composition of the raw material, since this determines the procedures needed to refine the crude vegetable.

2.4 Factors affecting the Degumming Process of Crude Vegetable Oil

In the process of degumming, Pan *et al.*, (2001) and Eshratabadi *et al.*, (2008) postulated factors such as *concentration of degumming agent, temperature, agitation speed and contact time* of degumming as the main factors that affect the process of degumming. This was also confirmed by List *et al.*, (1981).

In the refinery, the quality of the crude vegetable oil is a basic requirement in the processing of oil. This is due to the fact that, a crude oil with bad quality will need more production utilities such as phosphoric acid and bleaching earth in order to refine the oil. Increase in the degree of dispersion increases the reaction area hence increasing the rate of the degumming reaction. A similar trend is expected with increasing the reaction time, degumming agent concentration and degumming agent-crude oil ratio. With the effect of temperature, the rate of reaction increase with increasing temperature but not beyond 100 °C (Deffense *et al.*, 2009 and Rohani, 2006).

2.5 Economics of Degumming

The economics of degumming is directly related to attributes of the degumming agent used during the process of degumming. Attributes such as the availability of the degumming agent, cost and efficiency of degumming method are the main factors that affect the economics of degumming. The losses which occur in the use of a degumming method is also predominant in the economics of degumming. The cost and maintenance involved in the procurement and running of the equipment used in the degumming process is also essential in the economics of degumming. In most cases the refiner usually evaluates the economics of a degumming process based on the price of the degumming agent used in the process.

Comparing the cost of the degumming agent ethylene diamine tetra acetic acid (EDTA) was the most expensive, the cost per tonne ranges between \$2000 to \$4000. The price of phosphoric acid ranges between \$1000 to \$2000 per tonne, electrolyte such as sodium chloride cost between \$100 to \$200 per tonne. In cases where sodium hydroxide is used the cost of sodium hydroxide ranges between \$500 to \$700 per tonne. During membrane degumming, the cost of a set of membrane ranges from \$30000 to \$100000 (Prayon Technologies, 2012)

Losses during degumming are due mainly to the emulsifying nature of the phospholipids present in the oil. The formation of soap when sodium hydroxide is used during the degumming process is also one of the factors affecting the losses entailed during degumming. Emulsion

formed during the process of degumming reduces the separating ability of the precipitated phospholipid in the degummed oil.

2.4 Types of Degumming

2.4.1 Water Degumming

Water degumming is the process of removing gums through precipitation by hydration, where water is used as a degumming agent. During the process water is added to the crude vegetable oil, agitated for short contact/reaction time and separated by centrifugation. This method is used when extracting gums for production of lecithin and for crude oil with minimum phosphorus content of 300 ppm (Oyebeket *et al.*, 2008). The process can be carried out in a batch or continuous process depending on the type of oil being degummed and amount of oil processed.

Water degumming involves the treatment of crude vegetable oil with 2 % v/v of soft water at temperatures range between 70 - 90 °C whilst stirring using a high shear mixer for about 20-30 min. The mixture is then centrifuged to separate the oil from the precipitated gum after which the two products are dried and sent to storage for further processing. This degumming method produces degummed oil containing 200 ppm residual phosphorus content (O'Brien, 2009 and Andrew, 2009). The degumming method is insufficient since it does not enhance the removal of non-hydratable phospholipids (NHP) from the crude vegetable oil (Eshratabadi *et al.*, 2008). Below in figure 2.2 is a flow chart of water degumming.

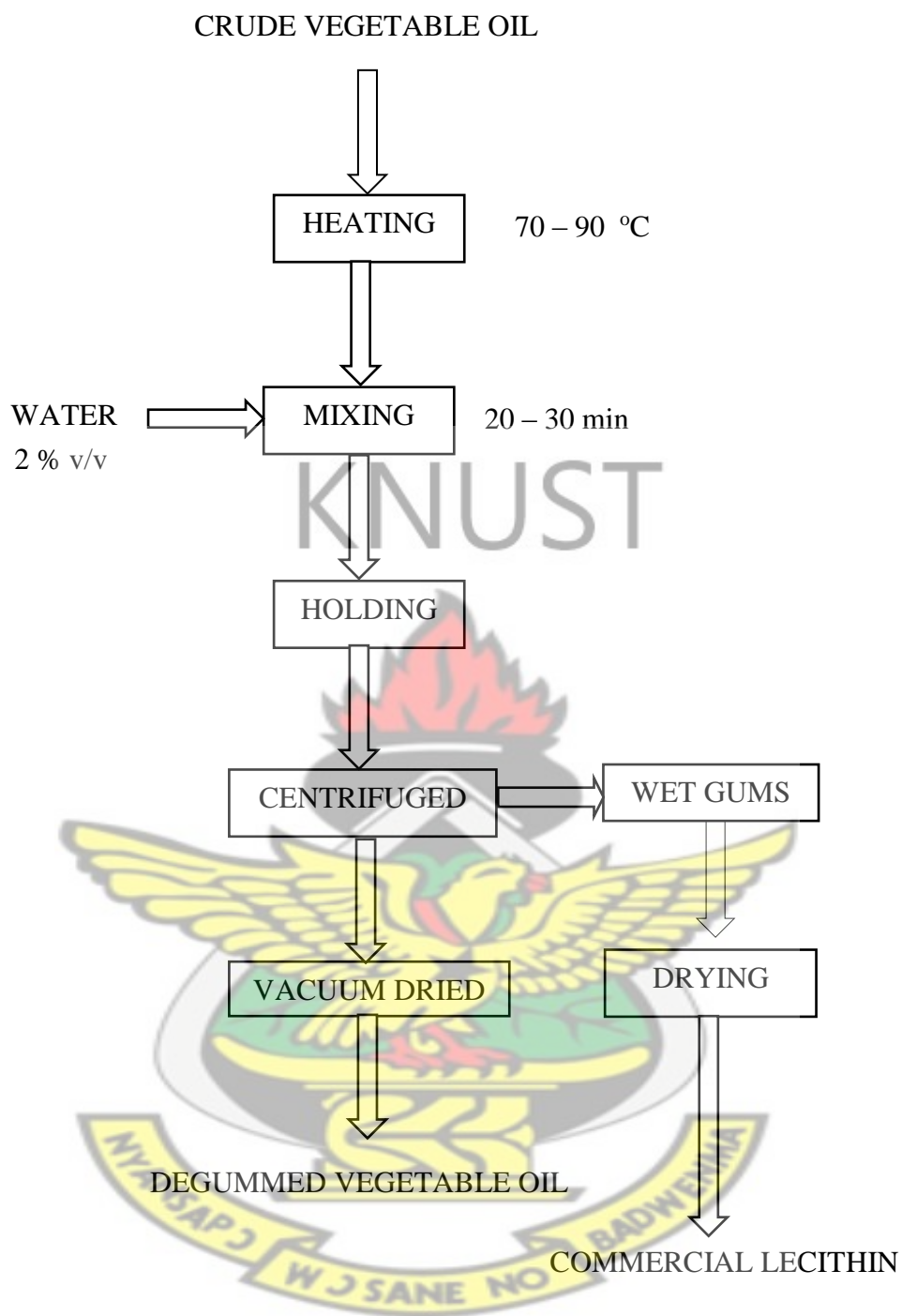


Figure 2.2: Flow diagram of water degumming process (Andrew, 2009).

2.4.2 Acid Degumming

Acid degumming can be considered as an alternative to water degumming, because it uses a highly concentrated acid as a degumming agent in precipitating the NHP present in crude vegetable oil. Crude vegetable oil either water degummed or not is treated using a concentrated acid, usually phosphoric, citric or malic acid. For oils containing relatively low amounts of non-hydratable phospholipid (e.g. sunflower oil, palm oil, water degummed soya bean oil) this process can lead to degummed oil with a lower residual phosphorus content (5 to 30 ppm) compared to water degumming (100 to 200 ppm) (Deffense, 2011 and Rohani, 2006).

In acid degumming, both phosphoric acid and citric acid forms a complex with divalent cations of non-hydratable phospholipid present in the crude vegetable oil in order to become hydratable. Separation is mainly by filtration as attached to the surfaces of the bleaching earth. This degumming method is mainly used for crude vegetable oils containing low NHP (O'Brien, 2009).

Acid degumming involves the treatment of crude vegetable oil with 0.05 % v/v of 85 % w/v food grade phosphoric acid at a maximum temperature of 100°C, whilst stirring using a high shear mixer for 15 to 30 min. The precipitated gum is then removed by centrifugation after which the oil is vacuum dried and then sent to storage for further processing (Andrew, 2009). The residual phosphorus content of degummed oil is usually below 30 ppm (Oyebek *et al.*, 2008). Due to high content of non-hydratable

phospholipids in some crude oils, acid degumming is not able to reduce the phosphorus content to the required level suitable for physical refining. (Copeland *et al.*, 2001). Figure 2.3 is a flowchart of acid degumming process depicting the various unit process that occur during degumming.

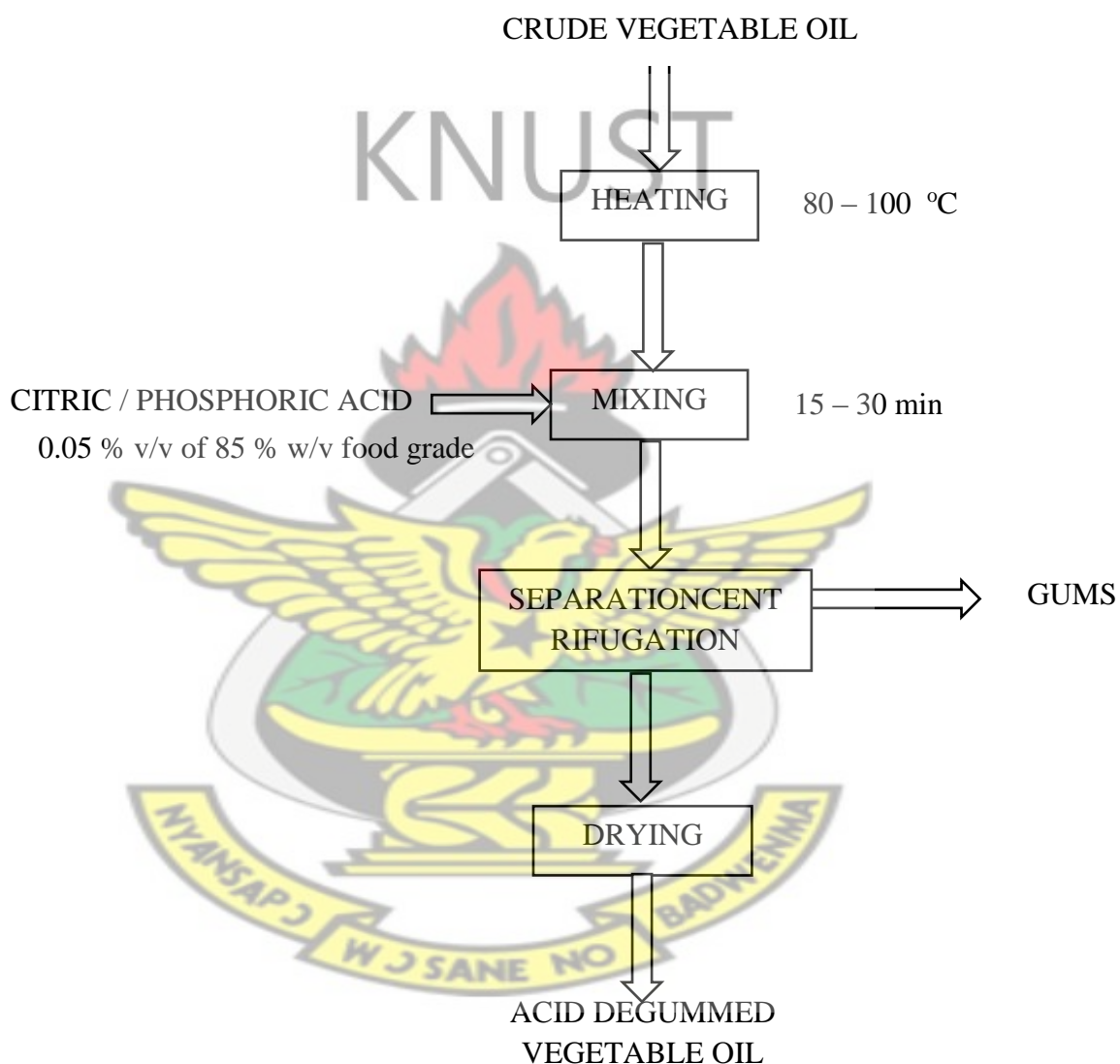


Figure 2.3: Flow diagram of acid degumming of crude oil (Andrew, 2009).

