การใช้ระบบไมโครอีมัลชั่นเพื่อทดแทนการใช้เฮกเซนในการสกัดน้ำมันถั่วเหลือง

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HEXANE REPLACEMENT FOR SOYBEAN EXTRACTION USING MICROEMULSION SYSTEMS

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นรวีร์ คล่องแคล่ว: การใช้ระบบไมโครอีมัลชั่นเพื่อทดแทนการใช้เฮกเซนในการสกัดน้ำมันถั่วเหลือง (HEXANE REPLACEMENT FOR SOYBEAN EXTRACTION USING MICROEMULSION SYSTEMS) อ. ที่ปรึกษา: ดร. จันทรา ทองคำเภา, อ.ที่ปรึกษาร่วม: Prof. Dr. David A. Sabatini 70 หน้า ISBN 974-14-2385-3

โดยทั่วไปเฮกเซนจะใช้เป็นตัวทำละลายในกระบวนการสกัดน้ำมันพืช แต่เนื่องจากเฮกเซนเป็นสารที่มีคุณลักษณะในการระเหยสูงซึ่งคุณลักษณะนี้ส่งผลกระทบต่อสิ่งแวดล้อม งานวิจัยนี้จึงมีวัตถุประสงค์เพื่อประยุกต์กระบวนการไมโครอีมัลชั่นเพื่อนำมาใช้สกัดน้ำมันถั่วเหลืองในสารละลายน้ำเพื่อที่จะหาตัวทำละลายทดแทนกระบวนการสกัดน้ำมันถั่วเหลือง เป้าหมายหลักของการศึกษาครั้งนี้เพื่อนำเสนอวิธีการใช้สารลดแรงตึงผิวที่ละลายน้ำได้เพื่อทดแทนตัวทำละลายอินทรีย์ที่ใช้โดยทั่วไปในกระบวนการสกัดน้ำมันพืชในอุตสาหกรรม การหาตัวทำละลายที่ละลายน้ำทดแทนนี้ถือได้ว่าเป็นเทคโนโลยีสะอาด โดยการศึกษาครั้งนี้มีวัตถุประสงค์ที่จะเพิ่มการแยกตัวของน้ำมันถั่วเหลืองจากเมล็ดถั่วเหลือง ในขณะเดียวกันให้ระบบสามารถลดการละลายของน้ำมันถั่วเหลืองในไมโครอีมัลชั่น ผลการศึกษานี้แสดงให้เห็นว่าสารผสมของสารลดแรงตึงผิวระหว่าง 3% คอมเพอร์แลนด์เคดี (Comperland KD) และ 0.1% อัลโฟเทอรา ห้าพีโอ (Alfoterra 5PO) และ สารผสมของสารลดแรงตึงผิวระหว่าง 4% สแปน 80 (Span 80) และ 0.1% อัลโฟเทอรา ห้าพีโอ (Alfoterra 5PO) สามารถเกิดไมโครอีมัลชั่นกับน้ำมันถั่วเหลือง ดังนั้นระบบทั้งสองนี้จึงถูกเลือกไปใช้ในกระบวนการสกัดน้ำมันถั่วเหลือง อย่างไรก็ตามผลการทดลองพบว่ามีเพียงระบบผสมของสารลดแรงตึงผิว 3% คอมเพอร์แลนด์เคดี และ 0.1% อัลโฟเทอราห้าพีโอ เท่านั้นที่สามารถสกัดน้ำมันถั่วเหลืองและเกิดชั้นน้ำมันอิสระ ได้ ในขณะที่ระบบของสารผสมของสารลดแรงตึงผิวระหว่าง 4% สแปน 80 และ 0.1% อัลโฟเทอราห้าพีโอ ทำให้เกิดอีมัลชั่นซึ่งไม่เป็นที่ต้องการ ทั้งนี้อาจเนื่องมาจากระบบไมโครอีมัลชั่นมีการละลายเป็นคุณสมบัติที่มักเกิดขึ้นร่วมกับการลดแรงตึงผิว ด้วยเหตุนี้จึงได้มีการทดลองใช้ระบบสารลดแรงตึงผิวเดี่ยว โดยเลือกใช้คอมเพอร์แลนด์เคดี ที่ความเข้มข้นต่าง ๆ เพื่อสกัดน้ำมันถั่วเหลืองจากเมล็ดถั่วเหลือง เนื่องจากเชื่อว่าระบบสารลดแรงตึงผิวเดี่ยว อาจช่วยลดแรงตึงผิว โดยไม่เพิ่มการละลาย อย่างไรก็ตามผลการทดลองพบว่าสารผสมระหว่างสารลดแรงตึงผิวสองชนิดมีประสิทธิภาพดีกว่าระบบที่ใช้สารลดแรงตึงผิวชนิดเดียวในกระบวนการสกัดน้ำมันถั่วเหลือง โดยการศึกษาในครั้งนี้สรุปได้ว่าระบบสารลดแรงตึงผิวระหว่าง 3% คอมเพอร์แลนด์เคดี และ 0.1% อัลโฟเทอรา ห้าพีโอ ที่เกลือ 7.5% สามารถสกัดน้ำมันถั่วเหลืองได้มีดีที่สุดที่ 85.77% โดยใช้เมล็ดถั่วเหลืองขนาดระหว่าง 0.212 มิลลิเมตร-0.425 มิลลิเมตร นอกจากนี้ได้มีการทดลองที่เวลาสัมผัสและใช้ปริมาณของเมล็ดถั่วเหลืองที่ต่างกัน พบว่าเวลาที่เหมาะสมในการสกัดคือ 30 นาที ที่ปริมาณถั่วเหลือง 1 กรัมต่อสารละลายของสารลดแรงตึงผิวผสม 10 มิลลิลิตร

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KEY WORD: SURFACTANT SYSTEM/ MICROEMULSIONS/ SOYBEAN OIL/ HEXANE/ SOYBEAN OIL EXTRACTION

NORAVEE KLONGKLAEW: HEXANE REPLACEMENT FOR SOYBEAN EXTRACTION USING MICROEMULSION SYSTEMS. THESIS ADVISOR: CHANTRA TONGCUMPOU, Ph.D. THESIS CO-ADVISOR: PROF. DAVID A. SABATINI, Ph.D. 70 PP. ISBN 974-14-2385-3

Hexane is commonly used as a solvent in vegetable oil extraction process. However, hexane has high volatility which results to the environmental problem from its process. So, the main objective of this study was to apply the microemulsion formation for soybean oil extraction. In order to investigate an alternative for vegetable oil extraction, this study proposed a technique by using surfactant-based aqueous extraction for replacing organic solvent extraction in conventional industrial process. This alternative can be categorized as one of Clean Technologies. The study aimed to introduce a surfactant system that was able to maximize soybean oil mobilization while minimize soybean oil solubilization. For this study, the result showed that the surfactant mixture of 3% Comperland KD and 0.1% Alfoterra 5PO and the surfactant mixture of 4% Span 80 and 0.1% Alfoterra 5PO were able to form microemulsion with soybean oil. These two systems were then selected for further study on soybean oil extraction. The results showed that only the system of mixed of 3% Comperland KD and 0.1% Alfoterra 5PO was able to extract and yield free soybean oil while the system of 4% Span 80 and 0.1% Alfoterra 5PO only yielded an aqueous emulsion phase. This may be because the solubilization that occurred in a microemulsion system. Therefore, a single surfactant; Comperland KD at different concentrations were carried out for soybean seeds extraction in comparison since it was expected that the single surfactant may reduce interfacial tension but not to enhance solubilization. However, the result of the single surfactant systems showed less efficiency for oil extraction as compared to the mixed surfactants system. Conclusively, from the condition used in this study, the mixed of 3%Comperland KD and 0.1% Alfoterra 5PO with 7.5% NaCl showed the maximum soybean oil extraction (85.77%) from soybean seeds at size 0.212 mm-0.425 mm. In addition, the contact time and soybean load were also varied and it was found that the extraction yielded the optimum efficiency for the extraction were at 30 min and1g load of soybean load per the surfactant solution 10 mL respectively.

