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Investigation of phospholipid separation from soybean oil for biodiesel production

Naveen Kekre
Iowa State University

Investigation of phospholipid separation from soybean oil for biodiesel production

By

Naveen Kekre

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J. Adin Mann, Co-Major Professor
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TABLE of CONTENTS

1. INTRODUCTION

The world currently relies heavily on fossil fuels; coal, oil, and natural gas for its energy. Fossil fuels are *nonrenewable*, that is, they draw on finite resources that will eventually dwindle, becoming too expensive or too environmentally damaging to retrieve. In contrast, *renewable energy* resources are constantly replenished and will never run out. Renewable energy is an alternative source of supplying the energy needs of the world. Alternative fuels, also known as non-conventional fuels, are any materials or substances that can be used as a fuel, other than conventional fossil fuels. The definition of Alternative Fuel varies according to the context of its usage. In the context of petroleum substitutes, the term 'alternative fuel' can imply any available fuel or energy source, and does not necessarily refer to a source of renewable energy. In the context of environmental sustainability, 'alternative fuel' often implies an ecologically benign renewable fuel.

In the United States, as per ASTM standards, pure biodiesel (B100) is considered to be an alternative fuel under the Energy Policy Act of 1992. Biodiesel refers to a diesel-equivalent, processed fuel derived from biological sources (such as vegetable oils), which can be used in unmodified diesel-engine vehicles. It is thus distinguished from the straight vegetable oils (SVO) or waste vegetable oils (WVO) used as fuels in some diesel vehicles

In recent years, biodiesel fuels have received significant attention both as a possible renewable alternative fuel and as an additive to existing petroleum-based fuels. Besides just an additional fuel supply, biodiesel exhibits several advantages when compared to existing petroleum fuel. Many researchers have shown that exhaust particulate matter, unburned hydrocarbons, carbon monoxide, and sulfur levels are all significantly reduced when using biodiesel fuels [1,2]. Research in this field has also shown increases in the levels of oxides of nitrogen, primarily as a result of advanced injection timing with biodiesel. Considerable

research has been undertaken to understand the performance characteristics of biodiesel fuels as well as the methods used to produce them [3,4,5,6].

Another advantage of biodiesel is that it can be distributed using today's infrastructure, and its use and production are increasing rapidly. Fuel stations are beginning to make biodiesel available to consumers, and a growing number of transport fleets use it as an additive in their fuel. Biodiesel is generally more expensive to purchase than petroleum diesel but this differential may diminish due to economies of scale, the rising cost of petroleum and government tax subsidies.

The state of Iowa and Iowa State University through the Iowa Energy Center sponsors research regarding alternate energy and efficiency. The Iowa Energy Centers' Biomass Energy Conversion (BECON) facility in Nevada, Iowa houses the state's most innovative and collaborative biomass conversion projects for developing new fuels and chemicals from biomass and enhancing existing processes. The BECON facility plays a key role in the successful demonstration of biomass technologies to promote increased acceptance and implementation of these technologies.

The BECON biodiesel pilot plant was constructed primarily through the efforts of Mustafa Canakci in an effort to test various methyl ester production methods using high free fatty-acid feed stocks such as yellow and brown grease [7, 8]. The facility consists of storage tanks for commercially purchased soybean oil and animal fats, a 9-gallon pretreatment reactor and recirculating heat pipe used for esterification of animal fat, two open top settling tanks for pretreated oil, an open top 75-gallon main reactor for the transesterification process, a 120-gallon cone-bottom tank for gravity separation of glycerin and washing, two finished fuel storage tanks, and the necessary plumbing.

Later on, to develop this research facility as an effective demonstration tool, several additions were made. An on-site soybean oil processing facility for the production of

soybean oil feedstock was installed [8]. A new main reactor system capable of investigating the effects of high temperature and pressure on the transesterification reactions was installed. A recirculating cyclonic separator was installed to investigate the potential of increased mixing during transesterification reactions and its effect on product quality. A drying system was added to remove the water content. A diesel-powered generator was installed, which gave the capability of producing electrical power from biodiesel fuels produced onsite and to characterize the effect of biodiesel on the emissions from this generator [17].

Addition of these new facilities allowed several new experimental opportunities. The effects of transesterification of soybean oil over a range of temperatures on reaction rates and total glycerol levels were explored. The addition of the cyclonic separator allowed a preliminary exploration into enhanced mixing action and real time glycerin removal during the transesterification process [17].

The biodiesel pilot plant evolved slowly from producing biodiesel at a laboratory scale with fats and oils to pilot scale production in batches. Efforts were made to make this a continuous process starting from preparing feedstock to final production of biodiesel. The work represented by this thesis was undertaken in order to get one step closer to the goal of making a continuous process.

Crude soybean oil contains impurities that can be classified into two categories: oil insoluble and oil soluble. The oil insoluble impurities consist of seed fragments and meal fines, free water and long chain hydrocarbons or waxes. Most of these materials can be removed by filtration. The oil-soluble materials include free fatty acids, phosphatides, gummy or mucilaginous substances and some other compounds [9]. Phosphatides in soybean oil are most commonly referred to as '*gums*'. Currently, at BECON the soybean oil produced from the soybean processing plant was stored in a tank and the gums (mainly

phosphatides) were removed by gravity separation by settling in the tank and through filtering. Gravity separation takes a long time to remove the gums and it is not very efficient in removing gums because some can not be removed. Therefore, a process capable of separating gums in real time to match with the speed of soybean oil production was needed at the pilot plant. Centrifugal separation of gums is fast and is widely used in the food processing industry. Therefore, centrifugal degumming was chosen and a degumming system was put in place as a tool to demonstrate its use for the biodiesel industry. This thesis will describe the system and present performance results.

2. BIODIESEL BACKGROUND

2.1 Biodiesel

Biodiesel is the name for a variety of ester-based oxygenated fuels made from soybean oil, other vegetable oils, or animal fats. Biodiesel is accepted as a renewable fuel alternative to petroleum-based diesel. Dr. Rudolf Diesel demonstrated in 1900 the concept of using vegetable oil as an engine fuel when he started an engine at the World Exhibition in Paris using peanut oil as fuel [11]. Biodiesel is produced from renewable oilseed crops, such as soybeans, canola etc. that are grown and harvested in what experts call a closed loop carbon cycle. As they grow, these oilseed crops take up carbon dioxide and then release it back into the air when the biodiesel is burned. In a joint study, the U.S. Departments of Energy and Agriculture found that biodiesel reduces CO_2 greenhouse gas by 78% over its entire life cycle compared to petroleum diesel, and has a positive energy balance of 3.2 to 1 (i.e., 3.2 units of energy are produced for every one unit of energy needed for biodiesel production, while diesel fuel's energy balance is 0.83 to 1 [10, 13].

2.2 Biodiesel's Attributes

Across the globe, environmental concerns and energy security issues have prompted legislative action spurring the demand for alternative fuels to displace petroleum-based fuels. The biggest roadblock to the widespread use of most alternative fuels are the need for engine modifications, suffering a loss in equipment performance and requiring capital investment in new fuel supply infrastructure. Biodiesel has many positive attributes associated with its use as an alternative fuel, but by far the most notable asset highlighted by fleet managers is its similar operating performance compared to conventional petroleum diesel fuel and the absence of any changes required to the engines, facilities or maintenance. Because it has similar properties to petroleum diesel fuel, biodiesel can be

used alone (known as B100) or mixed in any proportion with petroleum diesel fuel. Biodiesel has many advantages as a vehicle fuel. For example, producing biodiesel from soybeans or other domestic crops reduces the United States' dependence on foreign petroleum, increases agricultural revenue, and creates jobs. Many federal and state fleet vehicles are already using biodiesel blends in their existing diesel engines. In addition to its contributions to reduced global warming, the use of biodiesel in a conventional engine, either as a pure fuel or blended with diesel fuel, results in a reduction of unburned hydrocarbons, carbon monoxide, and particulate matter. Emissions of nitrogen oxides, which lead to the formation of ozone, are either slightly reduced or slightly increased depending on the duty cycle of the engine and its design. When burned in a diesel engine, biodiesel replaces the exhaust odor of petroleum diesel with the pleasant smell of popcorn or French fries [11].

2.3 Biodiesel Performance

Biodiesel has slightly less energy content and a higher cetane number than the average diesel fuel used in the U.S. However, in-field demonstrations of biodiesel have shown similar fuel consumption, horsepower, torque and haulage rates as conventional diesel [11]. When biodiesel is blended with conventional diesel in proportions of 20% or less, any differences in engine performance are nearly imperceptible. The standard mixture for biodiesel fuel in public transportation fleets and other programs is 20% biodiesel, and 80% conventional diesel fuel. This standard mixture is known as B20.

Biodiesel provides significant lubricity improvement over petroleum diesel fuel. Even the blending of as little as 1 percent biodiesel can provide up to a 30 percent increase in a diesel fuel’s overall lubricity. Currently, some companies are marketing a premium diesel fuel grade containing proprietary additive packages that feature biodiesel as a lubricity component [11].

Compatibility of biodiesel with engine components can be a concern to nonroad operators. Biodiesel will soften and degrade certain types of elastomers and natural rubber compounds over time. The use of high biodiesel percent blends can attack fuel hoses and fuel pump seals. There has been promising research carried out by the Agricultural Research Service involving mixing additives with biodiesel that could overcome this problem [11]. However, due to similar materials issues discovered with low sulfur diesel fuel, the experience has caused many Original Equipment Manufacturers to change over to materials suitable for use with biodiesel. While the problem solving technology is available, new biodiesel users are advised to contact their equipment manufacturer for specific information [11].

Cold weather can cloud and even gel any diesel fuel, and biodiesel and its blends are no exception. Users of a 20 percent biodiesel blend can experience a degradation of the cold flow properties (e.g. cold filter plugging, cloud point) where gelling of the B20 will occur approximately 3 to 5° F sooner than with conventional No. 2 Diesel [11]. All the same precautions employed for conventional diesel are necessary when fueling with biodiesel.

Another major concern with biodiesel fuel is its shelf life, frequently referred to as its stability. Whereas, it is true that all fuels, including No. 2 diesel have storage limits, but the high level of unsaturated carbon bonds in biodiesel makes it much more susceptible to oxidation break down into gummy residues and varnish that can lead to filter plugging or injector deposits [11]. The storage limit for biodiesel blends is similarly affected and varies in proportion to the blend percent and the ambient temperature at which these fuels are stored. Biodiesel consumers are recommended to talk with their fuel distributor to determine the recommended storage limits for your particular fuel and set of circumstances. Various antioxidation additives are under development for biodiesel that can take some of the guesswork out of the stability concerns [10].