2.4.3 Enzymatic Degumming

Enzymatic degumming involves enzymes in the process of gum removal. In the degumming process the enzymes are the degumming agent for the degumming process, e.g. Lecitase 10L (pancreatic phospholipase A2) and Lecitase Novo (microbial lipase) are examples of enzymes used in the degumming method (Yang *et al.*, 2006 and Ji-Guo *et al.*, 2006). In enzymatic degumming, the use of chemicals such as phosphoric acid and sodium hydroxide has a dual role in the hydratability of phospholipids. This also aids in the optimization of the enzyme performance regarding the pH of the oil-water emulsified mixture (Dijkstra, 2010).

In enzymatic degumming the crude oil is first heated temperature between 80 to 100 °C at moderate agitation, after which an acid and a base is added whilst agitation continues for about 30 min. The mixture is then cooled to temperature between 40 to 70 °C and emulsified for about 15 min. This has the benefit of allowing the formation of a stable emulsion required for the reaction and preventing unwanted saponification (Dixit *et al.*, 2010). The microbes producing enzyme are then added or dosed at concentrations between 30 to 200 ppm as calculated per the weight or volume of the oil flow (Yang *et al.*, 2006).

After 30 min of agitation at temperature between 40 to 60 °C, the mixture is held for 4 - 6 hours, after which 2 % v/v water is added to aid in precipitating the gums out of the oil by centrifugation. The centrifuged oil is then vacuum dried (70 °C, -480mmHg) and sent to storage for

further processing. The process is guaranteed to produce a degummed oil of residual phosphorus content less than 10 ppm (Galhardo *et al.*, 2011). In figure 2.4 is flowchart of enzymatic degumming depicting the various stages of the degumming method.

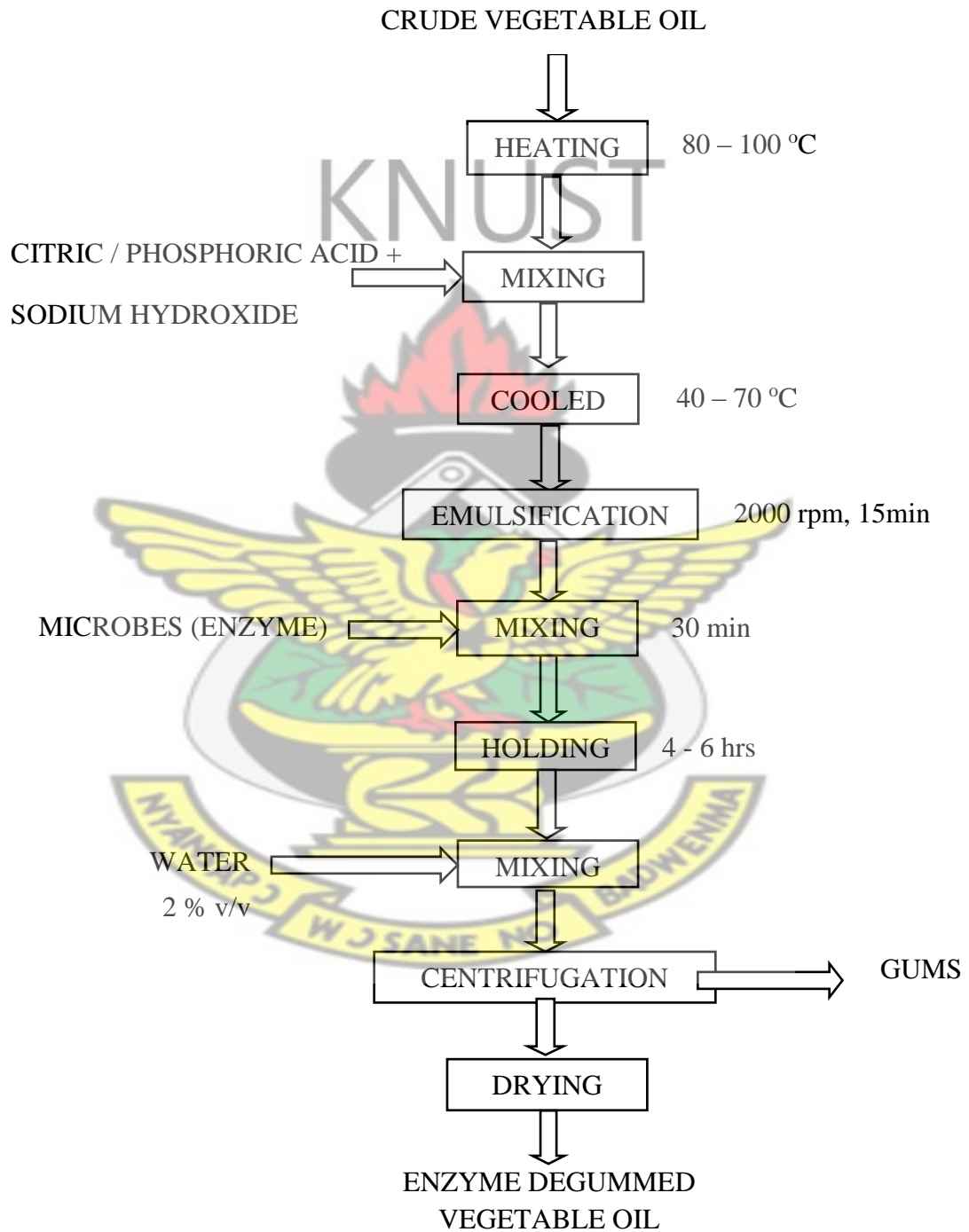


Figure2.4: Flow diagram of enzyme degumming.

In enzymatic degumming high cost equipment and maintenance is needed during the process, this is due to the high speed equipment used in the process. In order to obtain degummed oil with the requisite properties suitable for physical refinery, enzymatic degumming requires the minimum contact time 4 h (Yang *et al.*, 2006). Due to requirements listed above enzymatic degumming is not suitable for industrial purpose (Oybek *et al.*, 2008).

2.4.4 Membrane Degumming

Membrane degumming is a degumming process that utilizes the use of an organic synthetic membrane to filter micelle formed phospholipids present in the crude vegetable oil. Membrane filtration is a size-exclusion based pressure-driven process. Different components of the crude oil are separated according to the molecular weights or particle sizes of the impurity and on the interactions with other components of the oil (Subramanian *et al.*, 1997).

Membrane composition, temperature, pressure and velocity of flow are the main factors that affect the performance of membrane separation (Ribero *et al.*, 2008). This degumming process involves filtration of micelle formed by phospholipids at a maximum temperature of 60 °C using a pressure of 5 bar, with a volumetric flow rate of about 0.3 m³/h this conditions gives a retention of 77 % on the membrane used during the degumming process (Andras *et al.*, 2003). The degumming method is very efficient but the membrane used during this process is very costly.

2.4.5 Totaal Ontslijmings Process(TOP) Degumming

TOP is a Dutch acronym derived from “Totaal Ontslijmings Process” meaning total degumming process. TOP degumming is a process which involves the use of a concentrated acid and a base in the process of degumming. The method is applicable to both water degummed oil and raw crude vegetable oil. Similar to acid degumming, TOP degumming also utilize the use of an acid to decompose the NHP. In order to raise the pH of the medium, an alkali is added to neutralize partially the phosphatic acid present in the oil and make it hydratable (Deffense, 2011).

During TOP degumming, the acid is dosed at 0.02 % v/v of phosphoric-crude oil ratio at maximum temperature of 110°C at moderate agitation. After a sufficient contact time of 20 min a base (20 % w/v concentrated sodium hydroxide) is added to the mixture at 0.3 % v/v base-crude oil ratio as agitation continues for 45 min. The oil in the mixture is then removed by centrifugation and vacuum dried after which the oil is pumped to storage for further processing (Oybek *et al.*, 2008 and Andrew, 2009). During the process the acid initially breaks down the non-hydratable phospholipids metal complexes into insoluble metal salts.

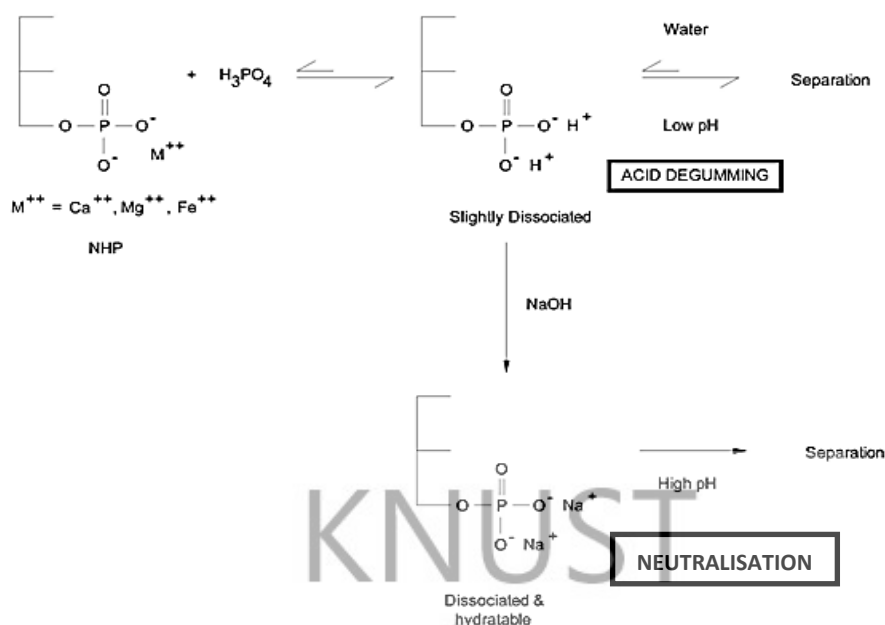


Figure 2.5: Chemical routes for TOP degumming (Deffense, 2009).

The non-hydratable phospholipid (phosphatic acid) formed is then hydrated by partial neutralization with the base and removed by centrifugation (Dijkstra *et al.*, 1989 and Deffense, 2011). In TOP degumming, the quality of the water-degummed oil is critical. When the water-degummed oil has a higher calcium/magnesium content, the method becomes less effective (Cleenewerck *et al.*, 1992). Consequently, TOP degummed oils usually contains lower residual phosphorus content, less than 10 ppm, and therefore suitable for physical refinery (deodorisation). This degumming method produces high effluent waste, since it involves the use of alkali and is expensive (O'Brien, 2009). The use of alkali also makes it unsuitable for crude vegetable oil with high content of free fatty acid (Oybek *et al.*, 2008). Figure 2.6 is a flowchart of TOP degumming illustrating the unit processes involved in the process.