Field of study: Environmental management Student's signature.....Noravee K.......

Academic year 2005 Advisor's signature..........................

Co-Advisor's signature..........................

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NOMENCLATURES

IFT = Interfacial Tension (mN/m)

CMC = Critical Micelle Concentration

HLB = Hydrophile-lipophile balance

PIT = Phase Inversion Temperature

O = excess oil phase

W_m = oil in water microemulsion

W = excess water phase

O_m = water in oil microemulsion

M = middle phase microemulsion

CNS = Central nervous systems

CHAPTER I

INTRODUCTION

1.1 Introduction

Soybeans are native to northeastern Asia and were first introduced into the United States in 1765 (Erickson et al., 1980). Today, the world's largest producer of soybeans is the United States with the majority of its cultivation located in the Midwestern and Southern United States (Comptonís, 1995). Since 1970 the United States has produced, on average, more than 1.5 billion bushels of soybeans per year (Comptonís, 1995). It has generally grown to produce in 2-3 crops/year in many parts of the country. The planted area increased from 396,800 rai (6.5 rai = 1 hectare) in 1967 to 2,600,000 rai in 1993/94 with a production of 513,000t, at an average yield of 216 kg/rai (Napompeth, 1994). Two main products are processed from soybean: oil and the dry solid residue known as meal. The oil is used to make many edible products such as salad and cooking oils, shortenings and margarines (Erickson et al., 1980). Soybean has been used in Thailand as a source of human food for many generations and it is well recognized as a very important source of protein and fat. The oil content of soybeans is around 20% on a dry weight basis. Soybeans are the world's largest oilseed crop, with about 13 million tons of oil produced per year (Patterson, 1989). Soybeans are in high demand due to their high protein and oil content (Erickson et al., 1980).

Soybean oil is normally produced by extraction with solvents. Hexane is commonly used as a solvent in the oil extraction process due to it being able to dissolve in many types of vegetable oil and being easy to separate by distillation and stripping. Although all modern process plants are well-constructed, it is estimated that 0.7 kg hexane per ton of seed is released into the environment (http://www.epa.govfedrgstrEPA-AIR2000MayDay-26a12794.html). However, hexane is highly volatile solvent which results in a negative impact on the environment. Studies of other alternatives have thus become of interest in order to replace the use of hexane and apply clean technology. This research seeks to investigate replacing organic solvents with surfactant-based aqueous solvents in industrial processes**.** To achieve the clean technology concept, the aqueous based solvent is introduced as an alternative solvent. However, since oil and water are immiscible, a surfactant must be applied to the system in order to reduce the interfacial tension and thus can extract oil from the seed. Microemulsion is a system proposed for this study since its properties are thermodynamically stable, providing low interfacial tension and high solubilization. However, vegetable oil is one of the oils that are found to have difficulty forming a microemulsion as compared to other hydrocarbon oils due to its bulky structure as well as its high hydrophobicity. Consequently, the study aims to discover a system of surfactant solution that is able to form microemulsion with soybean oil and select a suitable system for a batch study on the extraction of oil from the soybean seed. In addition to investigation for a microemulsion system with soybean oil, the oil extraction is a procedure that requires a low interfacial tension but not high solubilization. This is because the solubilization may cause reduction of oil extraction. As a consequent, this study need investigate a system that provides an optimum condition that yields high percent of oil extraction.

1.2 Objectives

The main objective of this study was to extract oil from soybean seeds. The specific objectives were:

1) To investigate the microemulsion systems and their behavior of systems formed by surfactant solutions and soybean oils study by using salinity scan.
2) To determine the optimum conditions for soybean oil extraction as a solvent replacement.

1.3 Hypothesis

The microemulsion formation of the system consisting of surfactant solution and soybean oil is expected to reduce the interfacial tension between oil, water and soybean seed. Thus the surfactant solution can be an alternative for hexane replacement in the process of oil extraction.

1.4 Scope of the Study

The study was divided into two parts: (1) a phase behavior study of the surfactant solution with soybean oil and their behavior and (2) a batch study for soybean seeds and a selected surfactant system from(1). In the first part, various kinds of surfactant that are able to form microemulsion with soybean oil were preliminary investigated. The transition of microemulsion phase was observed and interfacial tensions between the oil and surfactant solution were measured to explain their phase behavior. Then in the part II, the selected surfactant system was used for the batch study with soybean seeds to determine its efficiency for oil extraction.

CHAPTER II

BACKGROUND AND LITERATURE REVIEWS

2.1 Soybean

Mature soybeans are oval shaped and their sizes are variety-dependent. The seeds consist of three major parts: the seed coat or hull, cotyledon, and germ or hypocotyls. These structural components have approximate compositions as shown in the Table 2.1 and Table 2.2. The value of the oil seed lies in its content of high quality oil. Soybean is high in both oil and protein. For the soybean oil, its composition can be approximated by Erickson et al. (1980) as shown in Table 2.3.

Table 2.1 Chemical Composition (wt%) of Soybean Seed and Its Components on Dried Weight Basis (Gunstone, 2002)

Components	Yield	Protein	Oil	Ash	Carbohydrate
Cotyledon	90.3	42.8	22.8	5	29.4
Hull	7.3	8.8	1	4.3	85.9
Hypocotyls	2.4	40.8	11.4	4.4	43.4
Whole seed	100	40.3	21	4.9	33.9

Table 2.2 Composition of Soybean Seeds (Erickson et al., 1980)

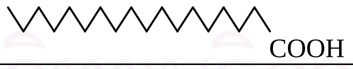
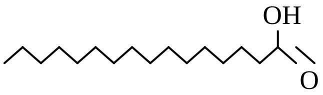
Compositions	Percentage
Protein	40
Lipid	20
Cellulose and Hemicelluloses	17
Sugar	7
Crude Fiber	5
Ash(dry weight basis)	6

Table 2.3 Constituents of Soybeans Oil (Erickson et al., 1980)

Compounds	Value
Triglycerides (%)	93.0-99.2
Crude Oil (%)	up to 4.0
Water-degummed (%)	up to 0.4
Acid-degummed (%)	up to 0.2
Free Fatty Acid (%)	0.3-1.0
Unsaponifiable (%)	0.5-1.6
Tocopherols (ppm)	1700-2200
Chlorophylls (ppm)	Trace
Sulfur (ppm)	Nil

Soybean fatty acids' relative concentrations are 10.7% palmitic, 3.4% stearic, 21.3% oleic, 56.4% linoleic and 8.2% linolenic for the conventional soybean cultivar sowed in Brasilia (Central Region); and 9.3% palmitic, 3.1% stearic, 33.8% oleic, 48.3% linoleic and 5.5% linolenic when sowed in Balsas (Northeast Region), respectively (Gunstone, 2002) (See Table.2.4). The fatty acid formula structure in the soybean oil is as shown in Table 2.4.

Table 2.4 Fatty Acid and Its Chemical Structure in Soybean Oil (Gunstone, 2002)

Fatty acid	Formula Structure	Composition (%)
Stearic		3.40
Oleic		21.30
Linoleic		56.40
Linolenic		8.20
Palmitic		10.70

2.2 n-Hexane

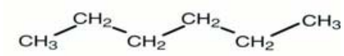
n-Hexane is a chemical made from crude oil. Pure hexane is a colorless liquid with a slightly disagreeable odor. It evaporates very easily into the air and dissolves only slightly in water. Hexane is highly flammable, and its vapors can be explosive. It is used in laboratories, primarily when it is mixed with similar chemicals to produce solvents. Common names for these solvents are commercial hexane, mixed hexanes, petroleum ether, and petroleum naphtha. The major usage of solvents containing n-hexane is to extract vegetable oils from crops such as soybeans, flax, peanuts, and sunflower seed. They are also used as cleaning agents in the textile, furniture, shoemaking, and printing industries, particularly in rotogravure printing. *n*-Hexane is also an ingredient of special glues that are used in the roofing, shoe, and leather industries. *n*-Hexane is used in binding books, working leather, shaping pills and tablets, canning, manufacturing tires, and making baseballs (McParland, 2002).