2.4 Biodiesel Economics

Using pure biodiesel B100 is very expensive at this time. Also, even if the total soybean oil crop produced in the US were converted to biodiesel including the biodiesel produced by waste restaurant oil and animal fats, it is not sufficient to be considered as a total replacement for diesel in most diesel applications. The most common blend is a mixture of 20% biodiesel with 80% petroleum diesel, or “B20,” while there are a host of other blends as well such as B10, B5 and others. The cost of biodiesel blends depends on the supplier and state you are in but the cost can vary from 20 to 40 cents more per gallon than No. 2 Diesel [11]. The benefits pertaining to reductions in emissions with biodiesel blends will be diminished in direct proportion to the ratio of biodiesel to conventional diesel in the blended fuel. In November of 1998, the federal Energy Policy Act (EPACT) was amended to allow biodiesel to have greater access to the alternative fuels market, and it has become one of the fastest growing alternative fuels in the country [10]. Moreover, it was recognized that biodiesel could be included as a low level blending component in ultralow sulfur diesel fuel as a means to improve its lubricity while providing environmental, economic, and energy security benefits at the same time. If just 1 percent biodiesel were blended with the on-highway diesel fuel pool, over 300 million gallons of biodiesel production would be required. To produce 300 million gallons of biodiesel fuel would take approximately 194 million bushels of soybeans, (in the US total production of soybean was a little over 3 billion bushels in 2006 [12]). This figure is based on the assumption that 7.5 pounds of soybean oil is required to produce one gallon of biodiesel, and that there are 11.3 pounds of soybean oil in one bushel of soybeans [11].

2.5 Biodiesel Formula and Feedstock

Biodiesel is derived from vegetable oils. The major components of vegetable oils are triacylglycerols (commonly called triglycerides). The tryacylglycerol shown in figure 1 is an esters of glycerol with long chain acids called fatty acids designated as R_1, R_2 and R_3. The composition of vegetable oils varies with the plant source. Often the terms fatty acid profile or fatty acid composition are used to describe the specific nature of the fatty acids occurring in vegetable oils. The chemical and physical properties of vegetable oils and the esters derived from vegetable oils vary with fatty acid profile.

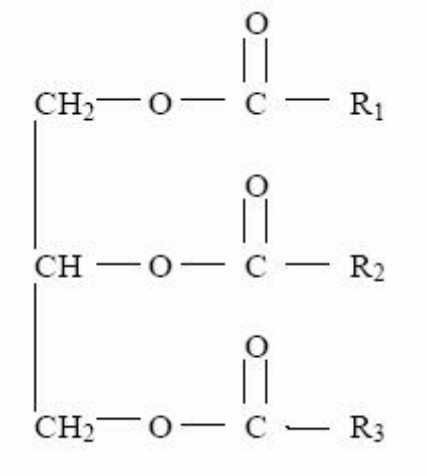


Figure 1: Triglycerol molecule structure

The second major feedstock for biodiesel production is alcohol, (methanol is widely used) which reacts with the triglycerol molecule in the presence of catalyst (acid or base) to form alkyl esters. In a variation of the formation of esters from acids and alcohols, an ester can react with another alcohol; in that case the new alcohol derived from the original ester is formed. Thus, an ethyl ester can react with methanol to form a methyl ester and ethanol. The process is called transesterification. Transesterification is extremely important for biodiesel production. Biodiesel as it is defined today is obtained by transesterifying the

triacylglycerols with methanol. Methanol is the preferred alcohol for obtaining biodiesel because it is the cheapest and most available alcohol. However, for this reaction to occur in a reasonable time, catalyst must be added to the mixture of vegetable oil and methanol. The transesterification reaction is shown in figure 2 below:

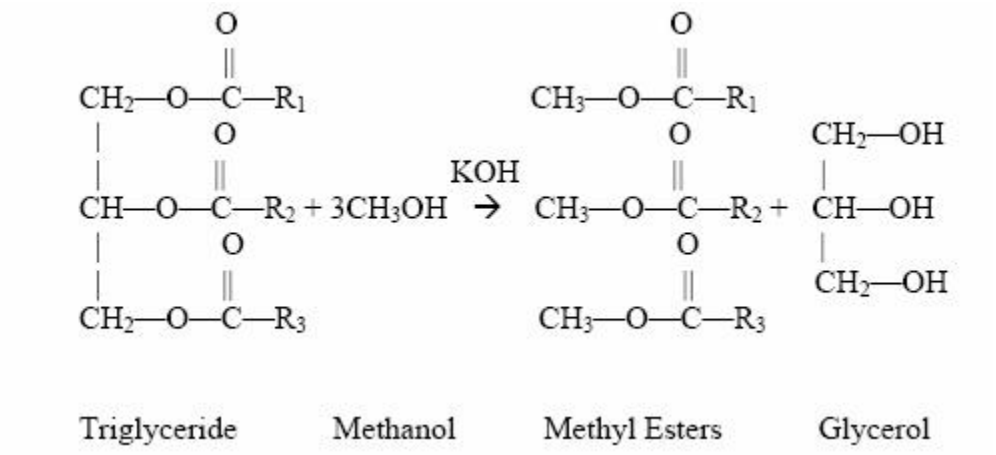


Figure 2: Transesterification of a triglycerol molecule with methanol and potassium hydroxide to produce methyl esters and glycerol

For transesterification to occur, usually 6 moles of alcohol are used for every mole of triacylglycerol, which is two times more than the equation indicates. The reason is that the reaction needs to proceed in the direction of the arrow, i.e. to the right. To force the equilibrium in the direction of the products (as is almost always desired) one or more parameters of the reaction have to be changed such as the molar ratio. Changing the reaction temperature, pressure and use of a catalyst will affect the rate of reaction.

Mono and diacylglycerols are formed as intermediates during the transesterification reaction. If the chemical reaction of transesterification does not reach completion, these

mono and diacylglycerols will form, which contaminate the final product. Other materials that can contaminate biodiesel are residual methanol, glycerol, and catalyst.

When the transesterification reaction is conducted there are two phases of the reactants clearly seen as the methanol and vegetable oil, which are not soluble with each other. Similarly, at the end of the reaction, there are two layers (phases) one consisting of mainly glycerol and other of the vegetable oil esters. Glycerol and vegetable oil esters do not mix readily.

The catalyst used for carrying out the transesterification is frequently sodium hydroxide (NaOH) or potassium hydroxide (KOH). These are inorganic compounds and are strong bases. Many acids can also be used as catalysts, however, it is found that base-catalyzed reactions have higher reaction rates.

Two other kinds of materials, free fatty acids and soap, are also important to the production of biodiesel. Sometimes, the fatty acids themselves can be found in the vegetable oil or fat from which biodiesel is made. When they are present in that form they are called *free fatty acids* (FFA). As a result, some modifications to the transesterification reaction are necessary. Soap arises from a reaction between the FFA and the catalyst used in base-catalyzed transesterification. The presence of water (moisture) influences the formation of soap, which is why the materials used in the transesterification reaction should be as dry as possible.

In accordance with the names of the fatty acids and their esters the methyl ester of soybean oil is often called *methyl soyate*. The term *soybean methyl ester* is also very common.

Fats and oils are simple lipids that are hydrophobic substances and can be found in animals and plants. Generally, fats are solid at room temperature and oils are liquid at room temperature. Fatty acids are long hydrocarbon chains that have a carboxyl group (COOH) at

the end of the chain. Figure 3 below shows the structure for the carboxylic acid. A fatty acid can be denoted as CXX:Y, where XX represents the number of carbon atoms and Y represents the number of double bonds. A saturated fatty acid such as palmitic acid, C16:0, contains no double bonds and is oxidatively more stable (i.e. less reactive compared to oleic acid, which is unsaturated). Saturated fatty acids tend to occur more naturally in animal fats but can also be found in some vegetable oils such as palm oil.

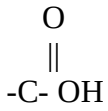


Figure 3: Carboxylic Acid

3. LITERATURE REVIEW

This chapter will review the work that has already been done in the field of biodiesel production and feedstock preparation for producing biodiesel. It will also explain the origin of phospholipids in vegetable oil and the need for their removal.

Soybean oil is the largest source of vegetable oil in the United States of America. Most of this is used in food products but it requires a considerable amount of processing before it can be considered edible. Table 1 below shows the composition of crude soybean oil compared with fully refined oil. This table shows that crude soybean oil typically contains 1.5 to 2.5% phosphatides, 1.6% unsaponifiable matter including 0.15 to 0.21% tocopherol (Vitamin E) and 0.3 to 0.7 % free fatty acids. These compounds contribute color, odor and strong flavors to the oil, and some of the compounds also contribute to poor storage stability. It has been shown that high quality biodiesel can be produced even from crude soybean oil [8]. However use of partially or fully refined oil simplifies the biodiesel production process.

After extrusion from the seed, crude soybean oil contains impurities that can be classified into two categories: oil soluble and oil insoluble. The oil insoluble impurities consist of seed fragments and meal fines, free water, and long chain hydrocarbons or waxes that cause cloudiness when the oil is refrigerated [9]. Most of this material can be removed by filtration. The oil soluble materials include free fatty acids, phosphatides, gummy or mucilaginous substances, color bodies, protein, tocopherols, sterols, hydrocarbons, ketones, and aldehydes. Some of these compounds, particularly the ketones and aldehydes, can cause the oil to have an unpleasant taste and smell. Other compounds are actually desirable, such as the tocopherols which act as antioxidants. The sterols are relatively inert and can remain in the oil. Food grade oil typically contains 0.5 to 1.5% oil compounds that are known collectively as unsaponifiable matter.

Average composition of crude oil and refined soybean oil		
	Crude Oil	**Refined Oil**
Triglycerides, %	95-97	> 99
Phosphatides, %	1.5-2.5	0.003 - 0.045
Unsaponifiable Matter, %	1.6	0.3
Plant sterols, %	0.33	0.13
Tocopherols, %	0.15 - 0.21	0.11 - 0.18
Hydrocarbons, %	0.014	0.01
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

Table 1: Composition of Soybean oil [14, p 14]

Processing of crude soybean oil to food grade oil generally consists of three steps: refining (degumming and caustic refining), bleaching, and deodorization. For biodiesel production, refining is important while bleaching and deodorization may not be done as they are of more importance for the edible oil industry. The refining step is designed to remove the phospholipids and free fatty acids from the crude oil. The main reason to remove the phospholipids is that some of the compounds, particularly the calcium and magnesium salts of phosphatidic and lysophatidic acids are strong emulsifiers. If these compounds are still present during the later alkali neutralization step, they will inhibit the separation of the soaps and lower the yield of neutral oil. Phospholipids, also called phosphatides, will react with water to form insoluble sediments that are not desirable especially when cooking with the oil.

3.1 Phosphorous in Soybean oil and biodiesel

Phosphorous can come from incomplete refining of the vegetable oil and from bone and proteins encountered in the rendering process. In order to achieve acceptable biodiesel quality it is required to remove the phospholipids. Dvorak's work showed that removing gums was important for product yield, not necessarily the quality. Table 2 below shows the profile of phospholipids from lecithin produced from soybean oil [10, 15].