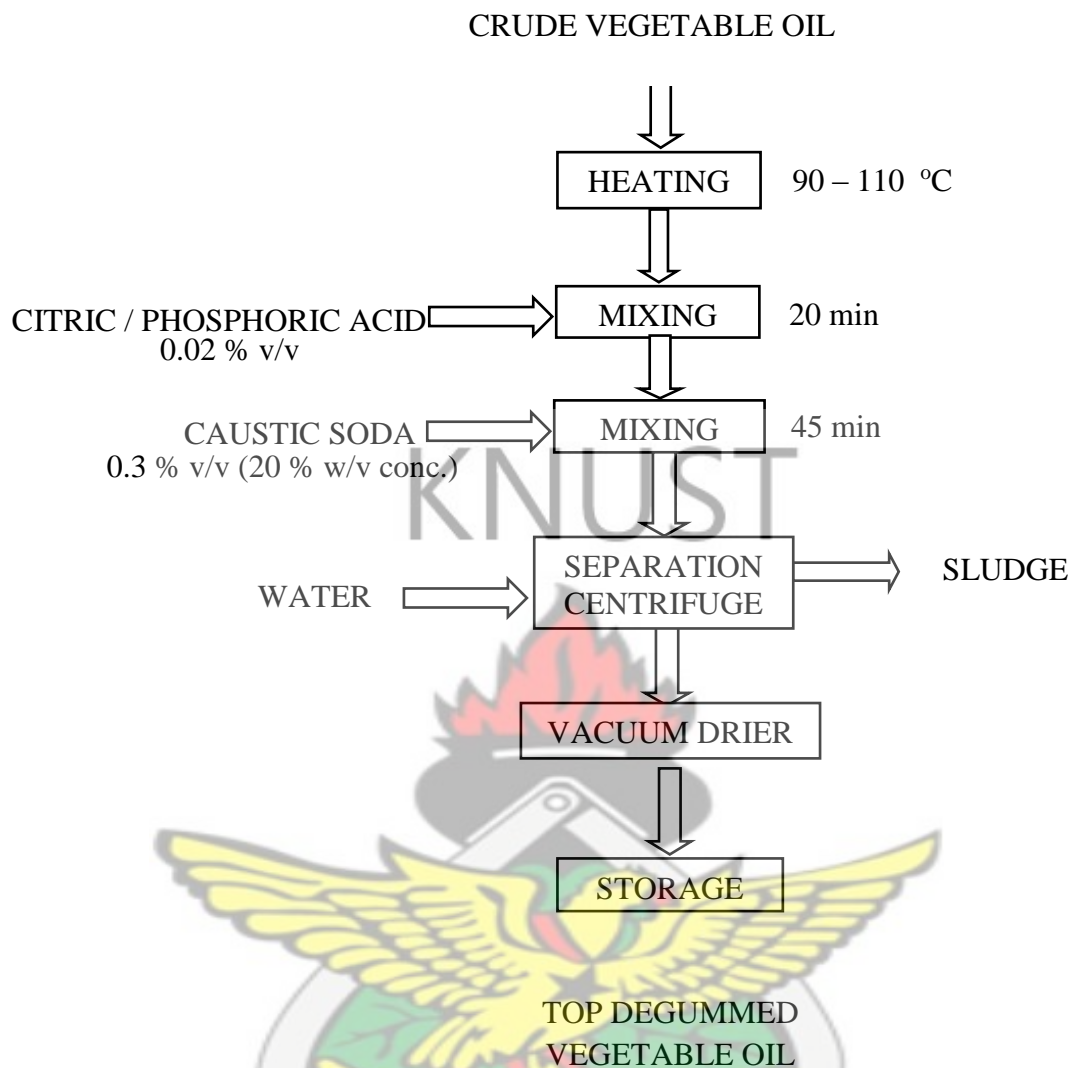


Figure 2.6: Flow diagram of TOP degumming (Andrew, 2009).

2.4.6 Soft Degumming

Soft degumming is a physico-chemical degumming process which involves elimination of phospholipids by the use of chelating agent. In this process ethylenediaminetetraacetic acid (EDTA) is used as degumming agent, in the presence of an emulsifying agent. In soft degumming, the NHP are decomposed by the chemical action of EDTA which has a much stronger affinity for divalent cations attached to PA (Ca^{2+} , Mg^{2+} and Fe^{2+}). When EDTA is added to oils, it forms a strong

complex or a 5-membered EDTA-Ca, Mg or Fe chelate as shown in **figure 2.7 (Deffense, 2011).**

Soft degumming consist of two stages: the first stage involves complexing, leading to increased hydration of phosphatidic acids of divalent cations (Ca^{+2} , Mg^{+2} and Fe^{+2}) and phosphatidylethanolamine. The second stage requires the transfer of the phospholipid into the aqueous phase forming an emulsion after which the mixture is decanted.

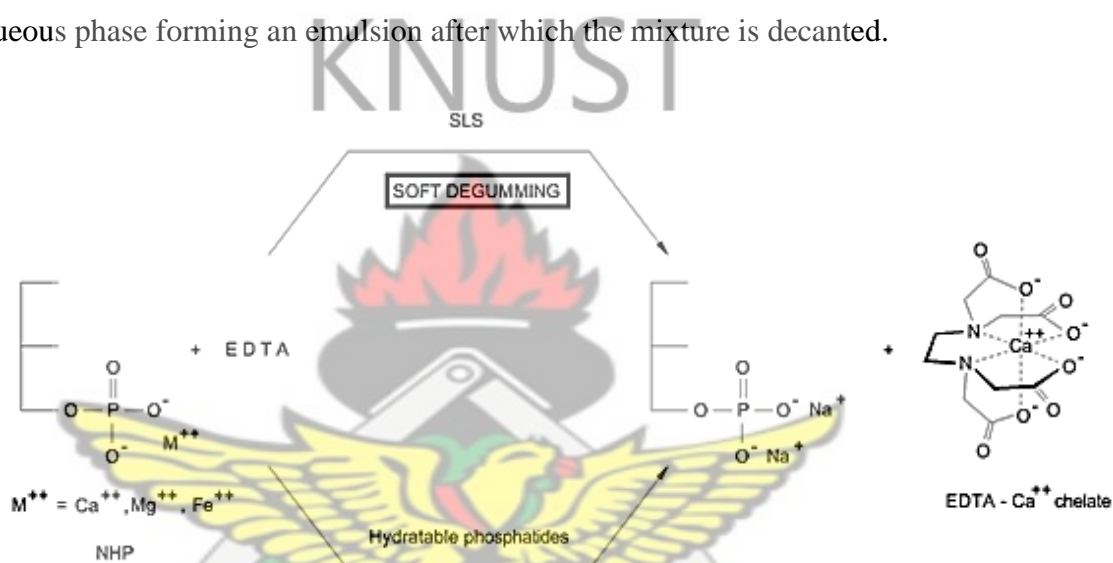


Figure 2.7: Chemical route of soft degumming (Deffense, 2011).

In the process of degumming, aqueous solution of chelating agent, EDTA or one of its salts is used to treat water-degummed oil. In the process ion exchange occurs between the oil-soluble ions (phospholipids) and the water-soluble EDTA. Soft degumming can only be efficient if the contact between the two phases is optimal. The formation of water-in-oil emulsion is indispensable and requires a special mixer designed for this purpose. In certain cases the emulsion is stabilized by adding an emulsifying agent during degumming (Choukri *et al.*, 2001).

The efficiency of the process depends on the degree of dispersion and contact time for the reaction between the chelating agent (EDTA) and NHP. In most cases during degumming emulsifying agents such as sodium lauryl sulphate (SLS) and sodium dodecyl sulphate (SDS) are sometimes used to facilitate the contact between the NHP in the oil phase and EDTA in the water phase (Choukriet *al.*, 2001).

In soft degumming, crude vegetable oil is heated to a maximum temperature of 90°C at moderate agitation using a shear mixer, 5 % of an aqueous solution containing a chelating agent EDTA (100 mM) and an emulsifying additive (sodium dodecyl sulphate, 50mM) is added. The mixture is homogenized for 1 min using a high speed mixer (9500 rpm), after which agitation continues for 2 min using a high shear mixer at a lower speed. A centrifuge is employed in the separation of emulsion formed after degumming (Choukriet *al.*, 2001 and Oyebek *et al.*, 2006). The degumming process is highly efficient for most crude vegetable oils. Due to the high cost of EDTA and equipment used, this leads to high operating and product cost hence making soft degumming unsuitable for industrial use (Choukriet *al.*, 2001). In figure 2.8 is a flowchart depicting the various unit operations involved in soft degumming process.

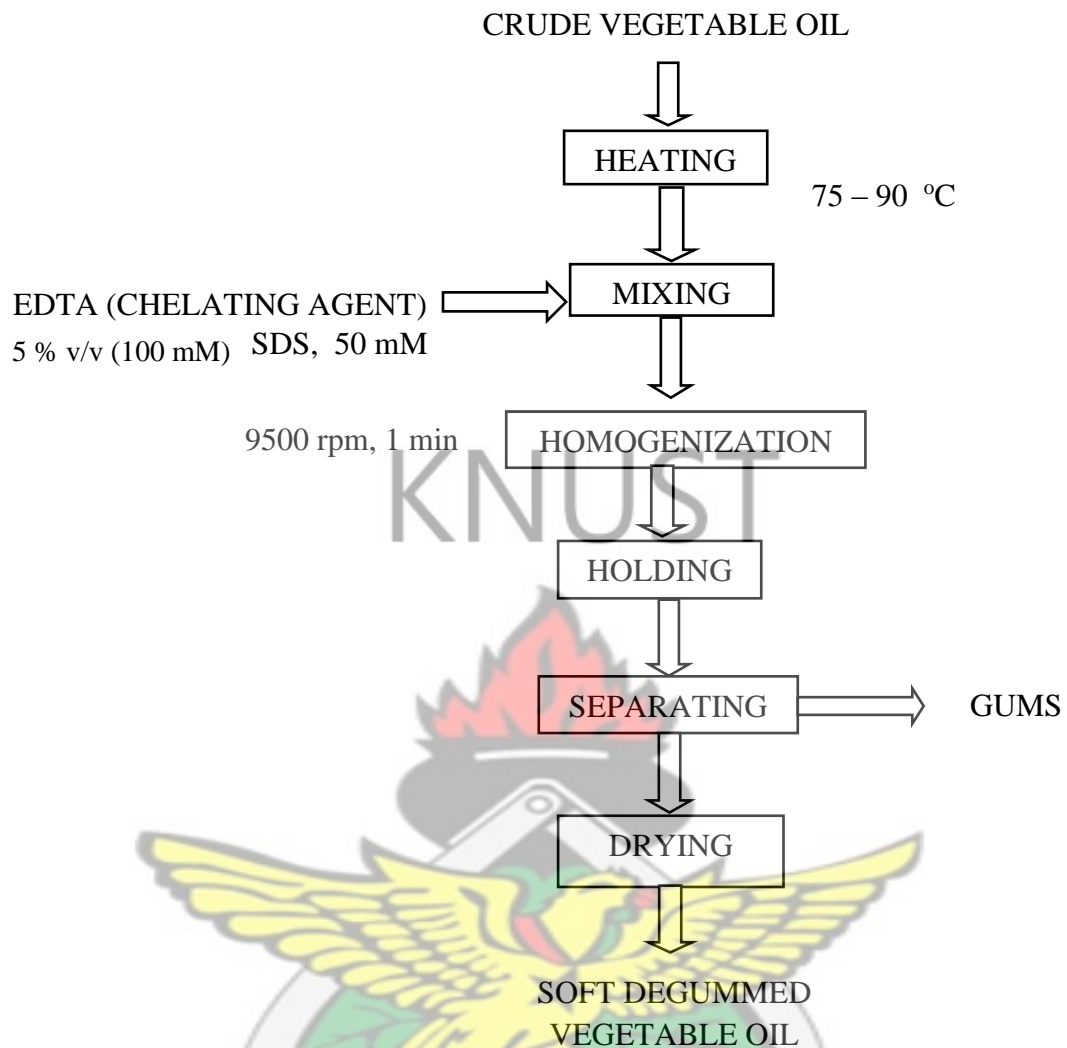


Figure 2.8: Flow diagram of soft degumming.

2.4.7 Super Critical Carbon Dioxide Degumming

This is a super critical fluid-based process that permits the counter current refinery treatment of crude vegetable oil to produce a degummed feedstock suitable for direct deodorization. The process makes use of a packed vessel that facilitates interfacial contact between the high-pressure carbon dioxide, and crude vegetable oil in a counter-current mode using a high-pressure liquid delivery pump (Marina *et al.*, 2012).

The degumming process involves contacting the crude oil with carbon dioxide at 55 MPa at a temperature of 70 °C within a contact time of 4 h/run for completion. Typically, the super-critical CO₂ refined oils can produce phosphorus content below 5 ppm, indicating most gums (99.2%) are removed (List *et al.*, 1993). Super critical carbon dioxide degumming process is very efficient but requires the use of high cost equipment and a well-trained technical human resource. The degumming process also requires a high contact time (4 h/run), hence not suitable for industrial degumming.

2.4.8 Electrolyte Degumming

Electrolyte degumming is a new degumming process that employs electrolytes in the removal of non-hydratable phospholipids from crude vegetable oil. However, when in combination with water degumming it removes both hydratable and non-hydratable gums. The electrolytes used contains potassium and sodium chlorides in the ratio of 95:5 (Nasirullah, 2005).