Health Hazard Information

Acute (short-term) inhalation exposure of humans to high levels of hexane causes mild CNS depression and irritation of the mucous membranes. CNS effects include dizziness, giddiness, slight nausea, and headache in humans. Moreover, hexane causes dermatitis and irritation of the eyes and throat in humans.

Chronic (long-term) inhalation exposure to hexane is associated with polyneuropathy in humans, with numbness in the extremities, muscular weakness, blurred vision, headache, and fatigue observed. Mild inflammatory, erosive, and degenerative lesions in the olfactory and .respiratory epithelium of the nasal cavity have been observed (McParland, 2002). The physical and chemical properties of hexane are as shown in Table 2.5.

Table 2.5 Physical and Chemical Properties of Hexane (McParland, 2002)

Properties	
Chemical formula	C_6H_{14}
Formula structure	
Molecular mass(g/mol)	86.13
Relative vapor density(air =1)	0.66
Flash point (^{o}C)	-21.7
Boiling point (^{o}C)	68.74
Autoignition temperature (^{o}C)	225
Refractive index	1.342
Explosive limits in air (%v/v)	1.1-7.5
Vapor pressure at 25C^{o} (mm Hg)	150
Threshold limit value (TLV) (ppm)	50

2.3 Extraction Theory

Solvent extraction is a diffusion process in which the solvent (hexane) selectivity dissolves miscible oil components during extraction; hexane solubilizes soybean oil from cotyledon lipid bodies in soy flakes rapidly, as soon as it enters the rapid body. The slowest process is solvent diffusion into the flakes and diffusion of the oil hexane miscella out. Nevertheless, this process is faster than the extraction of raw cotyledons or fresh beans, which are almost impenetrable by hexane (Lui, 1999).

Flake thickness is therefore very important in controlling diffusion. Flakes must be thick enough to avoid breaking up during handling. Crumbling of the thin flakes will result in fines that will not allow the solvent to flow through the fine particles. Commercially produced soy flakes are usually 0.210mm - 0.420mm thick. Orthmer and Agarwal (1955) expressed the rate of extraction mathematically, as being related to flake thickness and residual oil:

$$-dC/dt = kF^{-3.97}C^{3.5}$$

The oil concentration in the flakes is C, time is t, flakes thickness is F, and k is a constant. The equation shows that a doubling of F (flake thickness) would decrease –dC/dt (the extraction rate) by a factor of about 16 (Lui, 1999).

The % oil extraction can be calculated as following this formula.

%Oil Extraction = (wt of oil in soybean seed/d $_{\text{soybean oil}}$)*100
wt of oil in soybean seed = (wt% of oil from soybean seed)*wt of soybean seed

2.4 Extraction Process

The production of commercial soybean oil is complicated. The mechanical production of soybean oil with hydraulic presses is not often used because it is expensive and gives lower yields. Soybean oil is normally produced by extraction with solvents. This production consists of the following steps:

2.4.1 Pre-treatment

Soybeans must be carefully cleaned, dried and dehulled prior to oil extraction. Figure 2.3 depicts a soybean dehulling system. The soybean hull needs to be removed because it will absorb oil and result in a lower yield. Initially, the soybeans are fed into cracking rolls to crack the beans, followed by the separation of the hulls with the use of shaker screens and aspiration. The lighter hulls are removed from the heavier beans and kernels, with the use of fan aspiration and screens to strain for heavy objects. Magnets are used to separate any metals. The extraction of the oil is facilitated by conditioning the beans through the application of indirect steam to adjust the moisture content and temperature; cracked beans are exposed to temperatures of approximately 74 - 79 ^{o}C for 30 - 60 minutes prior to using smooth-surface rollers to flatten them into large flakes of uniform thickness. The purpose of the conditioning is the heat denaturation and subsequent coagulation of the proteins present in addition to the coalescence of oil droplets and reduction in the affinity of oil for the solid materials, making it easier to extract (Erickson et al., 1980). The picture of dehulling system for soybeans is show in Figure 2.1.

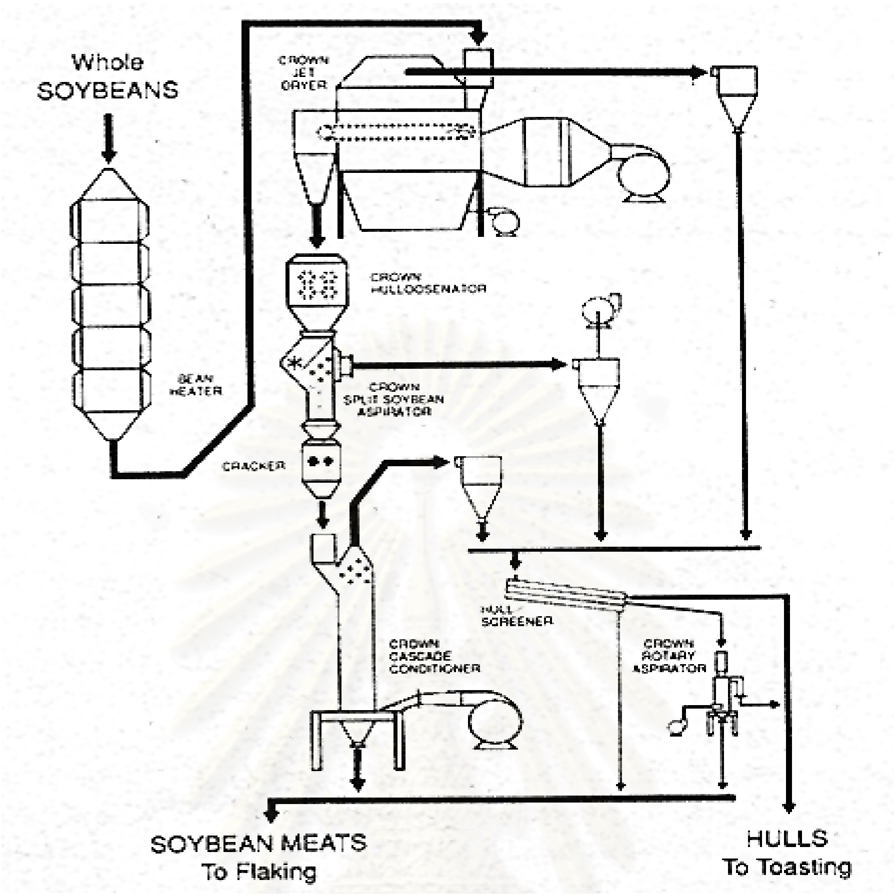


Figure 2.1 Diagram of a Dehulling System for Soybeans

2.4.2 Extraction of Soybean Oil

First the soybeans are cut in flakes which are put in percolation extractors and immerged with a solvent which is normally hexane. Counterflow is used as the extraction system because it gives the highest yield. After removing the hexane, the extracted flake only contains about 1% of soybean oil and is used as livestock meal or to produce food product such as soy protein. The hexane is separated from the soybean oil in an evaporator.

The evaporated hexane is recovered and returned to the extraction process. Solvent recovery is a very crucial step in vegetable oil processing because of economic, and increasingly, environmental and safety reasons. Currently, solvent is recovered by distillation and condensation. The operation is energy intensive and involves volatile solvents that may cause hazardous situations, especially at the solvent's boiling temperature. The unit that removes the residual hexane from the meal produces most of the hexane vapor. Although modern plants are well-constructed, it is estimated that 0.7 kg hexane per ton of seed is released into the environment. The picture of solvent extractor is show in Figure 2.2.