Phospholipid Composition [wt%] of Commerical Lecithins	
Phosphatidylcholine	21.9
Phosphatidylethanolamine	13.6
Phosphatidylserine	0
phosphatidylinositol	12
PG + DPG *	2.3
Phosphatidic Acid	5.8
N-Acyl-phosphatidylethanolamine	2.8
Lyso-phospholipids	2.9
Others	3.6

Table 2: Phospholipid Composition [10, p 52]

* PG+DPG = Phosphatidylglycerol and Diphosphatidylglycerol

The chemical formulae of three major phosphatides in commercial soybean oil are shown in Figure 4.

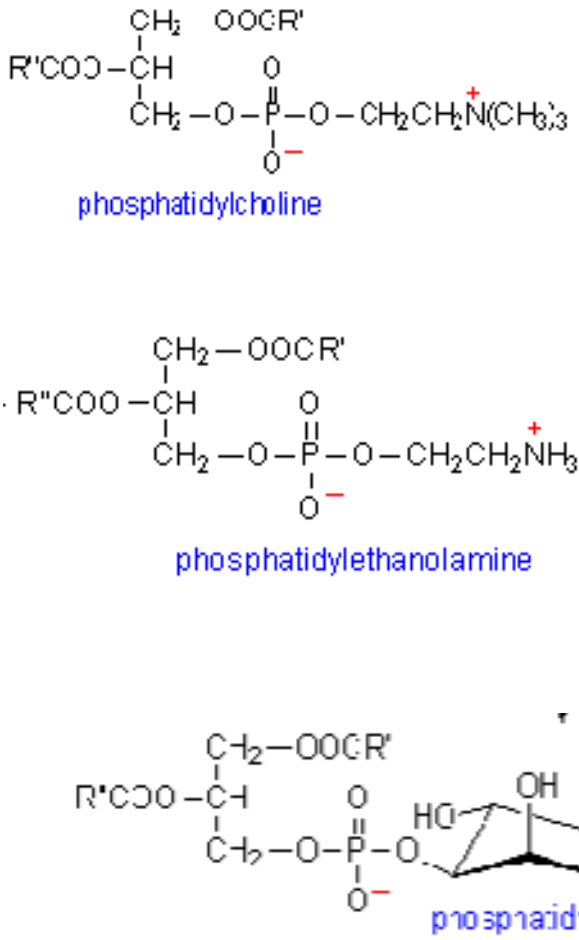


Figure 4 Chemical formula of three phospholipids [19]

The quality of crude oil and the phospholipid content depend primarily on the seed handling, and various parameters affecting the quality are shown in the table below

Abuse characteristics	**Increase in**
Weed seed	d,f
Imature beans	f
Field damaged beans	a,b,c,e
Splits (loading/transport/unloading)	a,b,c
Bean storage (time/temp/humidity)	a,b,c
Conditioning beans for extraction	a,b,d,e
Solvent stripping oil (overheating)	b,d
Crude oil storage (time/temp)	c,d
Oil from stripper (overheating)	b
a - Total gums / phosphatides b - Nonhydratable phosphatides c - Free Fatty Acids d - Oxidation products e - Iron / Metal content f - Pigments	

Table 3: Abuse Characteristics and effect on composition

3.2 Degumming and Lecithin Recovery

Degumming is a process that involves mixing crude soybean oil with 2-3% water, gently agitating for 30 - 60 minutes (being careful to prevent the introduction of air and subsequent oxidation of oil) at a temperature of 70° C [14]. This hydrates the phosphatides and other impurities that can be settled, filtered, or centrifuged out from the degummed oil. This process is commonly done to recover phosphatides to make soybean lecithin and also to remove materials that can settle out during shipment or storage of pure oil. The gum sludge material is processed into lecithin, after drying and bleaching, or added back to wet soybean meal. Lecithin is often desirable in a food due to its wetting, emulsifying, colloidal, antioxidant and physiological properties [14].

Conventional edible oil refining uses acid degumming for removing hydratable and non-hydratable phosphatides followed by alkali refining for removing the free fatty acids [16]. Crude oil is heated to 70°C in a heater and phosphoric acid is added to the mixing tank for converting the non-hydratable phosphatides to water soluble phosphatidic acid. Soft water is added to the mixing tank for the formation of an insoluble precipitate from the hydratable phosphatides.

During the degumming process the following materials are removed :

1. Hydratable non-oil materials, mostly carbohydrates and proteins, which are partially removed.
2. Hydratable non-glyceridic lipids such as phospholipids are partially removed.
3. Chlorophyll (partially removed), especially if phosphoric acid is employed.

3.3 An Overview of Components of Phospholipids, their Structure and Hydration Reaction

As shown in table 2 page 15, most of the phospholipids found in the crude soybean oil, are diacyl surfactants that form bilayer structures in aqueous solutions. The dominant components found in soybean oil are a mixture of phosphatidylcholine (PC), phosphatidylethanolamine (PE), phosphatidic acid (PA), phosphatidylinositol (PI) and a small amount of the monoacyl phospholipids: lysophosphatidylcholine (LPC) and lysophosphatidylethanolamine (LPE). Phospholipids are amphipathic because they usually have two long hydrocarbon chains as well as a short polar group attached to the glycerol moiety; the result is that the polar groups tend to interact with the solution water, whereas the hydrocarbon chains “avoid” the water environment. Depending on the water available, the temperature and the properties of the phospholipid in question, the lipids may thus form

a variety of structures. Additionally, intermolecular interactions in these various structures impose restrictions on the allowed conformations of the polar headgroups and the hydrocarbon chains [20]. During the hydration reaction liquid crystals are formed. It is noted that the hydration reaction by itself always already occurs in a limited extent, in view of the presence of water herein. A hydration reaction is actually only a weak interaction and there can hardly be a question of a real reaction. This implies that they can occur under mild conditions, but this weak interaction with an already strong bond, either covalent or ionic, cannot enter into competition [21].

During the water degumming process, the partially oil-soluble phospholipids in the crude oil are transformed into oil-insoluble lamellar liquid crystals (gum) by absorption of added water [22]. These oil-insoluble liquid crystals have higher density and can aggregate into bigger particles, which can be separated from oil by centrifugation as explained in chapter 3, section 3.3. The separated gums, also called lecithin, typically contain lipids entrained with an equal amount of water. Composition of dried lecithin is approximately 30 wt% neutral soybean oil (TAG) and 44 wt% polar phospholipids. The percent composition of oil-free lecithin is PC 24, PE 20, PI 10, PA 5; the remaining material are lysophospholipids, complex glycolipids, carbohydrates, and other minor components [23,24]. The amphiphilic nature of phospholipids controls the degumming process: Although partially soluble in oil, in the presence of water phospholipids always tend to form monolayers at the water–oil interface to lower the interaction energy associated with hydrophilic and hydrophobic parts of phospholipid molecules. Their acyl chain lengths can range from 16 to 20 carbons, mostly with 18 carbons with one or two unsaturated bonds [22].

3.4 Phospholipid Content Analysis of Crude Soybean Oil

It is advisable to have phospholipids content analysis of crude soybean oil done prior to the experiment. Depending on the composition of phospholipids an experiment should be designed as per the phase diagram explained below to get the optimum amount of water and temperature that result in higher degumming efficiency. It is recommended here to heat the oil and water separately up to the desired temperature and then mix both the streams slowly. Also recommendation is to test the separated gums for getting the exact composition of constituents, which will indicate if the process is optimum or there is still room for improvement.

3.5 Soybean Oil-Phospholipid-Water Phase Diagram [26]

Figure 5 below shows a ternary phase diagram of the PC–water–soybean oil system, the PC component of the phospholipid is chosen here because of their predominance in the phospholipids found in soybean oil. The phase diagram gives a very good picture of the relationships among the components and consequently to optimize important parameters such as the amount of water addition in the degumming process.

The phase diagram provides insight into the degumming process [26]. “During the water degumming process, both chemical composition and phases change. The crude oil, which is marked on the phase diagram, contains a mixture of 2–3 wt% phospholipids. Upon addition of water to the system along the dashed line, crude oil–water changes the compositions of phases and induces phase transitions in the system. When the system contains almost no water or very little water, the phospholipids are dissolved in the oil and the solution is a clear phase, marked L2 in the phase diagram. After water is added to the

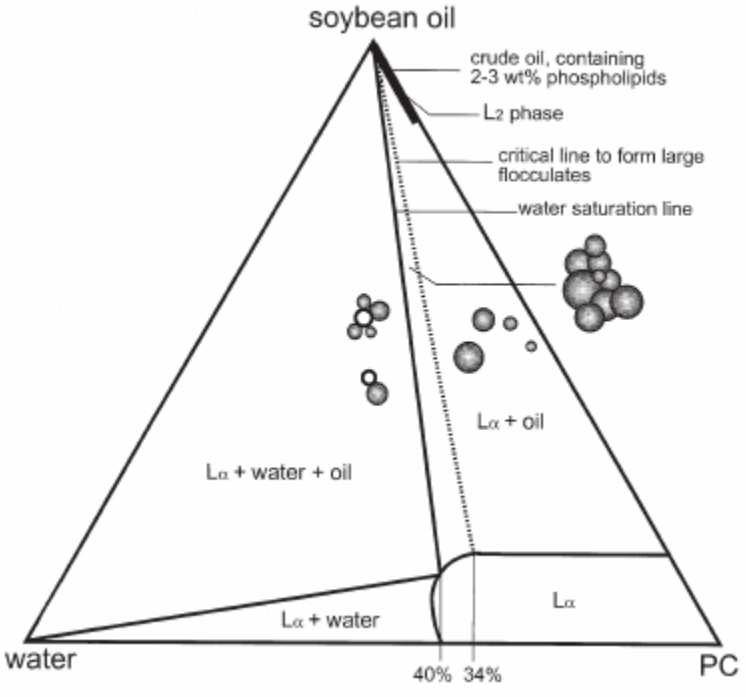


Figure 5: Room temperature ternary phase diagram of soybean oil–water–soybean PC. The dotted line is the critical line above which large flocculates can form. [26]

system, phase separation occurs, and phospholipids aggregate into lamellar liquid crystal by taking up water. As the amount of added water increases, the water uptake of the lamellar liquid crystal increases until a water adsorption saturation point is reached that is right on the phase transition boundary. Beyond the phase boundary, another phase, water, is observed. This water phase complicates the degumming process. The amount of water added to the crude oil is critical for the successful removal of phospholipids from the oil, and it is known in industrial practice that the amount of added water should be equal to or less than the amount of phospholipids in the crude oil. This can be understood from the ternary phase diagram. If the water added is more than the amount of phospholipids, the phases for the degumming process are three, of which the third phase is almost pure water. This would cause the extra problem removing water from the oil. Another problem resulting from excess water addition is the dramatic decrease in particle size."