In this process crude vegetable oil is first heated to temperature between 80 - 110 °C, at moderate agitation the electrolyte is added (2 % v/v) as mixing continues for about 30 - 45 min. After degumming the oil is centrifuged at 2000 rpm to remove the precipitated gums, the residual phosphorus content of the degummed oil is less than 0.06 % v/v. This produces degummed oil with residual phosphorus content less than 10 ppm which is acceptable for physical refinery (Nasirullah, 2005). Electrolyte degumming is efficient but still needs further research to

determine the optimum conditions for the degumming process. In figure 2.9 is a flow diagram depicting the various unit operations in electrolyte degumming.

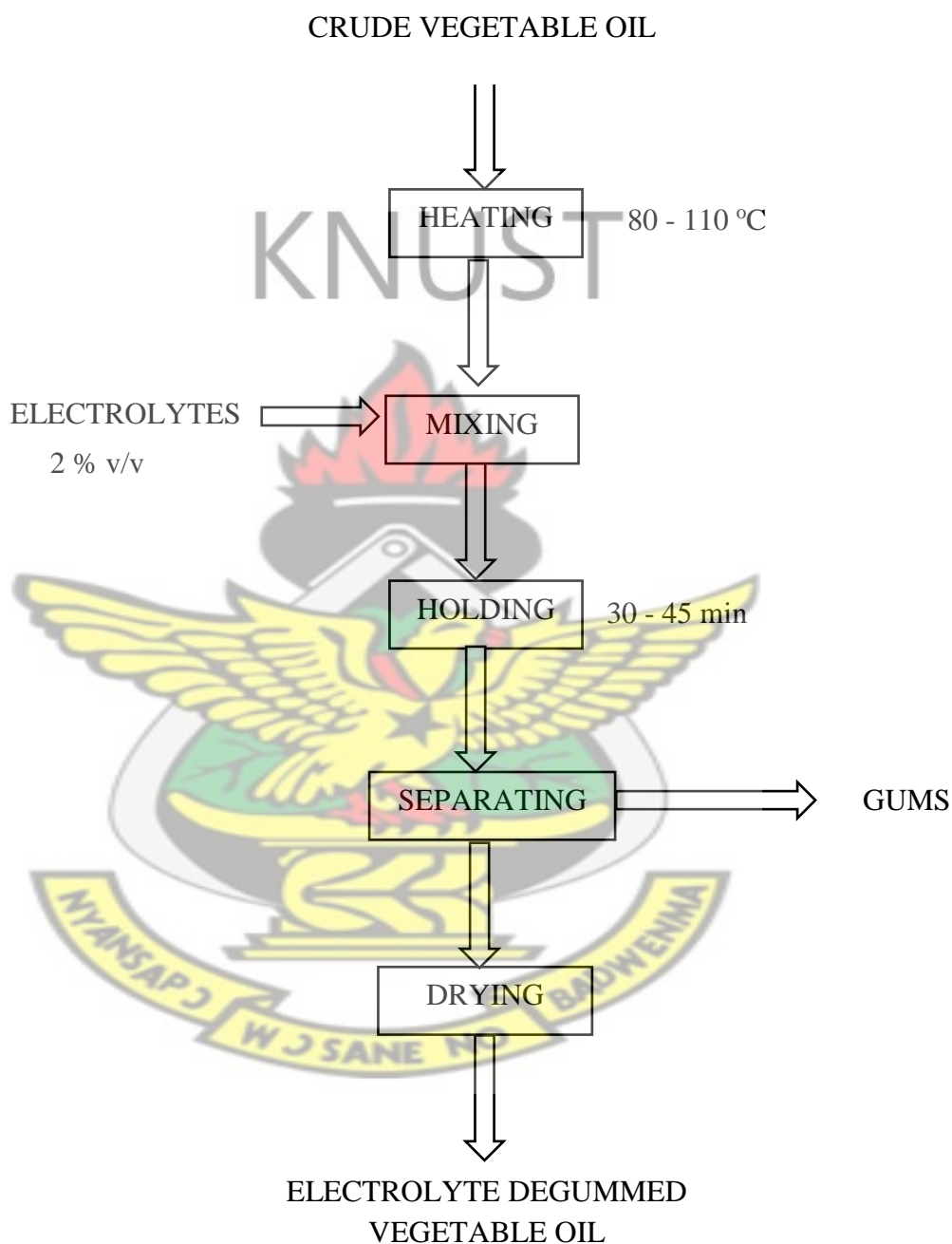


Figure 2.9: Flow sheet of electrolyte degumming

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Source of Materials

Crude soya bean oil was obtained from Ghana Nut Limited *Techiman* in the *BrongAhafo* Region Ghana for the research. All the chemicals used in the research were procured from Sigma-Aldrich Co. L. L. C (Sigma - Aldrich Corporate Office, 3050 Spruce, St. Louis, MO 63103).

3.2 Method

3.2.1 Treatment Method

In order to investigate the optimum conditions suitable to degum crude soya bean oil, a factorial design was used varying six factors at three levels each as presented in table 3.1. Design Expert 8.0.7 (2007) was used to randomize the factors indicating the specified levels of each factor. Below in table 3.1 are the factors with the levels of variation.

Table 3.1: Factors and levels of variation

Factors	Level of Variation
Temperature	50 – 80 °C
Time	30 – 60 min
Speed of Agitation	150– 300 rpm
Percentage Concentration of Electrolyte	0.5 – 3 % w / v
Percentage Electrolyte-to-Oil Ratio	1 – 5 % v / v
Electrolyte Combination	Sodium Chloride and Magnesium Chloride Potassium Chloride and Calcium Chloride

3.2.1.1 Electrolyte Solution

Each electrolyte combination was weighed in 50:50 ratio concentrations, for example looking at run 4 of table A1 (Appendix A), percentage electrolyte concentration was 3 % w/v, hence 1.5 g of potassium chloride and calcium chloride was weighed each into 100 ml volumetric flask, after which 70 ml of deionized water was added to dissolve the salt solution. More water was then added to the solution in order to meet the 100 ml mark.

3.2.2 The Degumming Treatment Process

This was done by measuring 100 ml of crude soya bean oil (CSBO) into the conical flask, and placing 38 by 8 mm spin bar in it. The setup was then placed on a Stuart magnetic stirrer (Model: SB162, Chelmsford - England) and the settings regulated to depict the specified conditions of each run as indicated in experimental design (Appendix A).

After degumming, each sample was centrifuged at 2000 rpm for 1 h to produce clear oils which was stored in a 200 ml plastic package. The degummed oil was then taken to the laboratory for phosphorus analysis.

3.2.3 Free Fatty Acid Determination

Free fatty acid content of the crude soya bean oil was determined by weighing 5g CSBO into an Erlenmeyer flask and adding 50 ml 95% neutralized alcohol using phenolphthalein as an indicator. The mixture was then titrated against 0.1 N sodium hydroxide until the color of the solution changed from golden yellow to pink (AOAC Official Method 940.28,

1997). The percentage free fatty acid content was calculated using the formula below:

$$\text{Percentage Free Fatty Acid Content} = \frac{\text{MW}(256) \times N \times V}{W}$$

N = Normality of sodium hydroxide (0.1 N or mol/1000ml)

V = Volume of sodium hydroxide titrated (ml)

W = Weight of sample (g)

MW = Molecular weight of fatty acid (g/mol)

3.2.4 Moisture Content Determination

Moisture content was determined by measuring 5g of crude soya bean oil was into a petri-dish and dried in the oven, at a temperature 105 °C for 6 h (AOAC Official Method 935.29, 1997). The mass loss was evaluated to determine the moisture content of the oil.

Mass of Petri-dish + lid = M₁

Mass of Petri-dish + sample + lid before drying = M₂

Mass of Petri-dish + sample + lid after drying = M₃

Percentage Moisture Content =

$$\frac{\text{Weight of Sample before drying} - \text{weight of sample after drying}}{\text{Weight of Sample before drying}} =$$

$$\frac{(M_2 - M_1) - (M_3 - M_1)}{(M_2 - M_1)}$$

3.2.5 Phosphorus Analysis

3.2.5.1 Preparation of Reagents

Sulphuric Acid, H_2SO_4 , 5N:

5N sulphuric acid was prepared by diluting 70 ml of concentrated sulphuric acid in 500 ml of distilled water.

Potassium Antimonyl Tartrate Solution($\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$):

Potassium antimonyl tartrate solution was prepared by dissolving 1.37 g solid of potassium antimonyl tartrate in 500 ml distilled water. The solution was stored in a glass stoppered bottle pending phosphorus analysis.

Ammonium Molybdate Solution:

In preparing the ammonium molybdate solution, 20 g of solid ammonium molybdate was weighed into a 500 ml volumetric flask, dissolved with 300 ml distilled water and made up to the 500 ml mark.

Ascorbic Acid, 0.1M:

0.1M solution of ascorbic acid was prepared by weighing 1.76 g of solid ascorbic into a 100 ml volumetric flask and dissolved with 100 ml distilled water up to the mark.

Combined Reagent:

The combined reagent was mixed in the following proportions: 50 ml 5N sulphuric acid, 5 ml potassium antimonyl tartrate solution, 15 ml ammonium molybdate solution, and 30 ml ascorbic acid solution. After each reagent was added the solution was mixed thoroughly.

Preparation of calibration curve

Approximately 1.05 g of anhydrous di-potassium hydrogen phosphate (KH_2PO_4) was weighed into a 1000 ml volumetric flask and diluted to 1000 ml mark,(this represents 1000 ppm or 1000 mg/l phosphate solution), Aliquots of the phosphate solution of 25 ml, 20 ml, 15 ml, 10 ml, and 5 ml, was transferred to a 50ml volumetric flask. Ten milliliter of the combine reagent was added and the solution made up to the 50 ml mark. Each mixture was mixed thoroughly and allowed stand for 30 min and the absorbance of the solution read using a spectrophotometer at 650 nm.

Wet Ashing

Wet ashing was done by measuring 50 ml of each sample in to a digestion flask and digested until the sample hardened. The burnt sample was allowed to cool to room temperature after which 10 ml of di-acid mixture of nitric acid (HNO_3) and perchloric acid (HClO_4) in the ratio 9:4 was added. Heating continued until a straw yellow colour solution was formed and the production of red brown NO_2 fumes ceased(Deloy, 1998 and Maurice, 2010). The ashed sample was cooled and filtered using Whatman paper (2), after which the volume was made up to the 50 ml mark. The resultant solution was then stored at 4 °C (AOAC Official Method 975.03, 1997).

3.2.5.2 Phosphorus Content Analysis

Phosphorus analysis was done by measuring 50 ml of the digested sample into a conical flask and mixed with 10 ml of the combine reagent. The solution was left to stand for 10 min after which the absorbance was measured using a spectrophotometer (Model: UV/VIS 160 Shimadzu Tokyo - Japan), at 650 nm (AOCS Official Method Ca 12 - 55). The phosphorus content was determined by means of standard curve using dipotassium hydrogen phosphate as a standard.

3.2.6 Data Analysis

Design Expert 8.0.7 (2007) was employed in the analysis of the data. The experiment employed a response surface methodology approach to predict the model that best fit the responses obtained from the experiment. The data obtained was processed to evaluate statistical coefficients such as regression, adjusted regression, predicted regression, and adequate precision. Analysis of variance was evaluated to identify the variations and significance of the various factors studied.

CHAPTER FOUR

4.0 RESULT AND DISCUSSION

In the refinery of crude vegetable oil, the properties of the raw material are essential in the processing of crude oil to produce a refined edible vegetable oil. Below in table 4.1 are the properties of crude soya bean oil that was used in the experiment;

Table 4.1: The properties of the crude soya bean oil

Characteristics of the Crude Oil	Expected Value	Actual Value
Free Fatty Acid content	5 % Max.	2.6 %
Colour(5¼ Lovibond cell)	10R	6.3R + 1B + 40Y
Moisture	0.2 % Max	0.12 %
Phosphorus Content	1200 ppm Max.	457 ppm

4.1 Data analysis

The experimental conditions with the corresponding responses from the experimental design are presented in appendix A (Table A1). The dependent(*residual phosphorus content of degummed crude soya bean oil*) and independent variables (*electrolyte concentration, electrolyte- oil ratio, temperature of medium, agitation speed, electrolyte combination and agitation time*) were analyzed to obtain a regression equation that predict the response within the given range. The regression equation for the electrolyte degumming was summarized as follows;

$$Y = \beta_0 + \sum_{i=1}^6 \beta_i^{(1)} x_i + \sum_{i=1}^6 \sum_{j=1}^6 \beta_{ij}^{(2)} x_i x_j + \sum_{i=1}^6 \sum_{j=1}^6 \sum_{k=1}^6 \beta_{ijk}^{(3)} x_i x_j x_k + \sum_{i=1}^6 \sum_{j=1}^6 \sum_{k=1}^6 \sum_{l=1}^6 \beta_{ijkl}^{(4)} x_i x_j x_k x_l$$

$i \leq j$ $i \leq j < k$ $i < j$ $k \leq i \leq j$

Y is the response of the experiment (*residual phosphorus content of the degummed oil*) $\beta_0, \beta_i, \beta_{ij}, \beta_{ijk}$ and β_{ijkl} are constants representing coefficient of linear, quadratic, cubic and quartic interaction terms,

respectively. x_i , x_j , x_k and x_l are coded independent variables of (*electrolyte concentration, electrolyte-oil ratio, temperature, agitation speed, time, and electrolyte combination*).