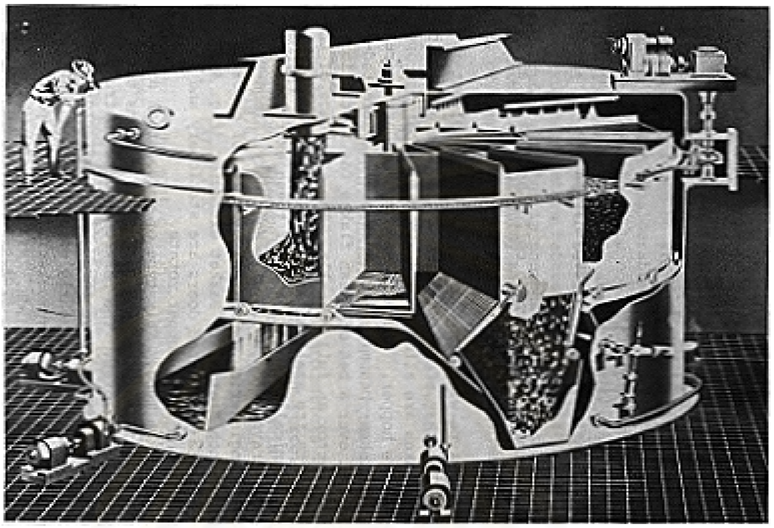


Figure 2.2 Drawing of a Solvent Extractor

2.4.3 Purification of Soybean Oil

The crude soybean oil still contains many oil-insoluble and oil-soluble impurities that need to be removed. The oil-insoluble materials are removed with filtration and the soluble materials are removed using different processes including degumming (the removal of phosphatides), alkali refining (washing with alkaline solution to remove free fatty acids, colorants, insoluble matter and gums) and bleaching with activated earth or activated carbon to remove color and other impurities.

In 2003, Hsu and Nacu studied the microemulsion formation of soybean oil with nonionic Tween series surfactants. In the case of Tween 85, the influence of pH and electrolytes on the ζ potential of emulsion drop was examined. High concentrations of Na^+ and K^+ are capable of separating the oil phase from the emulsion. Divalent cations such as Ca^{2+} and Mg^{2+} lead to two points of zero charge at high concentrations, which was not observed in cases with hydrolyzable trivalent species like Al^{3+} and Fe^{3+}.

Dantas et al., (2001) studied new microemulsion systems containing diesel and different percentages of vegetable oils (soy, palm and rice), that can be used as alternative fuels. The main parameters that have an influence on the microemulsion, such as the nature of the surfactant (T) and co-surfactant (C), the C/T ratio and the composition of the oil phase, have been studied. The results show that it was possible to obtain new microemulsion systems with different oil phase compositions (mixtures of diesel and vegetable oils).

Chen et al., (1997) studied the rate of solubilization of triolein by secondary alcohol ethoxylate Tergitol 15-S-7. The rates of solubilization of triolein by secondary alcohol ethoxylate Tergitol 15-S-7 at 30^{o}C and 35^{o}C, the latter temperature being just below the cloud point were several times the corresponding rates for the pure liner alcohol ethoxylate $C_{12}E_6$ with enough n-dodecanol added to give it nearly the same cloud point. In contrast, the rates of solubilization of n-hexadecane by two surfactant solutions were about the same and were several times greater than the triolein solubilization rates. The addition of short chain alcohols to the surfactant solutions increased the rate of solubilization of both triolein and n-hexadecane for both linear and secondary alcohol ethoxylates.

Raman et al., (2003) studied the phase behavior of palm oil olein (PO) and its derivative oils (palm oil methyl esters and medium chain triglycerides) with Imbentin coco 6.9EO, an ethoxylated $C_{12\text{-}14}$ alcohol in water to identify the composition where microemulsions occur. The techniques used were the optical microscope phase penetration scan and small angle X-ray diffraction (SAXS). Mixed surfactant/oil samples were prepared at wt. ratios of 0.1:1, 0.25:1, 0.5:1 and 1:1 for the phase penetration scan. For SAXS analysis, the initial concentration of surfactant in water (W) was fixed at 38% (w/w), which formed a hexagonal mesophase (H_1). Palm oil methyl esters (POME) and medium chain trigrecerides (MCT) were added to this at 0.04:1(or 0.05:1 for MCT), 0.1:1, 0.2:1 (or 0.25:1 for MCT), 0.5:1 and 1:1 ratios of oil to surfactant. Schematic phase diagrams were constructed to document the changes of phase structures in both bulk samples and phase penetration scan techniques.

2.5 Surfactant

Surfactant is one of the most versatile products of the chemical industry, appearing in such diverse products as motor oils, pharmaceutical, paints, cosmetic and detergents. A surfactant, which is a contraction of the term Surface Active Agent, is characterized by two distinct groups which differ greatly in their solubility relationship (Winsor, 1954). The structure of a surfactant molecule consists of a hydrophilic (water-loving) part which is usually an ionic or highly polar group and a hydrophobic (water-hating) part which is usually a long-chain hydrocarbon or non-polar group, or commonly and perhaps more pictorially as the head and the tail respectively. Figure 2.3 illustrates a basic structure of surfactants. One of unique properties of a surfactant is its tendency to adsorb onto the surface or interface of the system, resulting in the decrease of the surface or interfacial tension of the medium in which it is dissolved. The term interface indicates a boundary between any two immiscible phases. The term surface denotes an interface where one phase is gas, usually air.

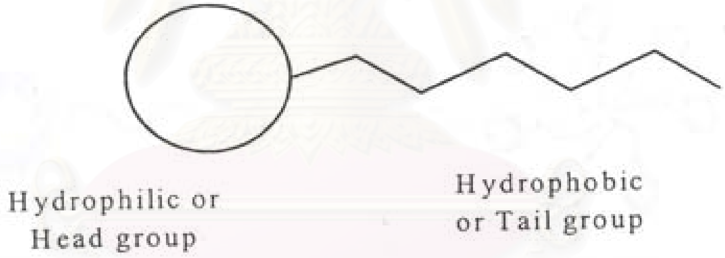


Figure 2.3 Structure of Surfactant Molecule

Depending on the nature of the hydrophilic group, surfactants are classified as anionic, cationic, zwitterionic and nonionic.

1) Anionic surfactants will adsorb onto a positively hydrophilic surface because the surface-active portion of anionic surfactants has a negative charge. Carboxylate, sulfate, sulfonate and phosphate are the polar groups of anionic surfactants. Anionic surfactants are the largest surfactant class. They are favorable because of their low cost of manufacture.
2) Cationic surfactant will be adsorbed onto a negatively hydrophilic surface because the surface-active portion has a positive charge. The polar head

groups found in cationic surfactants are both amine and quaternary ammonia based products. Their main uses are related to in situ surface modification because they adsorb strongly to most surfaces.

3) Zwitterionic surfactants, since they carry both positive and negative changes, can adsorb into both negatively and positively charged surfaces without changing the charge of the surface significantly. The positive charge is almost invariably ammonia, while a carboxylate group is the most common form of the negative charge. Due to their high price, they are the smallest surfactant class. This surfactant type is compatible with all other classes of surfactants. They are well suited for use in shampoos and other personal care products because of their very low eye and skin irritation.
4) Nonionic surfactants adsorb onto the surfaces with either the hydrophilic or the hydrophobic group oriented toward the surface, depending upon the nature of surface. A polyether consisting of oxyethylene units made by the polymerization of ethylene oxide are usually the polar groups of nonionic surfactants. The physicochemical properties of ethoxylated compounds are very temperature sensitive because their solubility increases with temperature. In contrast, ionic surfactants are less water soluble and more hydrophobic, at high temperature (Rosen, 2004).

A single molecule of surfactant is called a monomer and at sufficient concentration in solution, monomers or surfactant molecules will nucleate to form aggregates called micelles. This process illustrated in Figure 2.4, is called micellization and the lowest total surfactant concentration at which micelles are present is known as the critical micelles concentration (CMC). Micelles are highly soluble in water because of their polar exterior, while their non-polar interior provides a hydrophobic sink for organic compounds, thus increasing its organic compound solubility. Contaminant solubility and extraction efficiently is maximized when the surfactant concentration is above the CMC. On the other hand, a minimal effect on enhanced contaminant solubility is produced when adding surfactant concentrations near or only slightly above the CMC. The picture of micelle formation is shown in Figure 2.4.