3.6 Current Industrial Practice for Degumming Soybean Oil

The current industrial practice in degumming has been summarized as follows [25]: “The amount of water used in the industrial practice to hydrate the phospholipids during the degumming step is critical. Usually the amount of water added is 75% of the phospholipid content of the oil. The phospholipid content of soybean oil varies between 1.5 to 3 wt% of the oil, which requires roughly 1 to 2 wt% of water. It is known in the industry if too little water is added, the gums phase will be dark and viscous and the oil phase will be hazy due to the residual phospholipids. If too much water is added three phases will develop making the degumming operation difficult and also results in greater oil loss.” The goal of this paper is to relate current practice to the phase diagram and colloidal behavior of lecithin, oil, and water systems.

3.7 Centrifuge Feed Rate Variation

The hydrated phospholipids separation from soybean oil can also be affected by operating parameters of the centrifugal separator. Impure (gums + oil) oil feed rate to the centrifuge may affect the degumming efficiency. The slower the feed rate more residence time for the oil to be in the separator and consequently better separation. I would like to propose a study matrix to vary the flow from slowest, medium and fastest possible rate for the centrifuge feed and collect the separated gum and oil samples and get them tested for the component content. Repeat the measurements for several times to get a good statistical estimate. Based on the results an optimum condition for operation should be determined. A slower feed rate will result in better separation but obviously it will be more time and energy consuming, a faster feed rate will save the time and energy. Therefore depending on the

gum separation efficiency and maximum permissible limit of phosphorous content an optimum condition needs to be determined.

3.8 Non Hydratable Phospholipid Removal: Acid Degumming

As discussed earlier in section 3.1, table 3 on page 17: under the influence of different circumstances such as harvest, origin, variety, time, temperature, humidity, etc. the quality of crude oil is affected. Chemical alterations in the structure of the phospholipids take place because of the result of the metabolic enzymes still acting in the crude oil. Specifically a so-called salt bridge can develop between two phospholipids, mainly as follows [21]: (only the primary chain is depicted)

Lipid--P--O--Mg--O--P--Lipid

or

Lipid--P--O--Ca--O--P--Lipid.

This reaction causes a splitting within the large group of the phospholipids. Indeed when such a salt bridge occupies the last acid function of the phosphoric acid, the phospholipid can no longer be hydrated. There is then also an important difference made between, on the one hand, hydratable (one or two acid functions possible) and, on the other hand, unhydratable phospholipids (no acid function possible).

After the hydratable Phospholipids are removed the unhydratable side-constituents, mainly the phospholipids which have formed salt bridges, still remain present in the oil. In order to

be able to separate the unhydratable impurities, mainly unhydratable phospholipids, from the neutral oil, two procedures are thus far known which are applicable for industrial application.

The first, procedure consists in a treatment of the oil with a strong alkaline means of reaction, such as sodium hydroxide, in order to neutralize the free fatty acids present in the oil and in order to convert the unhydratable phospholipids into a hydratable form. Through this caustic treatment soapstock develops by the neutralization of the free fatty acids, which by separation on basis of force of gravity or centrifugal force. After the alkaline treatment and the separation of the soapstock, a number of remaining impurities are removed from the oil by adding Fuller's earth to this, after which the Fuller's earth together with the impurities is removed by filtration. The contaminated Fuller's earth forms an environmentally detrimental waste product [21].

The second, procedure consists in a treatment of the oil with phosphoric acid or citric acid. Typically the amount of acid used is between 0.05 and 0.2% of the oil weight [27].

3.9 Principle of Centrifuge separation

Gums are heavier than oil and can be separated by gravity in a tank, but it will take a long period of time. Another means of separating gums from oil is by centrifugal separation, which is quick. Following is the explanation of the working principle of centrifugal separation.

Centrifugal force acting on liquid

In a cylindrical vessel that rotates at an angular speed = ω (rad/s) or N (rpm) and contains a liquid ring of mean radius R (m) see figure 6, the centrifugal acceleration F_c (m/s^2) to which the particles are subjected is:

$$F_c = \omega^2 * R = 0.011N^2 * R$$

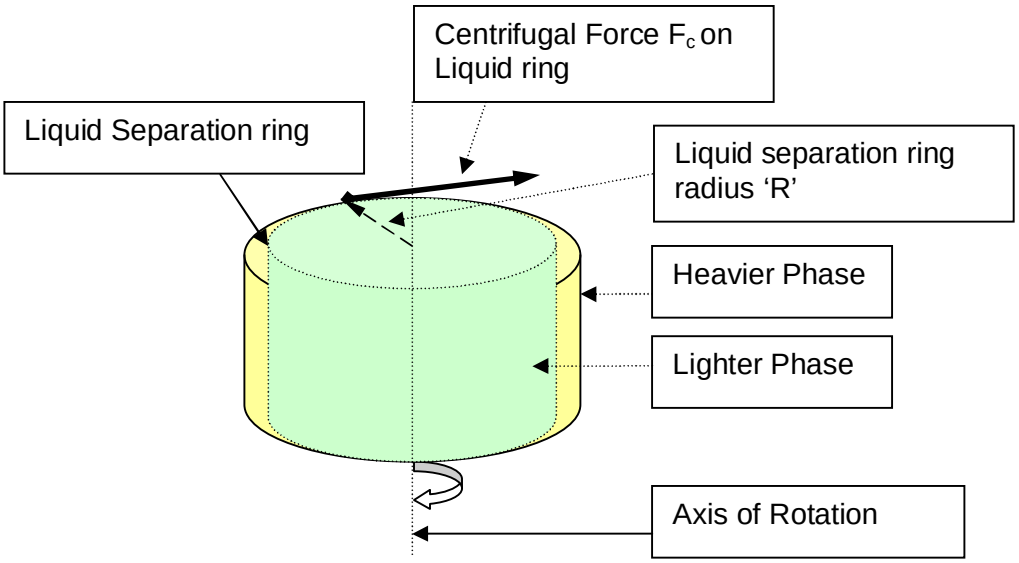


Figure 6: Liquid separation inside centrifuge

The forced exerted on a particle per unit of weight is expressed by:

$$F_c = \frac{0.011N^2 * R * (\rho_s - \rho_L)}{g}$$

$$F_c = G * (\rho_s - \rho_L)$$

Where,

The G-Force, $G = \frac{\omega^2 * R}{g}$

$$G = \frac{0.011N^2 * R}{9.81}$$

$$G = 11.2 * 10^{-4} * N^2 * R$$

ρ_s : Density of particle

ρ_L : Density of the interstitial liquid

Centrifuge

Centrifuges achieve separation by means of the radial acceleration force achieved by a rapid rotation. This can either replace normal gravity in the sedimentation of suspended solids or provide the driving force in the filtration through a filter medium of some kind. The most common application is separation of solid substances from highly concentrated suspensions. Used in this way for the treatment of sewage sludge it enables dewatering with the production of more or less consistent sediment depending on the nature of the sludge to be treated, and the accelerated thickening of low concentration sludge.

Principle

The separation is similar in principle to that achieved in a gravity separation process. The driving force is higher because it is resulting from the rotation of the liquid. In the case of gravity separation, where the driving force is resulting from the difference in density between the solid particles and the liquid, the separation is achieved with a force from 1000 to 20000 times that of gravity.

Types of Centrifuge

Types of centrifuge used for sedimentation include:

- hydrocyclone
- tubular bowl
- chamber bowl
- imperforate basket
- disk stack separator

- decanter

Disk stack separator

The separator used in this application was a disk stack separator, which is explained here in detail. The simplest design is a closed bowl, containing the disk stack, for forcing liquid to flow through small channels and letting solids collect at the outer part of the bowl, from which they have to be removed manually after stopping the rotation. The solids are discharged from the bowl by a number of methods, including the basic use of nozzles, which are open continuously allowing thick slurry to discharge. In a more complicated design referred to as self-cleaning, valved nozzles open automatically when the solid depth in the bowl reaches a certain value, and then close again when most of the solids have been discharged. In the most complicated design the bowl is opened and its shell splits circumferentially for a short period, with the opening also controlled by the solids depth in the bowl.

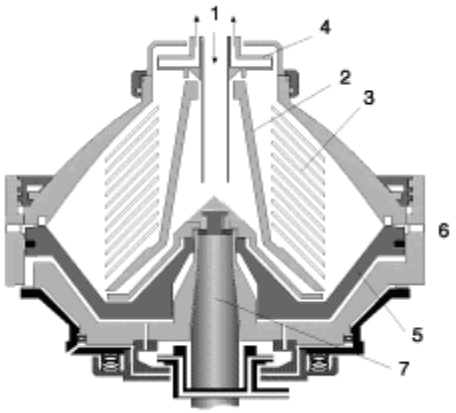


Figure 7: Centrifuge parts

1. Feed
2. Distributor
3. Disk Stack
4. Paring Disc
5. Sliding Bowl Bottom
6. Opening the solids
7. Vertical Spindle

Feed is introduced proximate to the axis of the bowl (1) shown in figure 7, accelerated to speed in a distributor (2) typically by a radial vane assembly, and flows through a stack of closely spaced conical disks (3) in the form of truncated cones. Generally 50 to 150 disks are used. They are spaced 0.4 to 3 mm (0.015 to 0.125 inch) apart to reduce the distance for solid/liquid separation. The angle made by the cone with the horizontal is typically between 40 to 55° to facilitate solid conveyance. Under centrifugal force the solids / phase settle against the underside of the disk surface and move down to the large end of the conical disk and subsequently to the bowl wall. Concurrently the clarified liquid / lighter phase moves up the conical channel. Each disk carries several holes spaced uniformly around the circumference. When the disk stack is assembled, the holes provide a continuous upward passage for the lighter clarified liquid released from each channel. The liquid collects at the top of the disk stack and discharges through overflow ports. To recover the kinetic energy and avoid foaming due to discharging of a high velocity jet against a stationary casing, the rotating liquid is diverted to a stationary impeller (4) in figure 7 from which the kinetic energy of the stream is converted to hydrostatic pressure.

4. BECON FACILITY

The state of Iowa and Iowa State University through the Iowa Energy Center initiated a research project which offers the potential to produce fuels and many chemicals and materials from a renewable resource - farm crops and farm wastes. The Biomass Energy Conversion (BECON) facility is a multi-million dollar investment by the State of Iowa to investigate the production of value-added products from farm crops and wastes and transferring that knowledge to industry. Associated with Iowa State University in Ames, Iowa, BECON uses plant materials and other organic wastes and converts them into fuels and chemicals. BECON's mission is to demonstrate the technology of converting the farm product into a value added fuel or chemical and assist in transferring the technology to industry.