Looking at Appendix B residual diagnostic analysis, the residuals obtained using reduce quartic model revealed that approximately 75 % of the values were within -1 to +1 this indicate a good fit by the model used. Eighty percent of both the internal and external studentized residual were within -1 and 1, thus indicating that the predicted values using the model are in the same trend as depicted by actual value. The maximum Cook's distance of 0.5 indicates that the change if one of the cases is omitted is within range of the model.

The result obtained from the summary of the statistical analysis table 4.3, revealed R^2 of 0.993 and adjusted R^2 of 0.982, this indicates that the model is good at predicting variations of the responses obtained about the mean. Predicted R^2 of 0.904 indicates that the model has a good predictive capability. Adequate precision obtained (35.61) was greater than 4 indicating adequate model discrimination; hence the model could be used to navigate the design space. The p-value of the regression model was less than 0.001 and the F- value 89.77 (table 4.2), thus indicating that the model is significant. The lack of fit p-value of 0.88 and F- value of 0.51 (table 4.2) enhances the fact that the model does not lack fit and the model could adequately represent the experiment.

Table 4.2: Analysis of Variance for Response Surface Reduced Quartic Model

	Sum of		Mean	F-value	p-value
Source	Squares	Df	Square		
Model	31880.16	38	838.95	89.77	< 0.0001*
A-% w/v Electrolyte conc.	1535.14	1	1535.14	164.27	< 0.0001*
B-% v/v Electrolyte –to – oil	1200.56	1	1200.56	128.47	< 0.0001*
C-Agitation speed	17.20	1	17.20	1.84	0.187
D-Agitation Time	42.36	1	42.36	4.53	0.0433*
E-Temperature of Medium	218.05	1	218.05	23.33	< 0.0001*
F-Electrolyte Mix	7.11	1	7.11	0.76	0.3914
AB	1183.39	1	1183.39	126.63	< 0.0001*
AC	63.66	1	63.66	6.81	0.0151*
AD	398.69	1	398.69	42.66	< 0.0001*
AE	60.43	1	60.43	6.47	0.0176*
CE	256.37	1	256.37	27.43	< 0.0001*
A ²	768.17	1	768.17	82.20	< 0.0001*
B ²	935.56	1	935.56	100.11	< 0.0001*
E ²	51.42	1	51.42	5.50	0.0272*
ABF	82.07	1	82.07	8.78	0.0066*
ACF	68.49	1	68.49	7.33	0.0121*
AEF	63.86	1	63.86	6.83	0.0149*
BCF	71.82	1	71.82	7.69	0.0104*
DEF	35.15	1	35.15	3.76	0.0638*
A ² B	97.48	1	97.48	10.43	0.0035*
A ² C	51.04	1	51.04	5.46	0.0277*
A ² E	180.10	1	180.10	19.27	0.0002*
C ² F	62.37	1	62.37	6.67	0.0160*
E ² F	58.22	1	58.22	6.23	0.0195*
A ² B ²	187.79	1	187.79	20.09	0.0001*
A ² CF	42.20	1	42.20	4.52	0.0437*
AB ² F	42.42	1	42.42	4.54	0.0432*
Residual	233.63	25	9.35		
Lack of Fit	101.29	15	6.75	0.51	0.8842
Pure Error	132.34	10	13.23		
Cor Total	32113.79	63			

Table 4.3: ANOVA Statistic Summary

Std Dev.	Mean	C.V %	PRESS	R-Squared	Adj R-Squared	Pred R-Squared	Adeq Precision
3.06	23.62	12.94	3096.78	0.99	0.98	0.90	35.61

4.2 Phosphorus content of degummed oil

Figure 4.1 revealed a maximum residual phosphorus concentration of 71 ppm at 0.5 % w/v electrolyte concentration and at minimum electrolyte-oil ratio. The minimum residual phosphorus content of the degummed oil was at the maximum electrolyte concentration (3 % w/v) and electrolyte-oil ratio (5 % v/v). From the graph, as the electrolyte concentration increased from 0.5 to 3 % w/v with the electrolyte-oil ratio from 1 to 5 % v/v, the residual phosphorus content of the degummed oil decreased consistently. With reference to figure 4.1, increasing electrolyte concentration beyond 1.75 % w/v and electrolyte-oil ratio beyond 3.0 % v/v the residual phosphorus content was within minimum region (less than 10 ppm). From table 4.2 the p- value of A, B and AB were less than 0.05. This reveals that the electrolyte concentration and the electrolyte- oil ratio had a significant effect on the degumming experiment ($p < 0.05$).

From figure 4.1, it can be deduced that the minimum residual phosphorus content (< 10 ppm) of the experiment was within electrolyte concentration of 1.75 to 3.00 % w/v within 30 to 60 min. Generally, the residual phosphorus content of the degummed oil was decreasing with increase in the reaction time of the degumming process. This trend reveals that the electrolyte concentration and the contact time were significant, and the combine effect of both was also significant ($p < 0.05$).

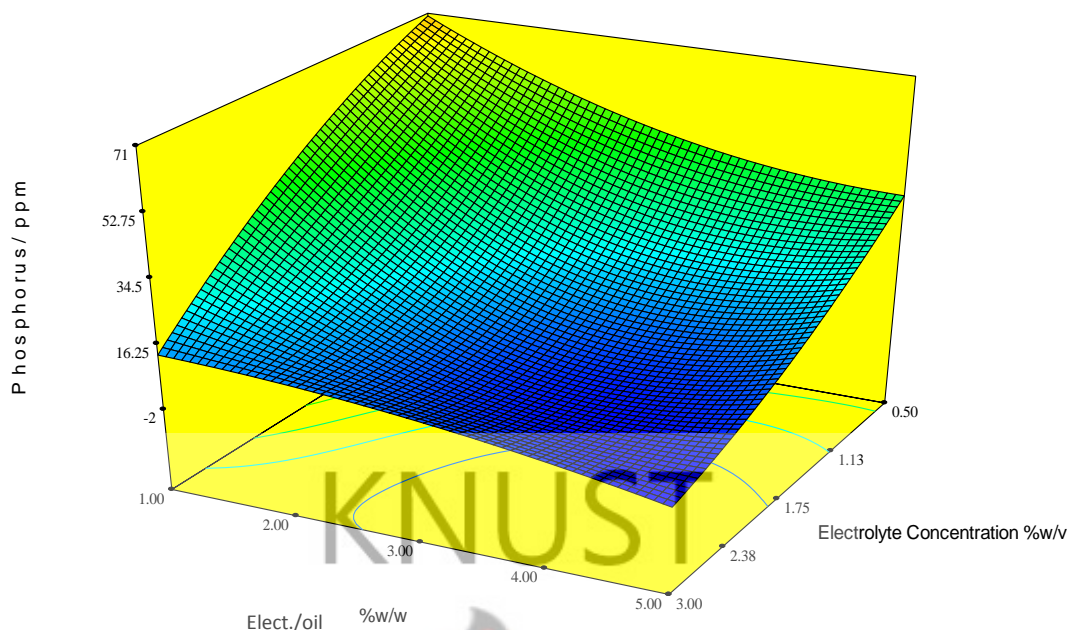


Figure 4.3: Response surface contour plot showing the variation of phosphorus content of degummed soya bean oil with the electrolyte-oil ratio and electrolyte concentration at 225 rpm within 65°C for 45 min using a mixture of sodium and magnesium chloride.

A similar trend was observed by Eshratabadi *et al* (2008), when crude soyabean oil was degummed. The minimum residual phosphorus content of the degummed oil was obtained at 0.2 % v/v acid-crude oil ratio at 85% concentration. As he explained, as acid-crude oil ratio increased from 0 to 0.2 %, the phosphatide removal efficiency increased from 30 % to 80 %. As the acid-crude oil ratio exceeds 0.2 % the inverse result occurred. The same trend was also observed by Pan (2001), where he investigated the effect of different variable related mainly to acid degumming.

Among the factors varied were the acid-crude oil ratio and the acid concentration, during degumming a 2.5 % v/v acid-crude oil ratio at concentration ranging from 8 to 13 % of the degumming agent was utilized during the process. As the concentration of the degumming agent increases, the phosphorus content of the degummed oil reduced consistently at 90 °C.

The lowest residual phosphorus content of degummed oil was at 13 % concentration, at a degumming temperature of 90 °C after 25 min. Pan (2001) reported that acid-crude oil ratio had a significant effect on the degumming experiment ($p < 0.05$).

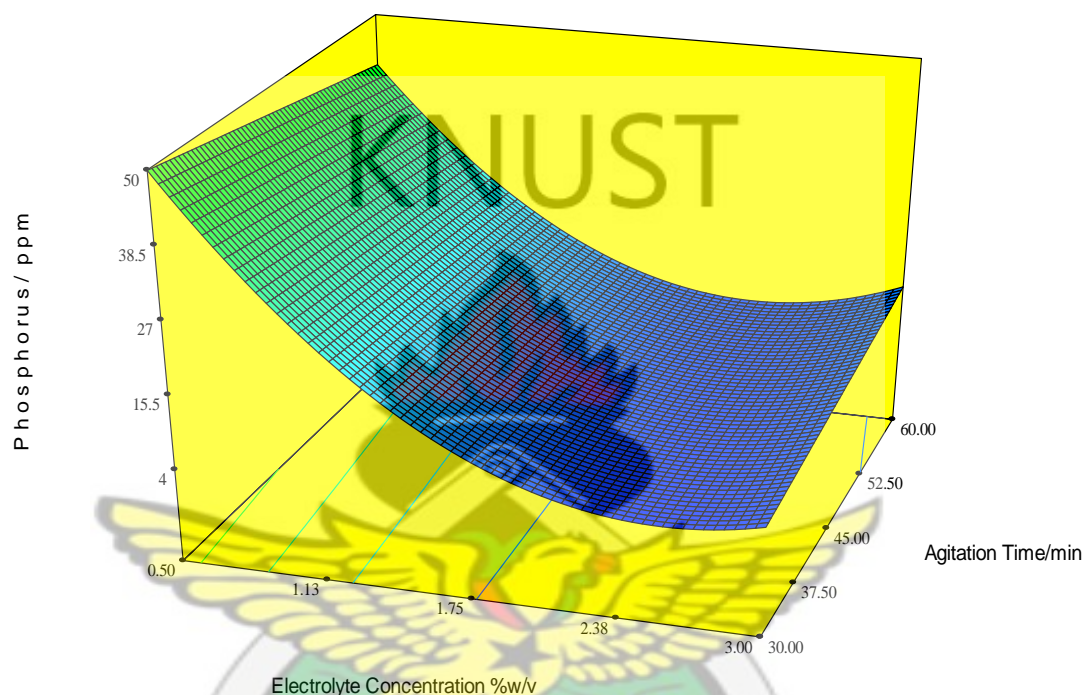


Figure 4.4: Response surface plot showing the variation of phosphorus content of degummed soya bean oil with the electrolyte concentration and agitation time agitated at 225 rpm for 45 min, at 65°C using a mixture of sodium and magnesium chloride.

Figure 4.2 represent the pattern of changes in the residual phosphorus content of degummed oil as affected by electrolyte concentration and agitation/reaction time. The illustration in figure 4.2 revealed a maximum residual phosphorus content of 50 ppm at 0.5 % w/v electrolyte concentration for 30 min. The minimum residual phosphorus content was observed at electrolyte concentration of 3 % w/v for 60 min.