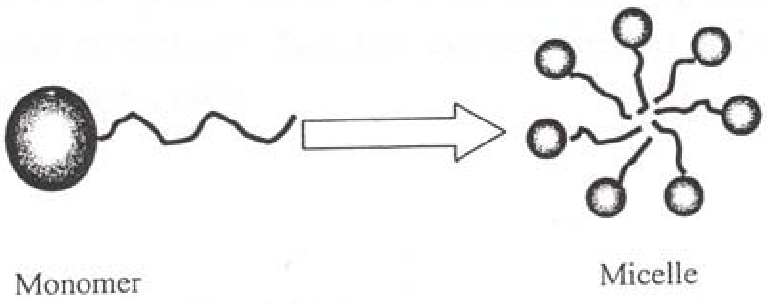


Figure 2.4 The Formation of a Micelle

There are two types of micelles normal and inverse (or reverse) micelles. In an aqueous solution, normal micelles are formed with the hydrophobic part in the interior and the hydrophilic part in the external. In contrast, inverse micelles are formed in a non-polar solvent with the hydrophilic part in the interior and the hydrophobic part in the external. The picture of normal and inverse micelles is shown in Figure 2.5.

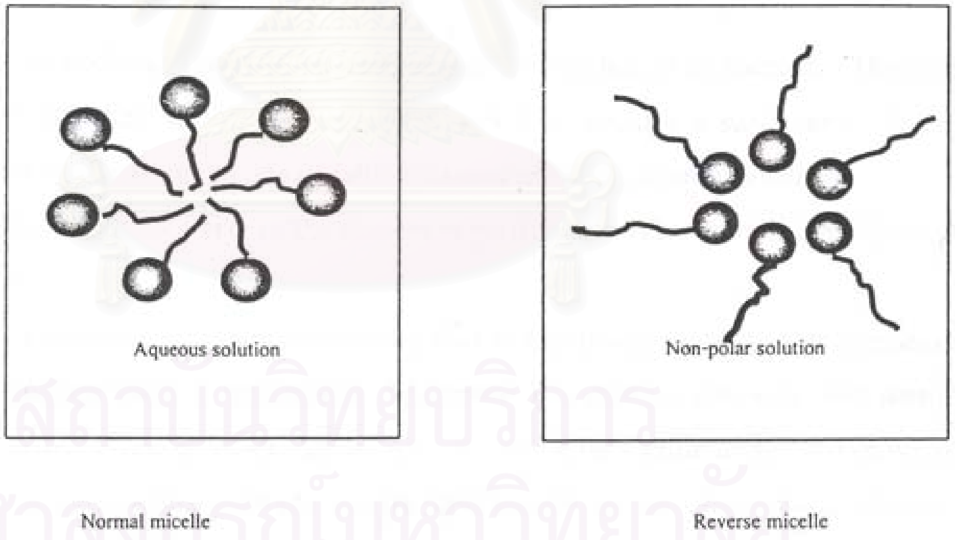


Figure 2.5 Normal Micelles and Inverse Micelles

2.6 Emulsion

Emulsion is a significantly stable suspension of a tiny particle of liquid within a second immiscible liquid. Normally, two immiscible pure liquids such as oil and water can not form an emulsion. The third component must be present in order to promote the suspension of one liquid in another. This third component is called as emulsifying agent and it is usually a surfactant. In fact, the effective emulsifying agents are usually a mixture of two or more substances.

Based upon the size of the dispersed particle, the emulsion is classified into three types.

1) Macroemulsion is an emulsion that is kinetically and generally milky-white in appearance. Moreover, it is an opaque emulsion with particle size greater than 400 nm which may be easily visible under a microscope. It often tends to spontaneously separate into two or more phases without a significant temperature change.
2) Microemulsion is a transparent dispersion with particle size less than 100 nm. It is thermodynamically stable and has been intensively studied during the past decade because of its various applications.
3) Miniemulsion is blue-white, with particle size between 100-400 nm.

2.7 Microemulsion

Microemulsions are transparent dispersions that contain two immiscible liquids with particles of 10-100 nm (0.01-0.1μm) in diameter that generally obtain upon gently of oil and water in the presence of surfactants. A microemulsion is generally prepared with more than one surfactant or with a mixture of a surfactant and cosurfactant (e.g., a polar compound of intermediate chain length)

Microemulsions are thermodynamically stable with homogeneous mixtures of oil, water and surfactant. This system can be divided into three types, which are known as Winsor Type I, II, and III microemulsion. A type I microemulsion corresponds to oil solubilized in aqueous micelles (surfactant aggregates having a hydrophobic interior and hydrophobic exterior). The IFT between oil and water decreases with increasing salinity for ionic surfactant system and increasing temperature for nonionic surfactant system making oil solubilized in micelles A type III microemulsion corresponds to an oil and water bicontinuous phase that is stabilized by a surfactant membrane. The IFT between the middle phase and oil equals to the IFT between the middle phase and water at the lowest IFT or the optimum salinity. A type II microemulsion corresponds to water solubilized in reverse micelles present in the oil phase. The phase changing of microemulsion is related to its hydrophile-lipophile balance (HLB). The HLB is a parameter that shows the partition of a surfactant between its oil and water phases relative to surfactant hydrophilicity. The transition of microemulsion and the decreasing of the HLB for an ionic surfactant is induced by increasing salinity. While for anonionic surfactant, the transition is occurs by raising the temperature of the system. Figure 7 shows an aqueous micelle at lower salt concentration and oil phase inverse micelles at a higher salt concentration for ionic surfactants. The concentration of salt that produce minimum IFT and balanced hydrophilic and lipophilic properties of surfactant at middle phase microemulsion is called optimum salinity. For a nonionic system, the varying temperature can also change the transition of the system. The temperature at the microemulsion inversion is known as the phase inversion temperature (PIT). Microemulsions enhance oil remediation because of their ability to reduce the oil-water interfacial tension to ultra low value (Rosen, 2004).

The most studied phase equilibria of microemulsion is probably known as the Winsor-Type microemulsion (Winsor 1954). There are three types of microemulsion of microemulsion as shown in the Figure 2.6.

1. Winsor type I: Oil in water microemulsion or oil-in-water microemulsion in equilibrium with an excess oil phase.

2. Winsor type II: Water in oil microemulsion in equilibrium with an excess water phase.

3. Winsor type III: The three phase system consist of non-droplet type microemulsion (bicontinuous phase) in equilibriumwith both excess oil and excess water phases. It is generally refer to as middle phase microemulsion.