The biodiesel pilot plant at BECON was initially constructed for the purpose of serving as a test bed for the production of biodiesel fuels from high free fatty acid (FFA) feed stocks. Later, the plant was modified for research into biodiesel production methods with soybean oil. Continuous research and improvements resulted in the addition of more equipment for the biodiesel production process. Originally, the soybean oil required for biodiesel production was supplied to the pilot plant by commercial suppliers or purchased locally in smaller batches. This method made the production of large quantities of soybean-based biodiesel impractical. In order to make the process continuous and demonstrate the process to industry, soybean processing equipment was added to the plant. This allowed continuous on-site soybean oil production with a direct supply of oil as a feedstock for the pilot plant. The soybean processing equipment consists of a soybean delivery system, the oil extraction equipment, a fine material screening system, and a crushed meal removal system. In-house mechanical extraction of soybean oil was started from soybeans

procured locally. Crude soybean oil contains many fine particles, or *foots*, from the soybean meal. These are removed by the screening tank (Insta-Pro Model 1500). This 400-gallon tank consists of three separate chambers divided by internal baffles. The oil flows from the first chamber to the next in a cascade fashion. The large volume and cascade flow ensures a lengthy residence time of at least 40 hours while running at full rated capacity. Over this time period the fine material settles to the bottom of each chamber, where it is picked up by a scraper conveyor system. When the screening tank approaches its high level mark, an automatic float switch engages an air-operated diaphragm pump that draws from the top of the last screening tank chamber. This screened oil is passed through a 50-micron bag filter before entering a mixing tank for further processing.

A process for removal of the gums from the soybean oil was required. This thesis explains the design and selection of equipment for the degumming system and the experiments done to find the optimal process parameters for the degumming process.

5. DEGUMMING SYSTEM

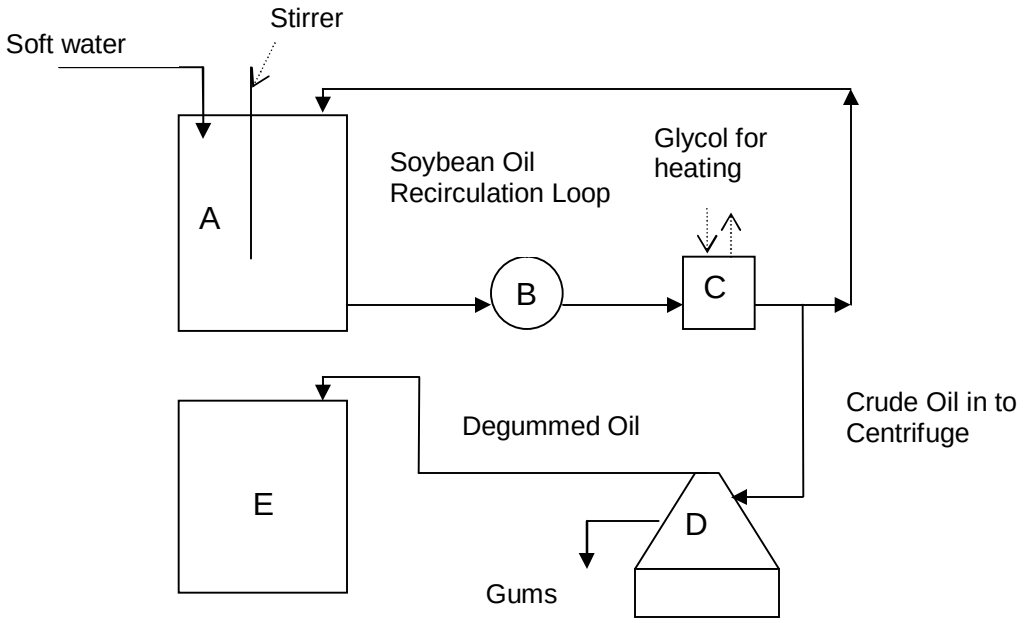
5.1 Degumming System Component and Design

The degumming system consists of the following components

1. Centrifuge separator – Westfalia
2. Mixing tank
3. Gear pump – Tuthill
4. Heat Exchanger (SEC brazed plate Hx)
5. Orifice meter to measure the soft water feed
6. Temperature sensor
7. Pressure gauge
8. Solenoid valve for soft water

A block diagram of the system is shown in figure 8 and a photograph of the degumming system is shown in figure 9. The system consists of a mixing tank ‘B’ made of stainless steel of 24 inches diameter and 36 inches tall. The volume of the tank is 70.5 gallons, which can hold a maximum of 536 pounds of soybean oil. The mixing tank has a stirrer with blades that look similar to a propeller. The stirrer is powered by an electric motor. The mixing tank had a direct connection from a soybean oil storage tank via a filter to catch any solid material in the oil. However, this connection was not used in this experimental study. Soybean oil from oil barrels was fed directly into the mixing tank. The bottom of the mixing tank has a pipe connection with a valve to an oil circulating pump ‘B’. A Tuthill half horsepower gear pump was used to circulate the oil for preheating and then to feed the centrifuge ’A’. The gear pump discharge was connected to a heat exchanger ‘C’ to control process temperature.

The standard operating procedure of the system are given in appendix A.



A – Mixing Tank
B – Oil Circulating Pump
C – Heat Exchanger
D – Centrifuge
E – Degummed Oil Tank

Figure 8: Degumming System block diagram

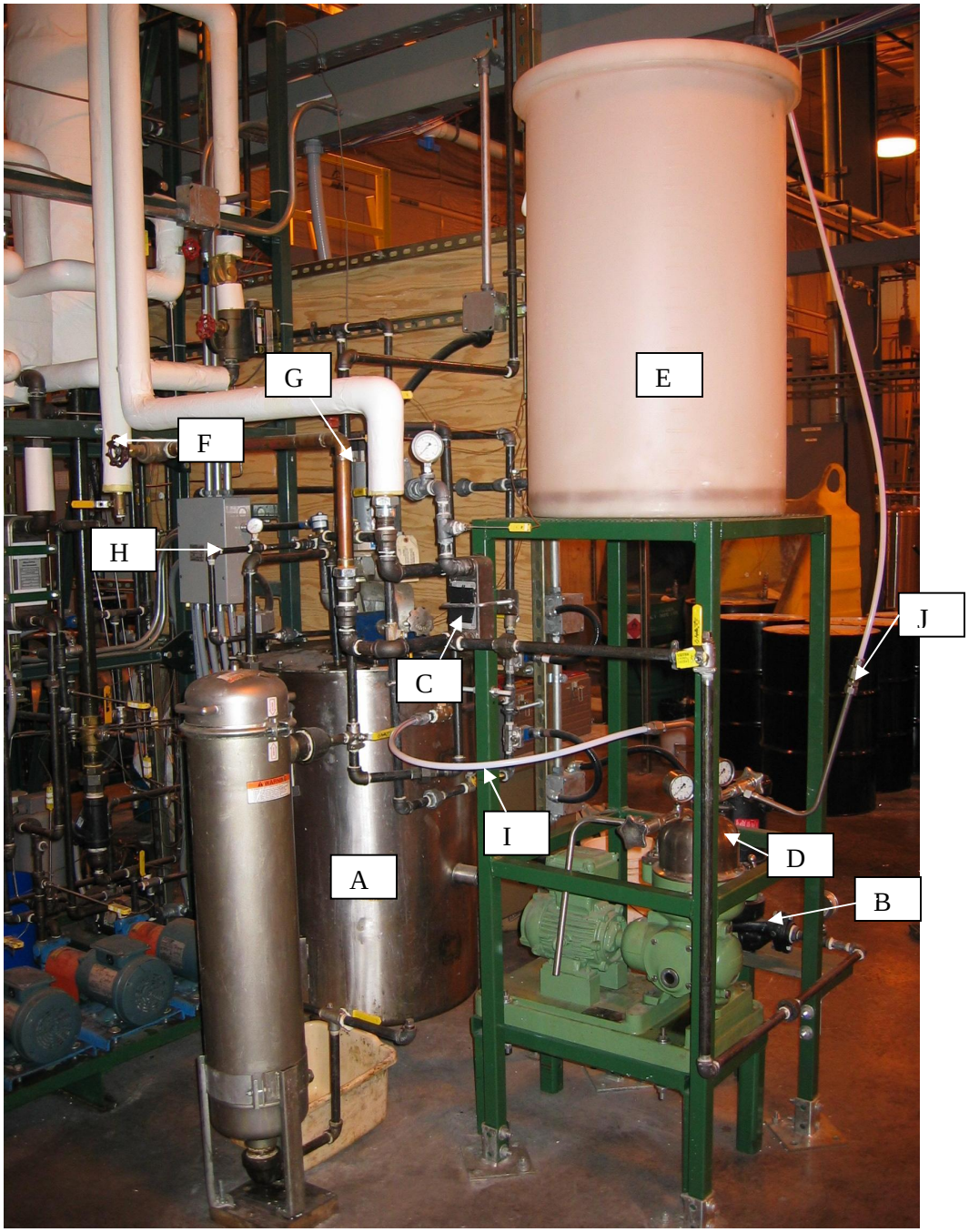


Figure 9: Degumming System

A – Mixing Tank
B – Oil circulating pump
C – Heat Exchanger
D – Centrifuge
E – Degummed oil tank
F – Glycol outlet valve (temperature control)
G – Stirrer
H – Soft water connection
I – Oil feed
J – Degummed oil out

A brazed plate heat exchanger, shown in figure 10, made by SEC was selected because this heat exchanger has a corrugated pattern, which promotes highly turbulent flow characteristics. High turbulence dramatically improves the heat transfer rate and reduces the amount and the possibility of deposit build up.

Hot propylene glycol was used as a heating media as it is readily available in the BECON facility. A thermocouple ‘A’ in figure 10 was inserted on the oil outlet side of the heat exchanger to record the temperature. The temperature of the outlet oil was controlled by manually operating the glycol outlet valve from the heat exchanger. The temperature of the oil was displayed on the control panel. The outlet line from the heat exchanger is connected to the mixing tank with a tee connected via a gate valve to the feed line of the centrifuge.

A gate valve was used to finely control the feed rate to the centrifuge. There are two discharges from the centrifuge. One was the heavy phase, which is the phospholipids in this case. These were collected in a waste bucket. The second discharge is the main separated oil discharge, which was connected to an oil storage tank. A high density polyethylene tank ‘E’ in figure 9 of 50 gallons capacity was used. The centrifuge feed line is connected with a water line, to feed the water required for sealing the centrifuge before the oil feed was started. The mixing tank has a water connection ‘H’ in figure 9 from the water softener. The water line has a solenoid valve, a pressure gauge, and an orifice placed to meter the water into the mixing tank. A small orifice used in agricultural chemical dosing equipment was used for this application. The orifice was calibrated at different pressures to get the exact amount of water flow required for mixing with the soybean oil before degumming. Appendix B provides the orifice calibration data.