From figure 4.2, it was observed that as the electrolyte concentration increased with corresponding increase of the reaction time, the residual phosphorus content of the degummed oil decreased steadily. Hence with respect to table 4.2 it is evident that both electrolyte concentration (A) and reaction time (D) had a significant effect on the degumming experiment ($p < 0.05$).

Eshratabadi *et al.*, (2008) had a similar trend in the case where the efficiency of degumming was varied with respect to time. Within the first 5 min of degumming at 85 °C using 0.2 % v/v phosphoric acid-crude oil ratio and 3 % v/v water – crude oil ratio, the efficiency of degumming was 64 %.

From 10 to 20 min the efficiency of degumming increased to 73 % and after 30 min as agitation continued for an hour resulted in only 4 % increase in degumming efficiency of the process. This is because at continued degumming beyond 30 min the reaction between phosphoric acid and the phospholipids has reached its equilibrium stage. Rohani (2006), had also observed a similar trend during the optimization of the degumming and bleaching processes of crude palm oil. As the phosphoric dosage increased from 0-0.2 % v/v, the residual phosphorus content of the degummed oil reduced drastically.

List *et al.*, (1981) also reported a similar trend in the degumming of soyabean oil to produce lecithin. He observed 91% lecithin recovery within the first 5 min of degumming. However as degumming continued for 1 h resulted in 3.8 % increase in the lecithin recovery. This was due to the instantaneous reaction of the degumming process which results in

precipitation of the NHP. Eshratabadi *et al* (2008), Rohani (2006) and List (1981) all indicated that the concentration and reaction time had a significant effect on the degumming rate of crude vegetable oil ($p < 0.05$).

Figure 4.3 illustrates the effect of electrolyte concentration and the temperature changes on the residual phosphorus content of the degummed oil. From the graph with reference to the statistical ANOVA table 4.2, it was deduced that both temperature and electrolyte concentration had a significant effect on the degumming experiment ($p < 0.05$). This is due to the curvilinear trend of electrolyte concentration and temperature of the medium as both increased within the specified limit. In figure 4.3, as the electrolyte concentration increases up to 3% w/v with increasing temperature from 50 °C to 80 °C, the residual phosphorus content of the degummed oil decreased steadily. The minimum residual phosphorus content region of the experiment was observed at electrolyte concentration between 1.75 % w/v to 3 % w/v and at a temperature between 65 °C - 80 °C. With the changing trends in figure 4.3 this indicates that both electrolyte concentration and temperature had a significant effect on the experiment ($p < 0.05$).

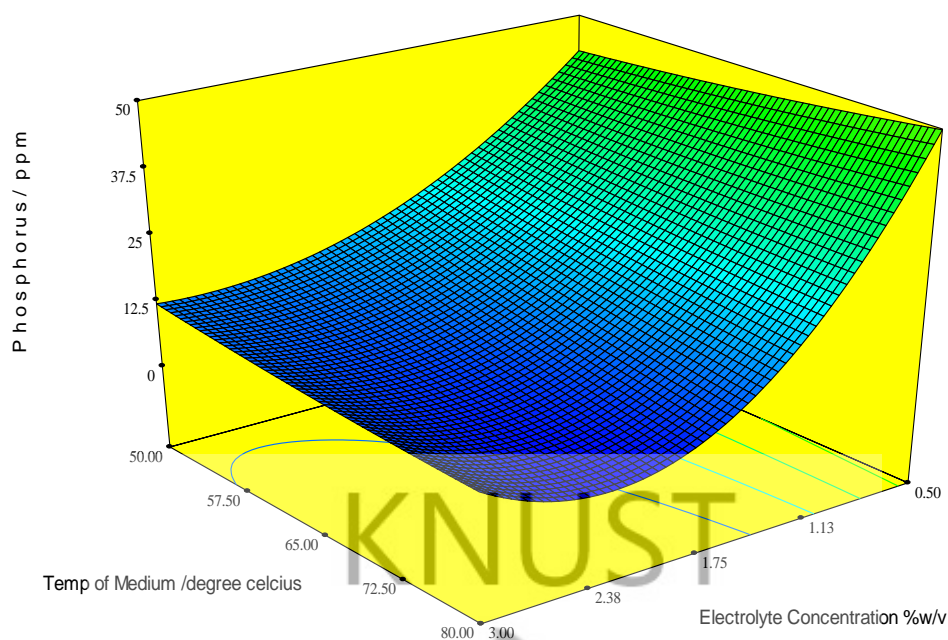


Figure 4.3: Response surface plot showing the variation of phosphorus content of degummed soya bean oil with the temperature and electrolyte concentration agitated at 225 rpm for 45 min, using electrolyte - oil ratio of 3 % v/v at a mixture of sodium and magnesium chloride.

Pan (2001) during acid degumming of crude soya bean oil recorded a similar trend. The residual phosphorus content of the degummed oil at 60 °C using acid concentration of 5 % gave minimum residual phosphorus content after 35 min for citric acid. A combined phosphoric and citric acid at 50:50 ratio gave the minimum residual phosphorus content after 25 min.

Eshratabadi *et al* (2008) also recorded a similar trend when the efficiency of degumming increased with increasing amount of phosphoric acid added during gum recovery. With increasing temperature, there was a decrease in the residual phosphorus content of the degummed oil within 65 °C to 90 °C. As the phosphoric acid-crude oil ratio increase from 0 to 2 % the residual phosphorus content of degummed oil reduces drastically.

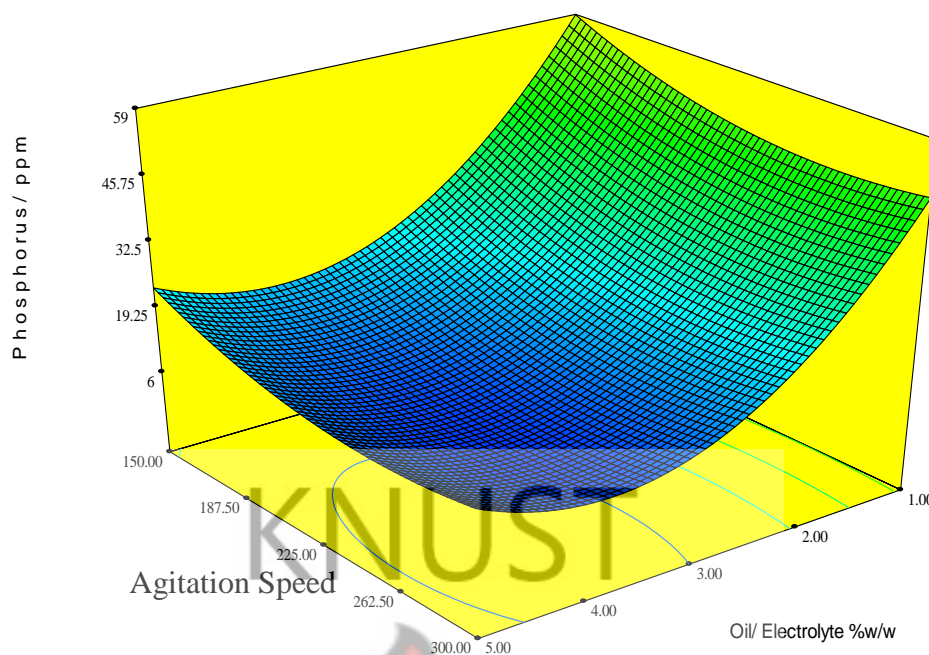


Figure 4.4: Response surface plot showing variation of phosphorus content of degummed soya bean oil with agitation speed and electrolyte – oil ratio at electrolyte concentration of 1.75 % w/v, at a temperature of 65°C for 45 min using a mixture of sodium and magnesium chloride.

The response surface graph in figure 4.4 illustrates the interactive effects of agitation speed and electrolyte-oil ratio on the residual phosphorus content of the degummed oil. The graph revealed that electrolyte-oil ratio demonstrated a curvilinear trend as it increases with increasing agitation speed of the medium. From table 4.3 it can be deduced that the agitation speed did not have a significant on the experiment ($p > 0.05$). In combination with the electrolyte-oil ratio, the agitation speed had a significant effect on the experiment ($p < 0.05$, table 4.2). As the agitation speed increased from 150 rpm to 300 rpm with increasing electrolyte-oil ratio from 1 % v/v to 5 % v/v the residual phosphorus content reduced drastically. From figure 4.4, the minimum phosphorus content was within the electrolyte-oil ratio from 3 % v/v to 5 % v/v with agitation speed range between 290 rpm to 300 rpm.

According to List (1981) and Pan (2001) during the degumming of crude soya bean oil, they both reported that the agitation speed was statistically insignificant and had no effect on the rate of degumming ($p > 0.05$).

Pan (2001) noted that varying the concentration of the degumming agent, temperature, and the reaction time at moderate degree of dispersion, had a significant effect on degumming of soya bean oil. This is because when acid is used as an agent and dispersed, finely in the crude oil an instantaneous reaction occurs (Dijkstra *et al.*, 1989).

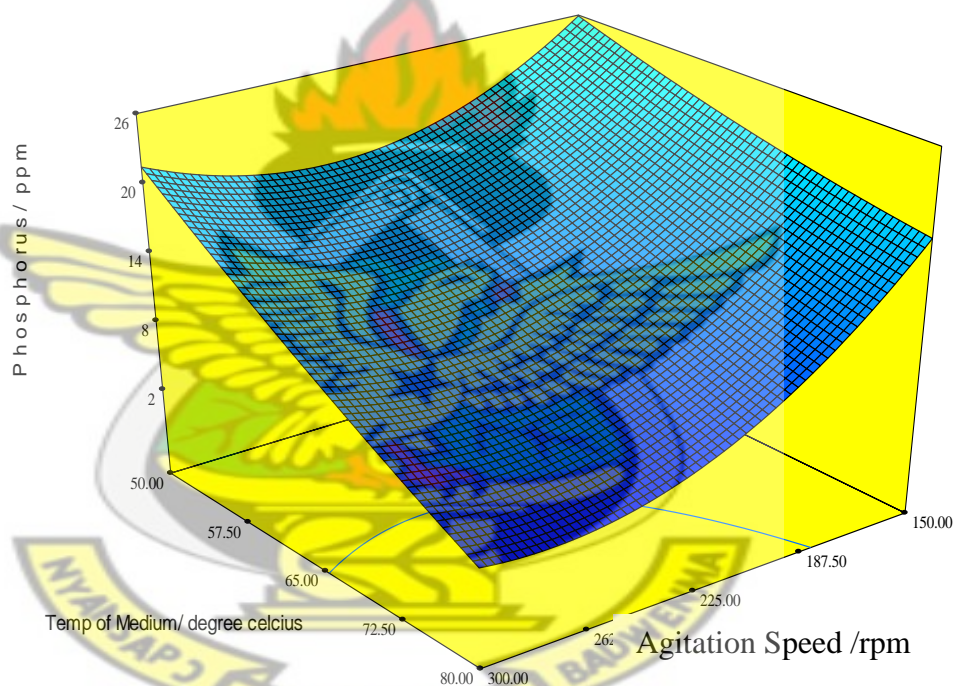


Figure 4.5: Response surface plot showing variations in phosphorus content of degummed soya bean oil at electrolyte concentration of 1.75 % w/v using electrolyte -oil ratio of 3 % v/v for 45 min using a mixture of sodium and magnesium chloride.

With reference to figure 4.5 it can be observed that as the temperature increases with increasing agitation speed the residual phosphorus content of the degummed oil reduced linearly. From figure 4.5 it can be evaluated that the temperature of the medium had a significant effect on the residual

phosphorus content of the degummed oil. From table 4.2, it can be deduced that the agitation speed was statistically insignificant ($p > 0.05$) but when combine with temperature had a significant effect on the residual phosphorus content of the degummed oil ($p < 0.05$).

Investigating the effect of the factors involved in the degumming experiment, table 4.2 revealed that factors A, B, D, and E were significant during the experiment. Looking at the quadratic interactional effects of the factors combine, factors AB, AC, AD, AE, A^2 , B^2 , E^2 and CE were found to be statistically significant ($p < 0.05$). Considering three factor effect interactions it was observed that the combine factor effect of ABF, ACF, AEF, BCF, A^2B , A^2C , A^2E , C^2F and E^2F were statistically significant ($p < 0.05$). Looking at the trend, when factor F interacted with other two factors (three factor effect) it was found to have a significant effect on the response of the experiment ($p < 0.05$). In the four-factor effect the combined doubled effect interaction of factor A and B (A^2B^2) was significant ($p < 0.05$) so was A^2CF and AB^2F .