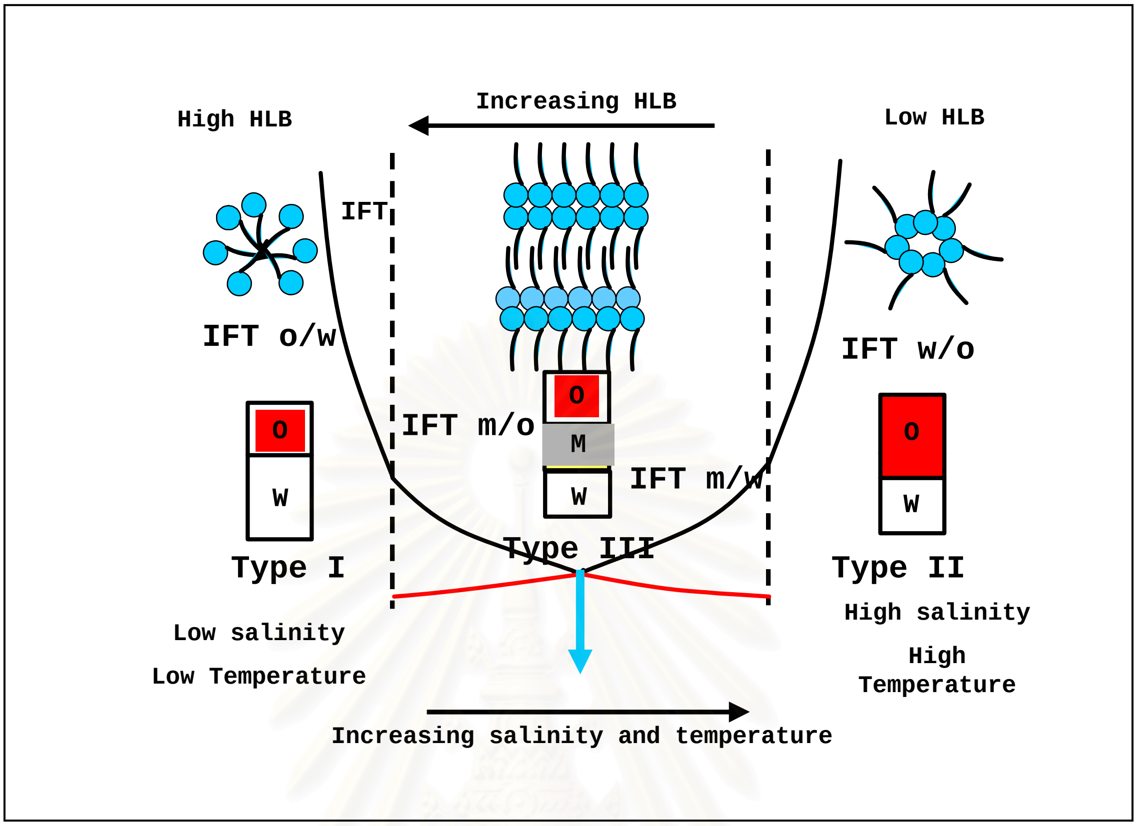
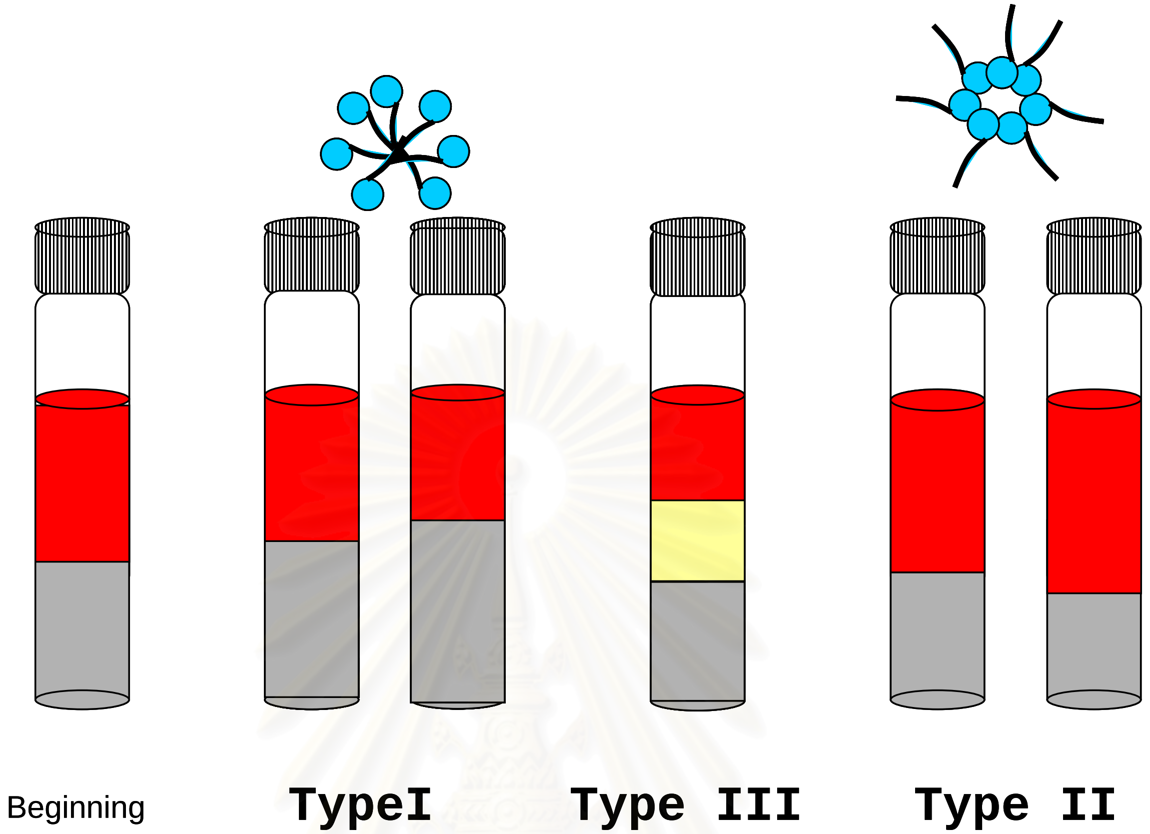


Figure 2.6 Winsor Phase Diagram

A transformation from in the system Winsor type I to type III to type II can be achieved by progressively changing the temperature, salinity, the molecular structure of surfactant and cosurfactant, an oil to water ratio, or the structure of oil in homogeneous series. This phase diagram, illustrated in Figure 2.7, is known as a salinity scan. By increasing the salinity, one can simply visualize a progressive change in the phase diagram.



O = excess oil phase
W = excess water phase
M = middle phase microemulsion

W_m = oil in water microemulsion
O_m = water in oil microemulsion

Figure 2.7 Effect of Salinity Scan on Phase Diagram of Microemulsion

Rodriguez et al. (2003) studied the phase behavior and structure of a sucrose ester/water/oil system in the presence of a long-chain cosurfactant (monolaurin) and a small amount of an ionic surfactant. They utilized study and small angle X-ray scattering. In the water/sucrose ester/monolaurin/decane system at 27 ^{o}C, instead of a three-phase microemulsion, lamellar liquid crystals were formed in the dilute region. Unlike other systems in the presence of alcohol as a co-surfactant, the HLB composition does not change with dilution, since monolaurin adsorbs almost completely in the interface. The addition of a small amount of ionic surfactant, regardless of the counterion, increases the solubilization of water in W/O microemulsions. The solubilization of oil in O/W microemulsions is not much affected, but structuring is included and a viscous isotropic phase is formed. At high ionic surfactant concentrations, the single-phase microemulsion disappears and liquid crystals are favored.

Watarai (1997) focused on some characteristic functions of o/w (L_1) and w/o (L_2) single phase microemulsion and two-phase microemulsion systems of Winsor type I and Winsor type II from the viewpoint of their use as separation media in solvent extraction, liquid chromatography and capillary electrophoresis. The practical advantage of microemulsion media in the separation of metal ions and biological compounds were assessed.

2.8 Interfacial Tension

Since a number of applications require low interfacial tensions between oil and water to achieve the desire state, interfacial tension is one of the most commonly measured properties of microemulsions. The interfacial tension or surface free energy per unit area is the minimum amount of work required to bring sufficient molecules from the interior to the surface to expand the surface by unit area.

At the interface between two condensed phases, the dissimilar molecules in the adjacent layer facing each other across the interface also have potential energies different from those in their respective phases. If surfactant is added to a system of two immiscible phases (e.g., hexane and water) and then adsorbed at the interface between them, it will be oriented with the hydrophilic group toward the water and the hydrophobic group toward to hexane. The tension across the interface is significantly reduced by the presence of the surfactant at the interface (Myers, 1999).

CHAPTER III

METHODOLOGY

3.1 Materials

3.1.1 Surfactants

Surfactants used in this research were secondary alcohol ethoxylate tergitol 15 S-3, secondary alcohol ethoxylate tergitol 15 S-5, sorbitan monooleate (Span 80), Coconut fatty acid diethanolamide (Comperlan KD), Texapon HBN (Sodium Dodecyl Sulfate) (98% active),and Alfoterra 145-5PO.