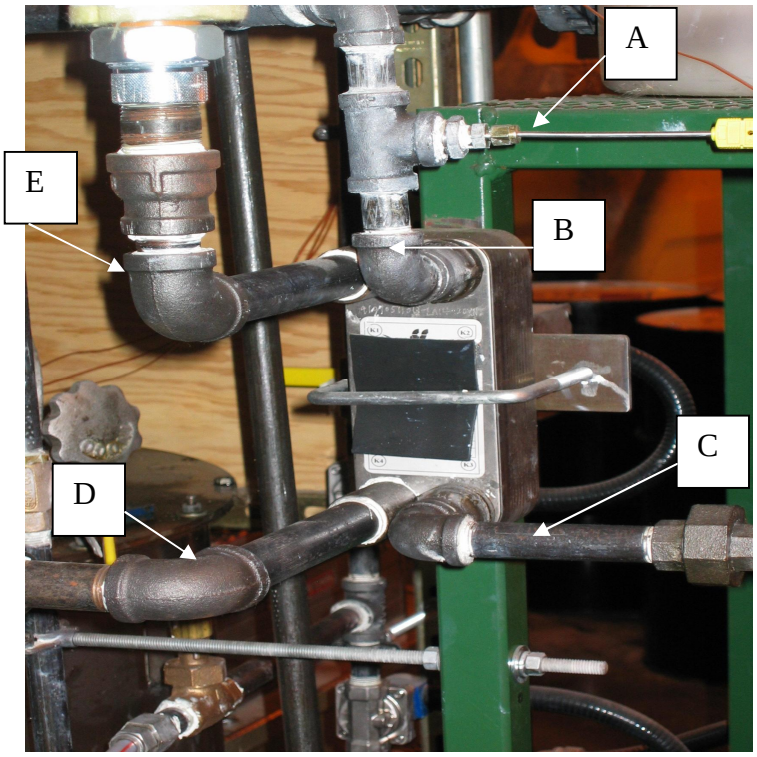


Figure 10: Heat Exchanger

A – Thermocouple
B – Oil outlet
C – Oil inlet
D – Glycol out
E – Glycol in

The centrifuge used for this application is shown in figure 11. It was a Westfalia separator, model # RTA 1-01-525, which had a solid bowl separator and a design capacity of 165 lbs/hr (79 gal/hr) for soybean oil at 9700 RPM. This separator model is used for the separation of two immiscible liquids. If solids are present they are removed simultaneously. The separator is equipped with a solid-wall bowl and a disc stack. Any especially heavy solids contained in the suspension are spun out into the sediment holding space. The light liquid phase flows towards the center of the bowl, the heavy liquid phase towards the periphery. The separated phases are then discharged foam free and under pressure through outlets and by means of stationary impellers inside the centrifuge.

Figure 11: Westfalia Separator

In operation, a quantity of hot water corresponding to a specified percentage of the soybean oil by volume was added to the oil and mixed intensively. The hydratable phosphatides swell immediately due to the particularly intensive mixing of oil and water and extended reaction time is not necessary. This mixture is fed to the separator and the phosphatides, being insoluble and heavier than the oil separate out and are expelled from the heavier phase outlet. Extreme care is required for adjustment of the oil feed rate because the interstitial liquid ring diameter inside moves towards or away from the center of the centrifuge bowl depending on reduction or increase of the oil feed rate. The location of

the interstitial liquid ring diameter governs the gum separation, the higher the feed rate the larger the diameter resulting in lower separation and vice versa.

In a typical degumming system, approximately 1-2% of water is proportioned into the crude oil, pre-heated to 60°-70° C (140° to 160°F) and passed through an in-line or static mixer and then into an agitated holding tank where it is retained for about 30 minutes (20 minutes is the recommended practice).

The oil is then passed through a disc type centrifuge to remove the hydrated gums. Only a portion of the phosphatides are hydratable and removed by this process. Non-hydratable phosphatides (phospholipids) or gums remain in solution in the oil. The amount of water used is just enough to hydrate the phosphatides and any excess is avoided since it may cause hydrolysis of the neutral oil to free fatty acids and increase losses during the later caustic refining step. Non-hydratable phospholipids can be removed by adding phosphoric acid or citric acid, which was not intended to do here because the level of degumming required to achieve for biodiesel production can be achieved by removing the hydratable phospholipids only. Another factor to be considered in a continuous degumming process is the discharge pressure of the oil from the centrifuge in order to keep neutral oil losses to a minimum.

6. RESULTS AND DISCUSSION

6.1 Experiment Design

This study was performed to understand the effect of the quantity of water addition and temperature of the oil on the separation of hydratable phosphatides from the oil. The main variables considered were the soybean oil temperature and the volume percent of water added to soybean oil for degumming.

The three temperatures looked at were:

1. 140 °F (60°C),
2. 160 °F (71°C),
3. 180 °F (82°C)

And the water percentages looked at were:

1. 1%,
2. 2%,
3. 3%,
4. 4%

6.2 Experimental Procedure

The soybean oil used in this experiment was received from the Creston Soybean Processing plant in Creston, IA. It was freshly processed oil and the experiments were performed immediately to avoid any natural separation of phospholipids and degradation of the soybean oil. Oil was received in three 50 gallon drums from which it was transferred to the mixing tank. System start-up procedure is given in appendix A.

For the first step, 1% water was added, the amount of water required was calculated based on the weight of soybean oil present in the mixing tank. This was calculated by measuring the depth of soybean oil in the mixing tank and computing the corresponding

volume and weight of soybean oil from the table given in appendix C. Based on the weight and volume of soybean oil, the amount of water required was calculated and the exact amount of water was introduced in the tank by opening the solenoid valve and recording the time based on the pressure given in the orifice calibration data provided in appendix B. After the water was added, the mixing tank stirrer was turned on allowing the water to mix intensively with the soybean oil. After a couple of minutes, the recirculation pump was started and the ethylene glycol inlet valve to the heat exchanger was opened to heat the oil. The temperature of the oil was monitored by manually opening or closing the glycol valve to get the desired temperature. Once the temperature reached the set point, the oil was recirculated through the heat exchanger back to the mixing tank to maintain the system at the set temperature. Once the system had stabilized at the set temperature the centrifuge could be fed with oil.

Before opening the oil feed to the centrifuge it is important to feed the centrifuge with water to form a seal in the centrifuge bowl. When water is added to the bowl the water fills up the bowl and afterwards when the centrifuge is fed with oil it displaces some of the water but since the liquids have different densities, they tend to experience different centrifugal force and the oil being lighter than the water, stays on the inner side of the bowl and water, being heavier stays on the outer side of the bowl and forms a ring that acts as a seal for the oil and doesn't allow oil to escape through the discharge opening for the heavier phase and overflow.

Another important variable to monitor is the oil feed rate to the centrifuge. The lower the feed rate the better the separation. Low oil feed rate gives more residence time for the oil inside the centrifuge bowl. Because of the longer residence time, the centrifugal force exerted on the oil molecules has a better effect on separation. Too large of a flow rate will eventually fill the complete space in the lighter phase outlet end and eventually get into the

heavier phase outlet end as there is no other space to move inside the bowl. This displaces all of the water, causing the water seal to break and eventually overflow from every possible outlet. Therefore, it is of utmost importance to maintain a steady feed rate to the centrifuge in order to have better separation and avoid any overflows. In the experiment here, the effect of feed rate on the separation of phospholipids was not studied and therefore it was kept constant for all experiments. However, calculations are done to investigate the heat energy requirement for different oil feed rates.

6.3 Results

Oil samples were taken at the discharge end of the centrifuge after each experimental condition of different temperatures and water percentage addition. All the test samples including a sample of soybean oil received from the Creston Soybean processing plant were sent to Eurofin Scientific, Inc, (3507 Delaware Avenue, Des Moines, IA). Test results give phosphorous content in ppm. The percent separation of phosphorous is calculated by the following formula. See appendix D for data.

$$\%Seperation = \left(1 - \left(\frac{ppm(afterseparation)}{ppm(beforeseparation)}\right)\right) * 100$$

6.3.1 Results for 140° F (60° C) oil temperature

The chart below shows the percent separation of phosphorous gums from soybean oil after centrifugal separation at oil temperature set at 140° F (60° C) with different amounts of water addition. The maximum separation in this case was 97.1% and was found with 3% water addition. However the separation was 96.7% with 1% and 2 % water and 96.4% separation with 4% water addition. Very little difference was observed with different amounts of water.

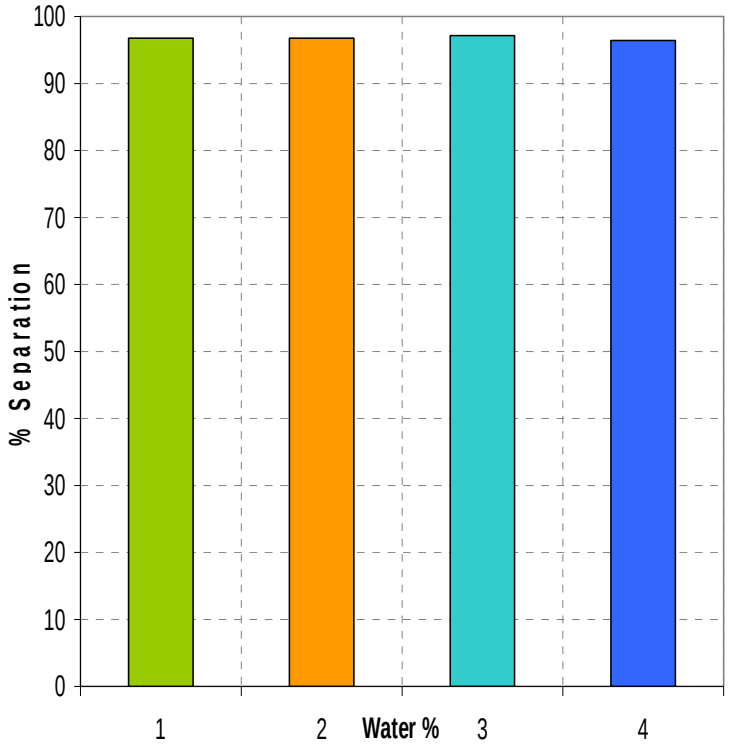


Figure 12: Separation results for 140° F (60° C)

6.3.2 Results for 160° F (71° C) oil temperature

The chart below shows the percent separation of phosphorous gums from soybean oil as obtained after centrifugal separation at oil temperature set at 160° F (71°C) with different amounts of water addition. The maximum separation of 97.1% was found with 1% water addition. However, 96.4% separation with 3% and 96.1 % separation with 2% water and 95.7% separation for 4% water fall behind with little difference.