In order to validate the optimum condition generated using reduced quartic model, the optimum condition was based on constrains of producing a degummed oil of residual phosphorus content less than 10 ppm ($Y =$ residual phosphorus content of degummed oil, $Y < 10$ ppm). Design Expert 8.0.7 (2007) was used to evaluate the optimum condition at the highest desirability of minimizing the residual phosphorus content of the degummed oil based on the factors varied.

Electrolyte degumming using electrolyte combination of sodium and magnesium chloride at concentration of 2.4% w/v and electrolyte-oil ratio of 3.9 % v/v, with a maintained condition at 68 °C and 235 rpm for 37 min gave an average residual phosphorus content of 4.0 ppm after validation (Appendix D).

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CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Crude vegetable oil was degummed to a minimum phosphorus content of 4 ppm at optimum condition of 68 °C, agitated at 237 rpm for 37 min using an electrolyte mix containing sodium and magnesium chloride. The concentration of the combination was at 2.4 % w/v and at electrolyte-oil ratio of 4 % v/v, this satisfies the condition for physically refined oil to give a refined product of good quality. Compared to acid degumming, electrolyte degumming is an inexpensive method due to the low utility cost, low energy requirement, low contact time and operating cost. Electrolyte degumming is efficient and suitable for crude vegetable oil with high content of gums.

5.2 RECOMMENDATION

On a laboratory scale electrolyte degumming was efficient compared to acid degumming. It is recommended that further research be performed using electrolyte degumming on a pilot scale imitating the specified optimize conditions on an industrial scale.

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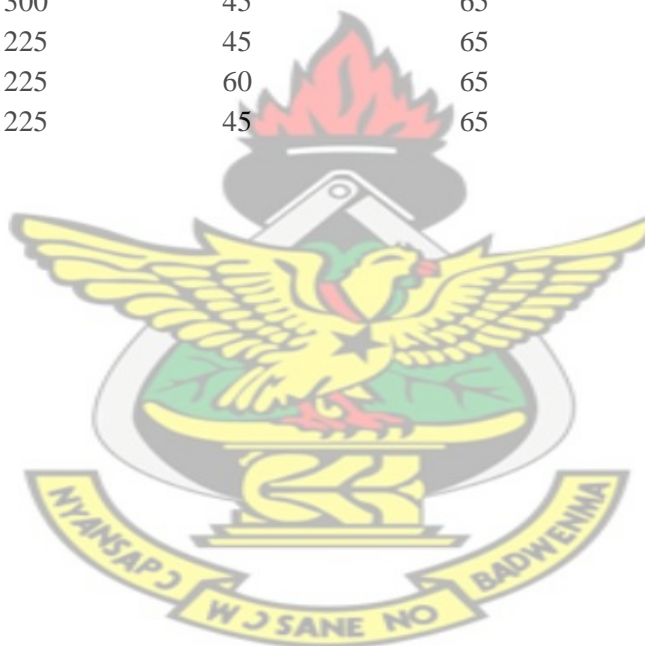
APPENDIX A: EXPERIMENTAL DESIGN

Table A1 : Experimental design with corresponding response after treatment

Run	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Factor 6	Response (ppm)	
	A:Electrolyte Concentration %	B:Oil/ Electrolyte Ratio %w/w	C:Agitation Speed Rpm	D: Time Agitation Minute(s)	E: Temp of Medium Degree Celsius	F:Electrolyte Combination	Actual Value	Actual Value
1	1.75	3	300	45	65	Na Mg	11.02	11.57
2	1.75	3	225	45	65	Na Mg	12.22	11.09
3	1.75	3	225	45	80	Na Mg	5.84	5.21
4	3.00	1	150	60	80	K Ca	9.37	9.63
5	1.75	1	225	45	65	Na Mg	46.69	48.06
6	0.50	5	150	60	80	Na Mg	39.83	40.87
7	3.00	5	150	60	50	K Ca	1.37	2.07
8	3.00	3	225	45	65	K Ca	4.06	3.51
9	1.75	3	150	45	65	K Ca	8.27	7.72
10	1.75	5	225	45	65	Na Mg	13.86	13.61
11	3.00	1	150	30	50	Na Mg	16.05	12.65
12	0.50	1	150	30	80	K Ca	72.40	72.72
13	1.75	3	225	45	65	Na Mg	6.71	11.09
14	0.50	5	300	30	80	Na Mg	51.36	52.73
15	1.75	3	225	45	50	Na Mg	16.56	18.29
16	0.50	1	300	60	80	Na Mg	74.12	72.74
17	3.00	1	300	30	80	Na Mg	16.05	15.78
18	0.50	1	300	30	50	K Ca	79.89	77.09
19	3.00	1	150	30	50	K Ca	2.19	2.27
20	1.75	3	150	45	65	Na Mg	21.01	21.56
21	0.50	1	150	60	50	K Ca	59.05	59.07
22	3.00	5	300	30	50	Na Mg	5.48	7.67

23	3.00	5	300	60	80	K Ca	4.32	3.08
24	1.75	1	225	45	65	K Ca	43.26	41.90
25	3.00	1	150	60	80	Na Mg	16.76	19.82
26	1.75	3	225	45	65	K Ca	5.58	10.02
27	0.50	1	300	60	80	K Ca	55.71	58.85
28	3.00	5	150	60	50	Na Mg	6.32	6.74
29	0.50	1	150	60	50	Na Mg	65.78	66.17
30	1.75	3	225	45	65	Na Mg	6.23	11.09
31	1.75	3	225	30	65	Na Mg	12.01	11.65
32	1.75	3	225	45	65	K Ca	10.47	10.02
33	1.75	3	225	45	65	K Ca	13.09	10.02
34	3.00	5	300	60	80	Na Mg	5.65	3.52
35	3.00	5	150	30	80	K Ca	2.52	1.75
36	1.75	3	225	45	50	K Ca	27.33	25.60
37	0.50	5	150	60	80	K Ca	30.98	28.14
38	0.50	5	300	60	50	Na Mg	44.79	43.48
39	3.00	1	300	60	50	Na Mg	31.87	31.80
40	0.50	1	300	30	50	Na Mg	78.81	79.85
41	1.75	5	225	45	65	K Ca	6.80	7.05
42	1.75	3	225	60	65	Na Mg	12.06	10.53
43	0.50	5	150	30	50	Na Mg	37.65	36.67
44	0.50	3	225	45	65	K Ca	45.98	45.43
45	1.75	3	225	45	65	K Ca	8.18	10.02
46	1.75	3	225	45	65	Na Mg	10.23	11.09
47	3.00	5	150	30	80	Na Mg	2.41	2.05
48	0.50	5	300	60	50	K Ca	20.76	21.62
49	0.50	5	300	30	80	K Ca	24.87	23.94
50	1.75	3	225	30	65	K Ca	12.37	11.63
51	3.00	5	300	30	50	K Ca	5.87	7.04
52	1.75	3	225	45	65	K Ca	10.69	10.02

53	0.50	3	225	45	65	Na Mg	45.30	45.85
54	0.50	5	150	30	50	K Ca	32.27	35.05
55	0.50	1	150	30	80	Na Mg	87.46	86.73
56	1.75	3	225	45	65	Na Mg	16.93	11.09
57	3.00	1	300	60	50	K Ca	25.96	24.87
58	1.75	3	225	45	80	K Ca	8.53	9.15
59	1.75	3	225	45	65	Na Mg	15.67	11.09
60	3.00	1	300	30	80	K Ca	11.80	13.23
61	1.75	3	300	45	65	K Ca	9.96	9.41
62	3.00	3	225	45	65	Na Mg	8.86	9.41
63	1.75	3	225	60	65	K Ca	7.70	8.41
64	1.75	3	225	45	65	K Ca	8.74	10.02



APPENDIX B: DIAGNOSTIC ANALYSIS

Table B1: DIAGNOSTIC ANALYSIS (Residual Analysis)

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residual	Externally Studentized Residual	DFFITS	Cook's Distance
1	11.02	11.57	-0.55	0.96	-0.91	-0.91	-4.50	0.52
2	12.22	11.09	1.13	0.10	0.39	0.38	0.13	0.00
3	5.84	5.21	0.62	0.74	0.40	0.39	0.66	0.01
4	9.37	9.63	-0.26	0.74	-0.17	-0.16	-0.27	0.00
5	46.69	48.06	-1.36	0.74	-0.87	-0.87	-1.46	0.05
6	39.83	40.87	-1.04	0.74	-0.66	-0.66	-1.09	0.03
7	1.37	2.07	-0.70	0.74	-0.45	-0.44	-0.73	0.01
8	4.06	3.51	0.55	0.96	0.91	0.91	4.50	0.52
9	8.27	7.72	0.55	0.96	0.91	0.91	4.50	0.52
10	13.86	13.61	0.26	0.74	0.16	0.16	0.27	0.00
11	16.05	12.65	3.41	0.74	2.17	2.36	3.93	0.34
12	72.40	72.72	-0.32	0.74	-0.20	-0.20	-0.33	0.00
13	6.71	11.09	-4.38	0.10	-1.51	-1.55	-0.53	0.01
14	51.36	52.73	-1.37	0.74	-0.87	-0.87	-1.45	0.05
15	16.56	18.29	-1.73	0.74	-1.11	-1.11	-1.87	0.09
16	74.12	72.74	1.38	0.74	0.88	0.87	1.46	0.05
17	16.05	15.78	0.27	0.74	0.17	0.17	0.28	0.00
18	79.89	77.09	2.80	0.74	1.78	1.87	3.11	0.23
19	2.19	2.27	-0.08	0.74	-0.05	-0.05	-0.08	0.00
20	21.01	21.56	-0.55	0.96	-0.91	-0.91	4.50	0.52
21	59.05	59.07	-0.03	0.74	-0.02	-0.02	-0.03	0.00

22	5.48	7.67	-2.19	0.74	-1.39	-1.42	-2.37	0.14
23	4.32	3.08	1.24	0.74	0.79	0.78	1.30	0.04
24	43.26	41.90	1.36	0.74	0.87	0.87	1.46	0.05
25	16.76	19.82	-3.07	0.74	-1.95	-2.08	-3.46	0.27
26	5.58	10.02	-4.44	0.10	-1.53	-1.58	-0.53	0.01
27	55.71	58.85	-3.14	0.74	-2.00	-2.14	-3.56	0.28
28	6.32	6.74	-0.42	0.74	-0.27	-0.27	-0.44	0.01
29	65.78	66.17	-0.38	0.74	-0.24	-0.24	-0.40	0.00
30	6.23	11.09	-4.86	0.10	-1.68	-1.75	-0.59	0.01
31	12.01	11.65	0.35	0.16	0.13	0.12	0.05	0.00
32	10.47	10.02	0.45	0.10	0.16	0.15	0.05	0.00
33	13.09	10.02	3.07	0.10	1.06	1.06	0.36	0.00
34	5.65	3.52	2.13	0.74	1.35	1.38	2.30	0.13
35	2.52	1.75	0.76	0.74	0.49	0.48	0.80	0.02
36	27.33	25.60	1.73	0.74	1.11	1.11	1.87	0.09
37	30.98	28.14	2.84	0.74	1.81	1.90	3.17	0.23
38	44.79	43.48	1.31	0.74	0.83	0.83	1.38	0.05
39	31.87	31.80	0.07	0.74	0.05	0.05	0.08	0.00
40	78.81	79.85	-1.04	0.74	-0.66	-0.65	-1.09	0.03
41	6.80	7.05	-0.26	0.74	-0.16	-0.16	-0.27	0.00
42	12.06	10.53	1.53	0.16	0.54	0.54	0.23	0.00
43	37.65	36.67	0.98	0.74	0.62	0.62	1.03	0.03
44	45.98	45.43	0.55	0.96	0.91	0.91	4.50	0.52
45	8.18	10.02	-1.84	0.10	-0.63	-0.63	-0.21	0.00
46	10.23	11.09	-0.86	0.10	-0.30	-0.29	-0.10	0.00
47	2.41	2.05	0.36	0.74	0.23	0.23	0.38	0.00
48	20.76	21.62	-0.86	0.74	-0.55	-0.54	-0.90	0.02