Secondary alcohol ethoxylate tergitol 15 S-3, a nonionic surfactant with 100 % activity was purchased from Dow Chemical Co. Secondary alcohol ethoxylate tergitol 15 S-5, a nonionic surfactant with 100% activity, was obtained from Dow Chemical Co. Sorbitan monooleate (Span 80), an anionic surfactant with 100 % activity was obtained from ICI Uniquema. Coconut fatty acid diethanolamide (Comperlan KD), a nonionic surfactant with 97% activity was obtained from Henkel. Texapon HBN (Sodium Dodecyl Sulfate) (98% active), an anionic surfactant with 98 % activity was purchased from Sigma Aldrich. Alfoterra 145-5PO, an anionic surfactant with 28.7 % activity, was obtained from Sasol Co. Properties; and the selected characterizations of these surfactants are shown in Table 3.1.

3.1.2 Oils

The soybean oils used in this study were commercial soybean oil and soybean oil. The commercial soybean oil was purchased from Loxley Trading. The soybean oil was purchased from Sigma Aldrich.

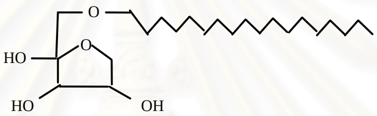
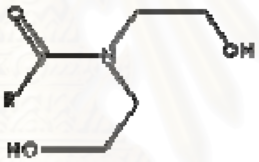
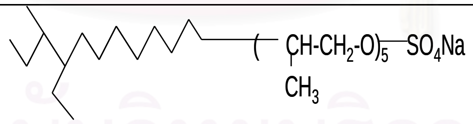
3.1.3 Soybean Seed

The commercial soybean seed was purchased from Krungsri Ayuthaya International Industrial Food

3.1.4 Electrolytes

The electrolytes used in this research were sodium chloride. Sodium chloride (NaCl), analytical grade with 99 % purity, was purchased from Carlo ERBA reagent Co. Ltd

Table 3.1 Properties and Selected Characterizations of Surfactants

Chemical name	Chemical structure	Type	MW	HLB
Secondary alcohol ethoxylate tergitol 15 S-3	$C_{12-14}CH_{25-29}[CH_2CH_2O]_xH$	Nonionic	340.6	8.0
Secondary alcohol ethoxylate tergitol 15 S-5	$C_{12-14}CH_{25-29}[CH_2CH_2O]_xH$	Nonionic	428.6	10.5
Sorbitan monooleate (Span 80)		Nonionic	428.6	4.3
Coconut fatty acid diethanolamide (Comperlan KD)		Nonionic	280	-
Texapon HBN (Sodium Dodecyl Sulfate)		Anionic	288	10.2
Alfoterra 145-5PO		Anionic	595	14.9

3.1.5 Water

Ultra pure water was used throughout this research for preparing aqueous surfactant solutions and other chemical solutions. It was also used as rinse water and for cleaning glassware.

3.1.6 Dyed Oil

Oil red O (solvent Red 27, CI No. 26125) was purchased from Aldrich Chemical Company, Inc. It was used for preparing the dyed oil solution to be applied on soybean oil and soybean seed.

3.2 Methodology

This study was divided into two parts: the phase behavior study and soybean oil extraction. Figure 3.1 shows the experimental procedure diagram.

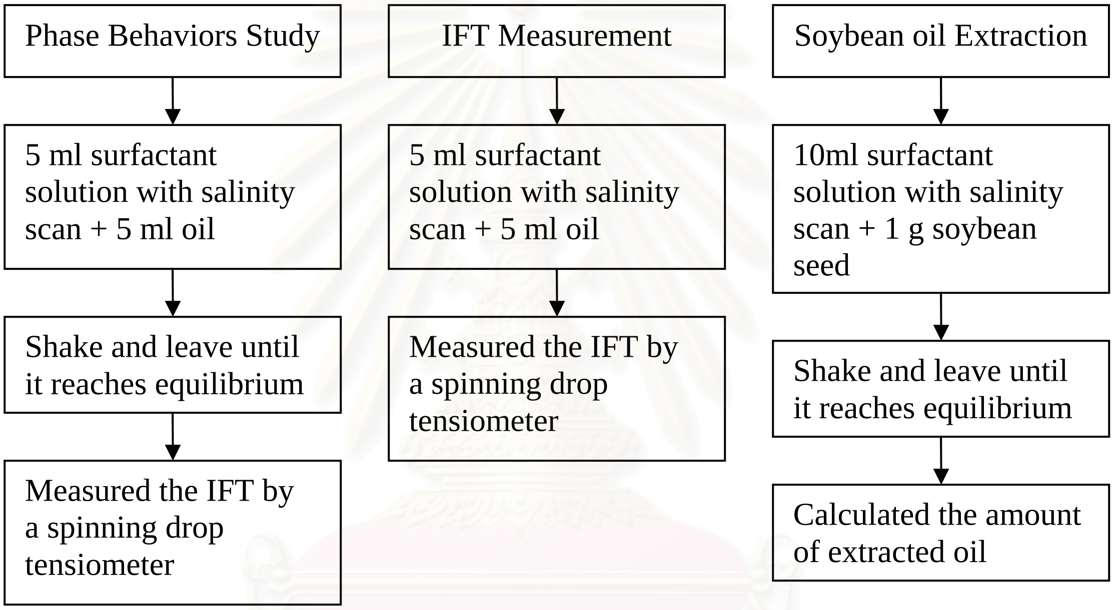


Figure 3.1 Experimental Procedure Diagrams

3.2.1 Phase Behavior Study

Phase behavior were investigated for systems of soybean oil with various types of surfactant solutions such as secondary alcohol ethoxylate (Tergitol 15 S-3), secondary alcohol ethoxylate (Tergitol 15 S-5), coconut fatty acid diethanolamide (Comperlan KD) with Texapon HBN (Sodium Dodecyl Sulfate) and secondary alcohol ethoxylate Tergitol 15 S-3, and secondary alcohol ethoxylateTergitol 15 S-5 with sodium dioctyl sulfosuccinate(AOT). All systems were conducted with salinity scans. As the soybean oil has similar color to the surfactant solution, the differences in phase transition can be clearly shown by dying red color. The phase studies for microemulsion transition with a salinity scan were prepared in the 15 mL flat-bottomed tubes. The ratio for the oil and surfactant solution was unity. After the surfactants solutions and oil were added to tubes, they were gentle shaken and left to reach equilibrium at 30^{o}C. The phase behavior of the microemulsion systems were explained by their phase transition which can be visually observed after the systems reaches equilibrium and by the interfacial tension between the oil and surfactant solutions. Table 3.2 summarize the condition for each system in preliminary experiment for investigation the surfactant systems and Figure 3.2 show how to prepare phase study experiment.

Table 3.2 Surfactant Systems Studied in the Phase Behavior Study

No	System	Salinity scan (NaCl)	Temperature(°C)
1	3-5 %Tergitol 15S3	0-20	30
2	3-5%Tergitol 15S5	0-20	30
3	3-5%Comperland KD)	0-20	30
4	3-5%SDS	0-20	30
5	3-5%Spand80	0-20	30
6	0.1-1 Alfoterra5PO	0-20	30
7	(3-5%Comperland KD) + (3-5%SDS)	0-20	30
8	(3-5%Comperland KD)+(3-5% Spand80%)	0-20	30
9	(3-5%ComperlandKD)+ (0.1-1%Alfoterra 5PO)	0-20	30
10	(3-5%Tergitol 15S3) +(0.1-1 Alfoterra5PO)	0-20	30
11	(3-5%Tergitol 15S5) + (0.1-1 Alfoterra5PO)	0-20	30
12	(3-5%Tergitol 15S3) + (3-5%Spand80)	0-20	30
13	(3-5%Tergitol 15S5) +(3-5% Spand80)	0-20	30
14	(3-5%Tergitol 15S3) + (3-5%SDS)	0-20	30
15	(3-5%Tergitol 15S5) + (3-5%SDS)	0-20	30
16	(3-5%Spand80)+ (0.1-1 Alfoterra5PO)	0-20	30

3.2.2 Interfacial Tension Measurement in Batch Study

The interfacial tension (IFT) between lower phase (surfactant solution) and upper phase (soybean oil) of each system were measured by a spinning drop tensiometer (Dataphysics, Model SVT20) to select the suitable surfactant solution system for the study on soybean extraction. The ratio of the oil and water volume was 0.01: 2 and the spinning speed was 3000 rpm.