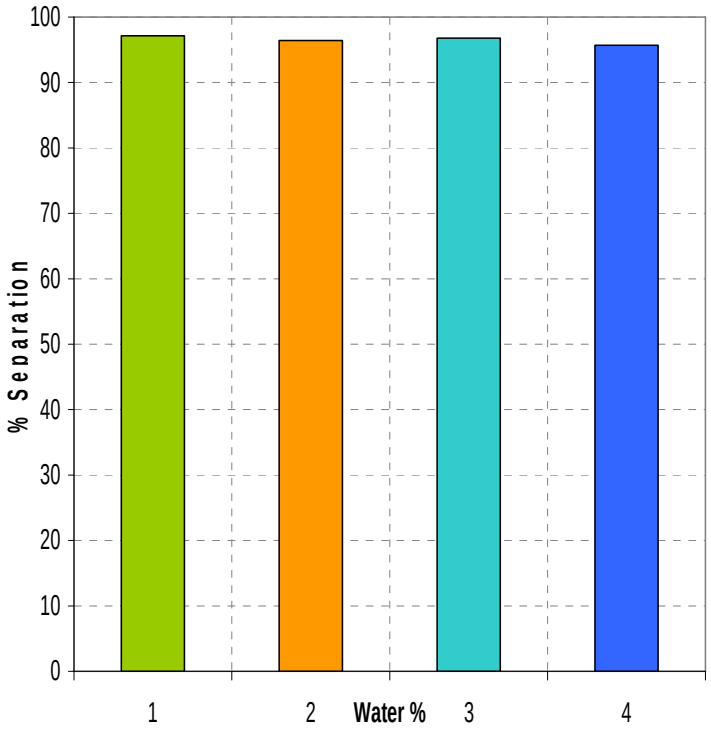


Figure 13: Separation results for 160° F (71° C)

6.3.3 Results for 180° F (82° C) oil temperature

The chart below shows the percent separation of phosphorous gums from soybean oil after centrifugal separation at oil temperature set at 180° F (82° C) with different amounts of water addition. The maximum separation of 98.5% was found with 2% water addition. However, 97.1% separation with 1% and 3 % water falls behind with very little difference as compared to only 92.1% separation for the case of 4% water addition.

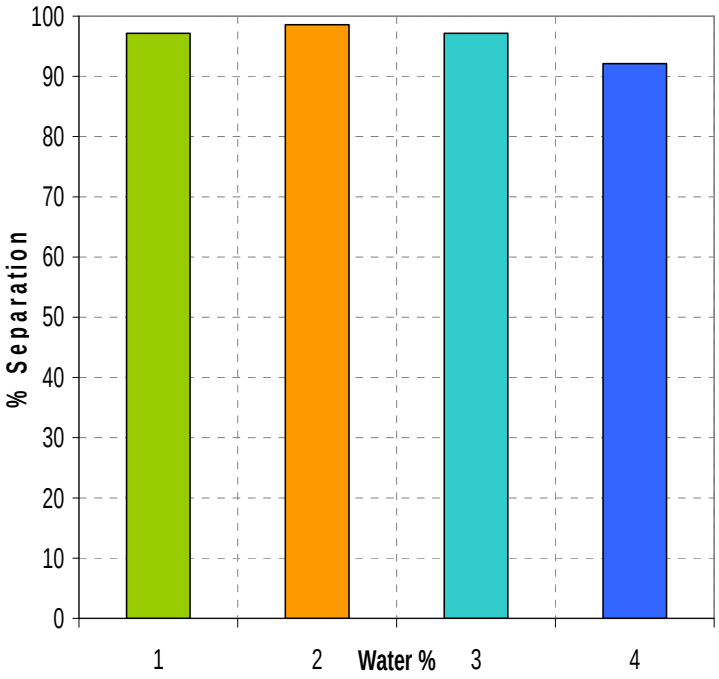


Figure 14: Separation results for 180° F (82° C)

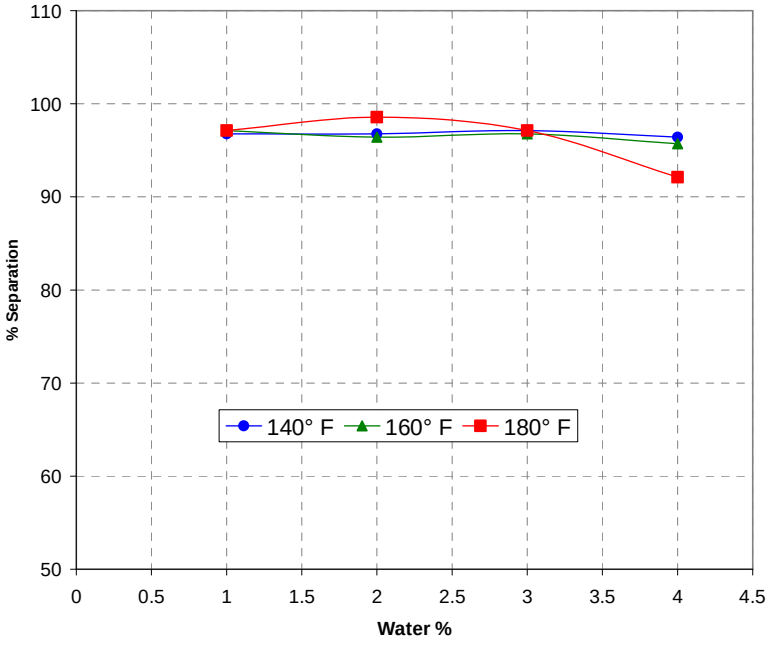


Figure 15: Phosphorous Separation with addition of water at different temperatures

In all the three cases of temperatures the addition of different amount of water from one to four percent did not show any significant difference in the effect of separation (figure 15). For both 140° F (60° C) and 160° F (71° C) the separation efficiency is flat. However, a small trend in decreasing separation efficiency for 180° F (82° C) after two percent of water addition is observed but it is very minimal and difficult to conclude because of the scarcity of data. . Time and resources did not allow for multiple tests so statistics were not possible.

6.3.4 Oil flow rate and energy required for degumming

Degumming of soybean oil consumes a significant amount of energy [13] and therefore it is important to look at the heat energy required at the above mentioned three temperatures. The bowl of the centrifuge in this study has a volume capacity of 0.6 liters and a maximum sediment holding capacity of 0.3 liters [18]. Based on these numbers an assumption is made that the centrifuge can be operated at a minimum oil feed rate of 0.3 liters/min which corresponds to 4.76 gal/hr to a maximum feed rate of 79 gal/hr, which was the maximum capacity of the centrifuge. As mentioned earlier, in order to obtain better separation efficiency it is important to have a slower feed rate and longer residence time for oil to be inside the bowl. Therefore, for varying flow rates starting from the minimum to the maximum a data table is developed (appendix E and F) showing the heat energy required. The same data is shown in figure 16.

Heat Energy is calculated by the following formula

$$Q = m * C_p * (T_2 - T_1)$$

Where,

$Q =$ Heat Energy [BTU/hr]

m = Mass flow rate of soybean oil [lbs/hr]

C_p = Specific heat of Soybean oil [BTU/lb-°F]

T_2 = Target temperature [°F]

T_1 = Room temperature [°F]

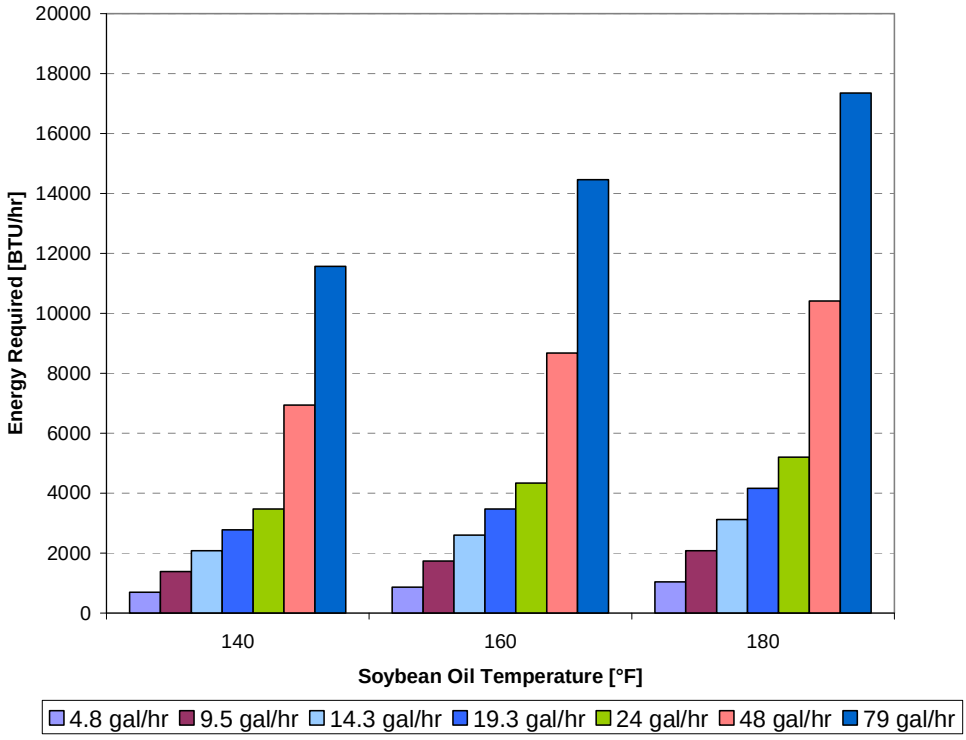


Figure 16: Various feed rates to the centrifuge and heat energy required at the three temperatures under study

From the chart it is obvious that higher the flow rate higher the heat energy required, therefore, it makes no sense to operate the centrifuge at a higher flow rate and also from the separation efficiency perspective as higher flow rate will not give higher efficiency but you

might get more oil through the system per unit of energy. If the three oil temperature in this study do not make significant difference in gums separation then it will be advisable to operate at lower temperature that will save energy cost.

7. CONCLUSION

Soybean oil is the largest source of vegetable oil in the United States and has the potential of being used as a major source of raw material for biodiesel production. As mentioned earlier, crude soybean oil contains about 1.5 to 2.5% of phosphatides, and some of the compounds, particularly the calcium and magnesium salts of phosphatidic and isophatidic acids, are strong emulsifiers. If these compounds are still present during the biodiesel production step of alkali neutralization, they will inhibit the separation of the soaps and lower the yield of biodiesel. Therefore, it is important to remove them as part of the process for feedstock preparation for biodiesel production. It is demonstrated here that use of a centrifuge is a highly efficient and rapid method for degumming of crude soybean oil and it can be applied to biodiesel feedstock preparation.

As is evident from the results, hydratable phospholipids can be easily removed by centrifugal separation. Soybean oil needs to be mixed with a small amount of water (up to 4%) and stirred well and heated to a certain temperature before it can be fed to the centrifuge.