49	24.87	23.94	0.93	0.74	0.59	0.58	0.97	0.02
50	12.37	11.63	0.74	0.16	0.26	0.26	0.11	0.00
51	5.87	7.04	-1.17	0.74	-0.75	-0.74	-1.23	0.04
52	10.69	10.02	0.67	0.10	0.23	0.23	0.08	0.00
53	45.30	45.85	-0.55	0.96	-0.91	-0.91	-4.50	0.52
54	32.27	35.05	-2.78	0.74	-1.77	-1.85	-3.09	0.22
55	87.46	86.73	0.72	0.74	0.46	0.45	0.75	0.02
56	16.93	11.09	5.83	0.10	2.02	2.16	0.73	0.01
57	25.96	24.87	1.09	0.74	0.69	0.68	1.14	0.03
58	8.53	9.15	-0.62	0.74	-0.40	-0.39	-0.66	0.01
59	15.67	11.09	4.57	0.10	1.58	1.63	0.55	0.01
60	11.80	13.23	-1.43	0.74	-0.91	-0.90	-1.51	0.06
61	9.96	9.41	0.55	0.96	0.91	0.91	4.50	0.52
62	8.86	9.41	-0.55	0.96	-0.91	-0.91	-4.50	0.52
63	7.70	8.41	-0.71	0.16	-0.25	-0.25	-0.11	0.00
64	8.74	10.02	-1.28	0.10	-0.44	-0.43	-0.15	0.00

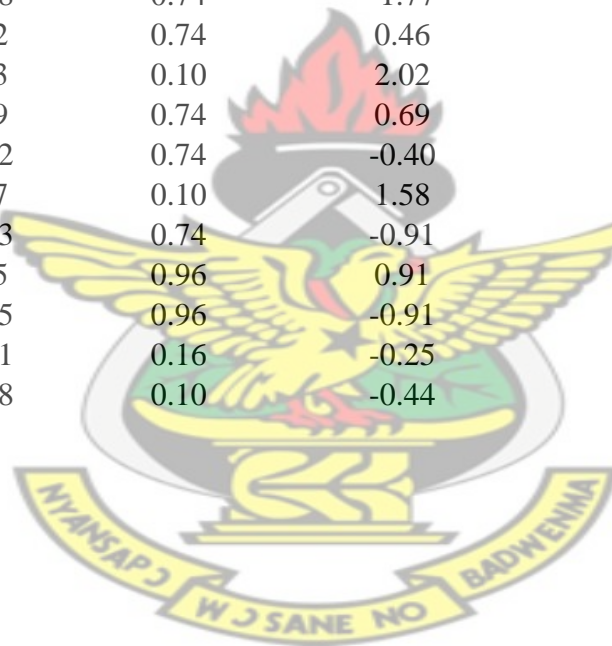


Table B2: Calibration Curve

Standard Concentration (mg/L)	Mean Absorbance
2.0	0.030
5.0	0.187
8.0	0.372
10.0	0.445
15.0	0.672

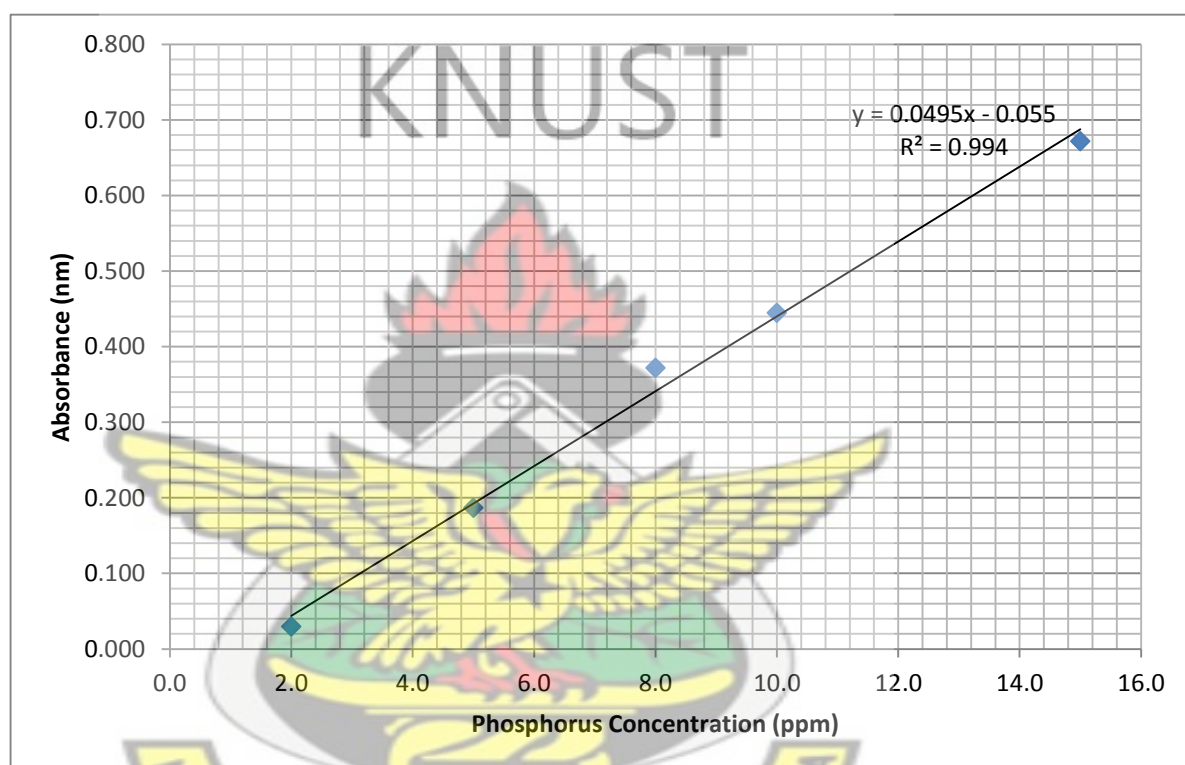


Figure B2: Calibration curve for phosphorus determination

APPENDIX C: Model Equation of Degumming

F1: Model equation of degumming experiment

Phosphorus =

$$\begin{aligned}
 &10.56 \\
 &-19.59 * A \\
 &-17.33 * B \\
 &-2.07 * C \\
 &-1.09 * D \\
 &-7.38 * E \\
 &-0.54 * F \\
 &6.08 * A * B \\
 &1.41 * A * C \\
 &3.53 * A * D \\
 &-1.37 * A * E \\
 &-1.37 * A * F \\
 &-1.11 * B * C \\
 &-0.10 * B * F \\
 &-2.83 * C * E \\
 &2.92 * C * F \\
 &-0.93 * D * E \\
 &-0.52 * D * F \\
 &-0.84 * E * F \\
 &15.49 * A^2 \\
 &17.10 * B^2 \\
 &2.01 * C^2 \\
 &4.01 * E^2 \\
 &1.60 * A * B * F \\
 &1.46 * A * C * F \\
 &1.41 * A * E * F \\
 &-1.50 * B * C * F \\
 &1.05 * D * E * F \\
 &5.24 * A^2 * B \\
 &3.79 * A^2 * C \\
 &7.12 * A^2 * E \\
 &-1.04 * A^2 * F \\
 &-2.03 * A * B^2 \\
 &-2.64 * B^2 * F \\
 &-3.46 * C^2 * F \\
 &3.35 * E^2 * F \\
 &-17.30 * A^2 * B^2 \\
 &-3.45 * A^2 * C * F \\
 &3.45 * A * B^2 * F
 \end{aligned}$$

The equation can also be written in the form;

Equation 1 : Model Equation of Degumming

Phosphorus =

$$10.56 - 19.59 A - 17.33 B - 2.07 C - 1.09 D - 7.38 E - 0.54 F + 6.08 A B + 1.41 A C + 3.53 A D - 1.37 A E - 1.37 A F - 1.11 B C - 0.10 B F - 2.83 C E + 2.92 C F - 0.93 D E - 0.52 D F - 0.84 E F + 15.49 A^2 + 17.10 B^2 + 2.01 C^2 + 4.01 E^2 + 1.60 A B F + 1.46 A C F + 1.41 A E F - 1.50 B C F + 1.05 D E F + 5.24 A^2 B + 3.79 A^2 C + 7.12 A^2 E - 1.04 A^2 F - 2.03 A B^2 - 2.64 B^2 * F - 3.46 C^2 F + 3.35 E^2 F - 17.30 A^2 B^2 - 3.45 A^2 C F + 3.45 A B^2 F$$

If the factors are represented as show in **table 9**:

Factors	Proposed Letters
A Electrolyte Concentration	x ₁
B Electrolyte –to - Oil Ratio	x ₂
C Agitation Speed	x ₃
D Agitation Time	x ₄
E Temperature of the Medium	x ₅
F Electrolyte Combination	x ₆
Phosphorus	y, f(x)

Equation 2: Modified Coded Equation of Model

Phosphorus (Y) =

$$10.56 - 19.59 x_1 - 17.33 x_2 - 2.07 x_3 - 1.09 x_4 - 7.38 x_5 - 0.54 x_6 + 6.08 x_1 x_2 + 1.41 x_1 x_3 + 3.53 x_1 x_4 - 1.37 x_1 x_5 - 1.37 x_1 x_6 - 1.11 x_2 x_3 - 0.10 x_2 x_6 - 2.83 x_3 x_5 + 2.92 x_3 x_6 - 0.93 x_4 x_5 - 0.52 x_4 x_6 - 0.84 x_5 x_6 + 15.49 x_1^2 + 17.10 x_2^2 + 2.01 x_3^2 + 4.01 x_5^2 + 1.60 x_1 x_2 x_6 + 1.46 x_1 x_3 x_6 + 1.41 x_1 x_5 x_6 - 1.50 x_2 x_3 x_6 + 1.05 x_4 x_5 x_6 + 5.24 x_1^2 x_2 + 3.79 x_1^2 x_3 + 7.12 x_1^2 x_5 - 1.04 x_1^2 x_6 - 2.03 x_1 x_2^2 - 2.64 x_2^2 x_6 - 3.46 x_3^2 x_6 + 3.35 x_5^2 x_6 - 17.30 x_1^2 x_2^2 - 3.45 x_1^2 x_3 x_6 + 3.45 x_1 x_2^2 x_6$$

Equation 3: Model Equation of Degumming Model

$$Y = \beta_0 + \sum_{i=1}^6 \beta_i^{(1)} x_i + \sum_{i=1}^6 \sum_{\substack{j=1 \\ i \leq j}}^6 \beta_{ij}^{(2)} x_i x_j + \sum_{i=1}^6 \sum_{\substack{j=1 \\ i \leq j < k}}^6 \sum_{k=1}^6 \beta_{ijk}^{(3)} x_i x_j x_k + \sum_{i=1}^6 \sum_{\substack{j=1 \\ i < j}}^6 \sum_{k=1}^6 \sum_{\substack{l=1 \\ k \leq i \leq j}}^6 \beta_{ijkl}^{(4)} x_i x_j x_k x_l$$

Y is the response of the experiment residual phosphorus content of the

degummed oil. $\beta_0, \beta_i, \beta_{ij}, \beta_{ijk}, \beta_{ijkl}$ are the constant, coefficient of intercept,

linear, quadratic, cubic and quartic interaction terms, respectively. x_i, x_j

and x_k are coded independent variables (electrolyte concentration,

electrolyte to oil ratio, temperature, agitation speed, time, and electrolyte combination).

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APPENDIX D: Validation

Table D1: Validation Results

SAMPLE ID	Absorbance			Response (ppm)			Avg. Conc.
	Ab 1	Ab 2	Ab 3	(ppm)	(ppm)	(ppm)	(ppm)
V 1	0.241	0.234	0.237	4.244	4.089	4.156	4.163
V 2	0.221	0.232	0.236	3.800	4.044	4.133	3.993
V 3	0.239	0.236	0.238	4.200	4.133	4.178	4.170

From Appendix D the average residual phosphorus content of the degummed oil is approximately 4 ppm.

Comparing electrolyte degumming to the industrially acid degumming, acid degumming at the following condition (3 % v/v water, 0.2 % v/v of phosphoric acid, at temperature 90°C and agitation time 30 min using a high shear mixer) the residual phosphorus in the acid degummed vegetable oil gave 15.4 ppm, which greater than 4 ppm of the electrolyte degumming.