At 140° F (60° C) oil temperature slightly higher gum separation was found with addition of three percent of water. However, no significant differences were seen for other cases. At 160° F (71° C) oil temperature, higher gum separation were found with addition of one percent water and again no significant differences were seen for other cases. However, a higher separation was found at 180° F (82° C) oil temperature and three percent water addition.

The separation results for all the three cases of temperatures and the addition of different amounts of water to the soybean oil from one to four percent did not indicate any one favorable condition that will give best separation. Time and resources did not allow for multiple tests so statistics were not possible. Overall it can be concluded that addition of water and heating of the soybean oil and water mixture helps fast removal of hydratable gums from the crude soybean oil. Lower oil temperature and lower water percent addition would save energy required for heating and drying. A minimum allowable phospholipid level in soybean oil needs to be determined that will give maximum biodiesel yield. Based on that the optimum temperature, feed rate and water requirement that would save the energy cost for the degumming of oil should be determined.

Further work can be done to study the effect of the oil feed rate on phospholipid separation. A slower feed rate will allow more residence time of the oil inside the centrifuge resulting in better separation and higher efficiency of separation. A higher flow rate will lower the residence time thus lower separation efficiency because eventually the oil will fill the complete space in the lighter phase outlet end and eventually get into the heavier phase outlet end as there is no other space to move inside the bowl. Another variable to study is the effect of acid degumming to remove the non-hydratable gums.

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10. APPENDICES

Appendix A

Degumming System Start-up Procedure

To start-up the system

1. Check the gear oil level in the centrifuge. If it is less than the lower mark level add some gear oil kept in the centrifuge box.
2. Check that the centrifuge discharge valves for the separated oil outlet and for the heavier phase are open.
3. Turn on the centrifuge power supply first as the centrifuge takes a long time (approximately 5 minutes) to come up to speed (9700 RPM).
4. Fill the mixing tank with soybean oil and measure the height.
5. Open the soft water valve on top of the water softener to the degumming system.
6. Add the appropriate amount of water to the mixing tank by operating the solenoid valve for the required amount of time as per the experimental plan and calculations given in the orifice calibration table.
7. Start the mixing tank stirrer.
8. Check that the system valves 1, 2 and 3 are open for recirculation. If not then open the valves.
9. Start the gear pump.
10. Make sure the oil is flowing continuously from the mixing tank to the pump and heat exchanger and back in to the mixing tank.
11. Once certain that the oil flow is continuous, open the ethylene glycol outlet valve from the heat exchanger fully and crack open the glycol inlet valve to the heat exchanger.

12. Monitor the temperature of the oil to get the set temperature as per the experimental plan. The temperature of the oil inlet to the gear pump and the outlet of the heat exchanger can be read on the display panel mounted on the control panel.
13. Once the desired temperature is reached, keep the oil recirculating for another five minutes to ensure that the oil is at a steady state temperature.
14. By this time the centrifuge should be at its full RPM. You can confirm this by the noise level. During start-up the noise is high as it is trying to overcome the inertia to rotate heavy steel parts. Once the rotor approaches full RPM the noise level goes down.
15. Once ensured that the centrifuge is ready, open feed valve number six one quarter turn and then open valve 5.
16. After a few seconds you should see water coming out from the heavier phase line, then close the valve 5 and keep valve 6 as it is. You should not see any water coming out from the lighter phase line.
17. Now the centrifuge is ready to take the oil for separation.
18. Open valve 4 slowly while keeping valve 3 open
19. After 20-30 seconds you should see yellow thick heavier phase coming out from the heavy phase line and lighter separated oil from the lighter phase line going up to the storage tank.
20. When the system is up and running just monitor the parameters to ensure system stability. Long continuous use will cause the centrifuge to fill up with heavy sludge and eventually stop separation. Therefore, it is important to clean the bowl after every use.

Appendix B

Orifice meter calibration

Pressure [psi]	Time [s]	Quantity [ml]	Flow [ml/s]	Flow [gal/s]	Flow [gal/min]
10.00	178.31	950.00	5.33	0.0014	0.0844
	172.72	935.00	5.41	0.0014	0.0857
	172.19	926.00	5.38	0.0014	0.0852
10.50	172.69	967.00	5.60	0.0015	0.0887
15.00	135.47	925.00	6.83	0.0018	0.1082
	141.16	955.00	6.77	0.0018	0.1072
	144.72	978.00	6.76	0.0018	0.1070
	141.63	956.00	6.75	0.0018	0.1069
20.00	125.97	982.00	7.80	0.0021	0.1235
	124.00	970.00	7.82	0.0021	0.1239
	125.03	979.00	7.83	0.0021	0.1240
25.00	112.68	989.00	8.78	0.0023	0.1390
	113.40	995.00	8.77	0.0023	0.1390
	111.59	980.00	8.78	0.0023	0.1391

Appendix C

Soybean Oil / Water Weights in Mixing tank

Diameter [inches]	Height [inches]	Volume [in3]	Volume [gallons]	Weight Soybean oil [lbs]	1% Water weight [lbs]	2% Water weight [lbs]	3% Water weight [lbs]	4% Water weight [lbs]
24.00	1.00	452.4	1.96	14.88	0.15	0.30	0.45	0.60
24.00	2.00	904.8	3.92	29.77	0.30	0.60	0.89	1.19
24.00	3.00	1357.2	5.88	44.65	0.45	0.89	1.34	1.79
24.00	4.00	1809.6	7.83	59.54	0.60	1.19	1.79	2.38
24.00	5.00	2261.9	9.79	74.42	0.74	1.49	2.23	2.98
24.00	6.00	2714.3	11.75	89.30	0.89	1.79	2.68	3.57
24.00	7.00	3166.7	13.71	104.19	1.04	2.08	3.13	4.17
24.00	8.00	3619.1	15.67	119.07	1.19	2.38	3.57	4.76
24.00	9.00	4071.5	17.63	133.95	1.34	2.68	4.02	5.36
24.00	10.00	4523.9	19.58	148.84	1.49	2.98	4.47	5.95
24.00	11.00	4976.3	21.54	163.72	1.64	3.27	4.91	6.55
24.00	12.00	5428.7	23.50	178.61	1.79	3.57	5.36	7.14
24.00	13.00	5881.1	25.46	193.49	1.93	3.87	5.80	7.74
24.00	14.00	6333.5	27.42	208.37	2.08	4.17	6.25	8.33
24.00	15.00	6785.8	29.38	223.26	2.23	4.47	6.70	8.93
24.00	16.00	7238.2	31.33	238.14	2.38	4.76	7.14	9.53
24.00	17.00	7690.6	33.29	253.02	2.53	5.06	7.59	10.12
24.00	18.00	8143.0	35.25	267.91	2.68	5.36	8.04	10.72
24.00	19.00	8595.4	37.21	282.79	2.83	5.66	8.48	11.31
24.00	20.00	9047.8	39.17	297.68	2.98	5.95	8.93	11.91
24.00	21.00	9500.2	41.13	312.56	3.13	6.25	9.38	12.50
24.00	22.00	9952.6	43.08	327.44	3.27	6.55	9.82	13.10
24.00	23.00	10405.0	45.04	342.33	3.42	6.85	10.27	13.69
24.00	24.00	10857.3	47.00	357.21	3.57	7.14	10.72	14.29
24.00	25.00	11309.7	48.96	372.09	3.72	7.44	11.16	14.88
24.00	26.00	11762.1	50.92	386.98	3.87	7.74	11.61	15.48
24.00	27.00	12214.5	52.88	401.86	4.02	8.04	12.06	16.07
24.00	28.00	12666.9	54.84	416.75	4.17	8.33	12.50	16.67
24.00	29.00	13119.3	56.79	431.63	4.32	8.63	12.95	17.27
24.00	30.00	13571.7	58.75	446.51	4.47	8.93	13.40	17.86
24.00	31.00	14024.1	60.71	461.40	4.61	9.23	13.84	18.46
24.00	32.00	14476.5	62.67	476.28	4.76	9.53	14.29	19.05
24.00	33.00	14928.8	64.63	491.17	4.91	9.82	14.73	19.65
24.00	34.00	15381.2	66.59	506.05	5.06	10.12	15.18	20.24
24.00	35.00	15833.6	68.54	520.93	5.21	10.42	15.63	20.84
24.00	36.00	16286.0	70.50	535.82	5.36	10.72	16.07	21.43

Appendix D

Phosphorous Separation Data table

Temp [F]	Water added [%]	Phosphorous in soybean oil before separator [ppm]	Phosphorous in soybean oil after separator [ppm]	Percent removed [%]
140	1	1395	45	96.77
140	2	1395	45	96.77
140	3	1395	40	97.13
140	4	1395	50	96.42
160	1	1395	40	97.13
160	2	1395	50	96.42
160	3	1395	45	96.77
160	4	1395	60	95.70
180	1	1395	40	97.13
180	2	1395	20	98.57
180	3	1395	40	97.13
180	4	1395	110	92.11

Appendix E

Centrifuge details and flow rates

Centrifuge		
Max speed [RPM]	9700	
Bowl Volume [Liters]	0.6	
Sediment holding space [Liters]	0.3	
Rated Power [kW]	0.75	

Volume flow [l/min]	Volume flow [l/hr]	Volume flow [gal/hr]
0.3	18	4.76
0.6	36	9.51
0.9	54	14.27
1.2	72	19.02
1.5	90	23.78
1.8	108	28.53
2.1	126	33.29
2.4	144	38.04
2.7	162	42.80
3	180	47.56
3.3	198	52.31
3.6	216	57.07
3.9	234	61.82
4.2	252	66.58
4.5	270	71.33
5	300	79.26

Appendix F

Energy required to heat the soybean oil

Heat Energy required					
Centrifuge Feed [gal/hr]	**mdot [lbs/hr]**	**Sp heat [BTU/lb-°F]**	**T_2 [°F]**	**T_1 [°F]**	**Q [BTU/hr]**
4.8	36.14	0.24	80	60	173.48
	36.14	0.24	100	60	346.97
	36.14	0.24	120	60	520.45
9.5	72.29	0.24	140	60	1387.88
	72.29	0.24	160	60	1734.85
	72.29	0.24	180	60	2081.82
14.3	108.43	0.24	80	60	520.45
	144.57	0.24	100	60	1387.88
	180.71	0.24	120	60	2602.27
19	144.57	0.24	80	60	693.94
	144.57	0.24	100	60	1387.88
	144.57	0.24	120	60	2081.82
24	180.71	0.24	80	60	867.42
	180.71	0.24	100	60	1734.85
	180.71	0.24	120	60	2602.27
48	361.43	0.24	80	60	1734.85
	361.43	0.24	100	60	3469.70
	361.43	0.24	120	60	5204.54
79	602.38	0.24	80	60	2891.41
	602.38	0.24	100	60	5782.83
	602.38	0.24	120	60	8674.24