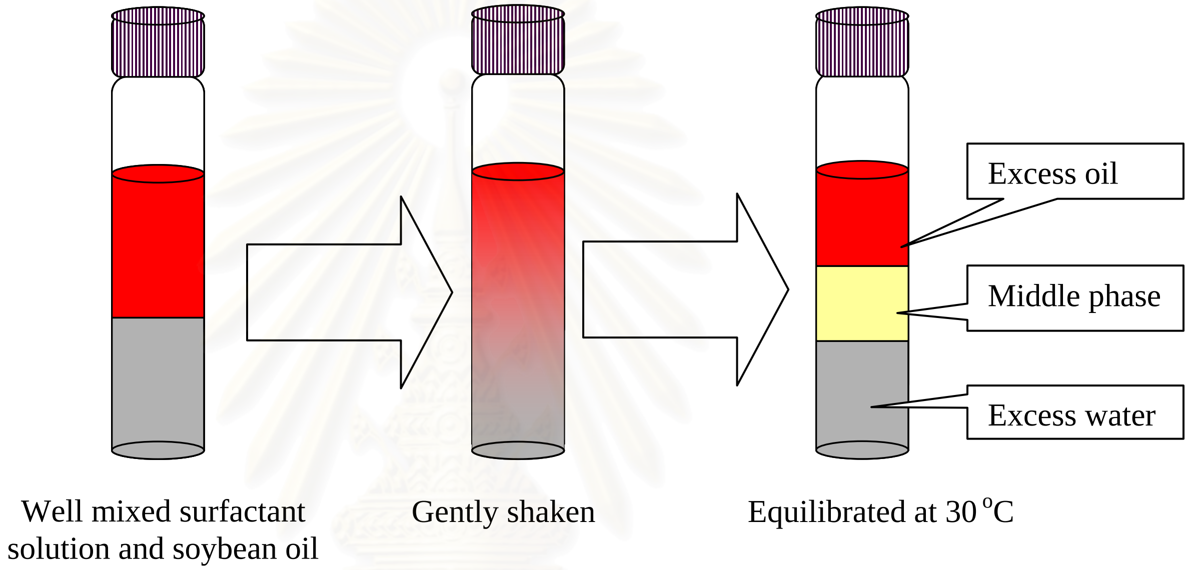
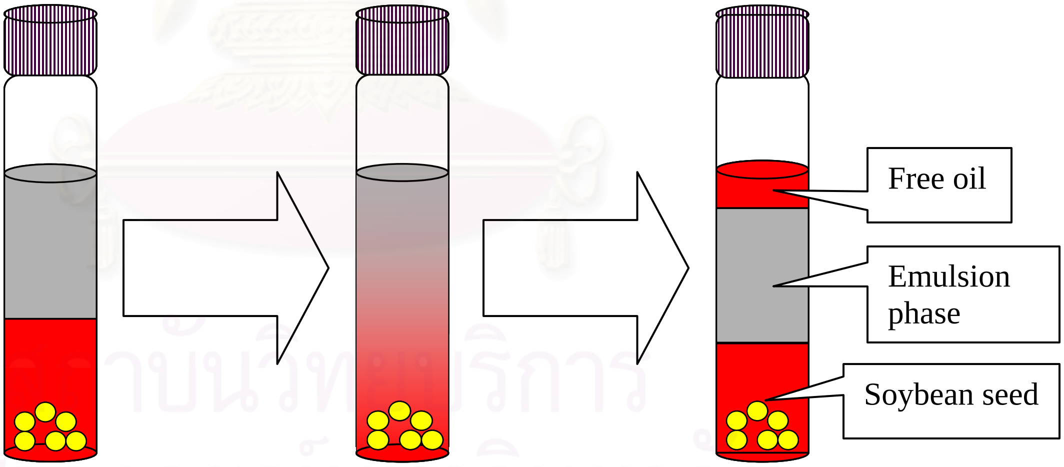


Figure 3.2 Schematic Experiment of Microemulsion Formation

3.2.3 Soybean Oil Extraction

Soybean oil extraction was used to for determination the optimum conditions of using surfactants as a solvent replacement in extracting soybean seeds. The assumption used for this study was based on that the soybean seed is contains oil 20% by weight (Gunstone, 2002), so the calculation for the total oil in soybean seeds can be obtained from their initial concentration of this 20% wt. Moreover, since this study used the surfactant that is hydrophilic surfactant, it was assumed that in the free oil phase the amount of surfactant is insignificant. The soybean seed was mixed homogeneously with dye so that the free oil phase can be easily distinguished from the emulsion phase and the free oil is obviously seen.

In soybean oil extraction, there are two different aqueous surfactant solution systems; a mixture of water, Alfoterra 5PO, Spand 80, and NaCl and a mixture of water, Alfoterra 5PO, Comperland KD and NaCl. The grained soybean was mixed with an aqueous surfactant solution in the flat-bottomed screw-capped tube. The solutions were mixed by vortex for 30 seconds, shaken at 180 rpm for 30 minutes, and centrifuged at 5000 rpm for 10 minutes, and then stored in incubator at 30°C. The optimum condition for soybean extraction were investigated by varying three parameters; the salinity, soybean load, grain sized, and agitation or shaking duration during the extraction procedure. There were 6 NaCl concentrations (wt %) for soybean oil extraction; 1, 2.5, 5, 7.5, 10, and 12.5. There were five soybean load (g) for soybean oil extraction; 0.5, 1, 1.5, 2, and 2.5. The typical flake size which is used in USA industrial processes, is 0.210 mm - 0.420 mm (Lui, 1999). Thus in this present study, there were three grain size ranges for soybean oil extraction; larger than 35 Mesh (0.425 mm), fine size: Mesh 35 - Mesh 65 (0.212- 0.425 mm), and very fine size of less than Mesh 65 (0.21 mm) were varied to determine which grain size yielded the high free oil phase.. There were 6 contact times for soybean oil extraction; 10, 20, 30, 40, 50, and 60 min.



Well mixed surfactant solution and soybean

Mixed by vortex for 30 seconds, shaken at 180 rpm for 30 minutes, and centrifuged at 5000 rpm

Equilibrated at 30 ^{o}C

Figure 3.3 Schematic Experiment of Soybean Oil Extraction

CHAPTER IV

RESULTS AND DISCUSSIONS

4.1 Phase Behavior Study

4.1.1 Investigation for System of Microemulsion with Soybean Oil

In order to select surfactant system for soybean oil extraction from soybean seeds, several surfactants were used to determine if any systems could be able to form microemulsion with the oil. The phase transition of each system was observed to ensure that microemulsion was formed. The experimental surfactants systems were carried out by salinity scan. The preliminary result of all surfactant systems with the soybean oils were shown in Appendix A. Two systems of mixed surfactants were selected for further study due to their capability of microemulsion formation with soybean oil as well as, the phase transition could be observed. The two systems are (1) the mixture of 3% Comperland KD and 0.1% Alfoterra 5 PO, and (2) the mixture of 4% Spand 80 and 0.1% Alfoterra 5 PO. For these two mixtures, other concentration of each surfactant were also studied, however, these two mixed surfactants system were selected because they can form Winsor type III microemulsion at the lowest concentration of surfactants and salinity. Table 4.1 shows the microemulsion phase transition for two selected surfactant systems with commercial soybean oil.

Vegetable oils e.g. soybean oil, canola oil, etc. are considered as an oil that is very difficult to form microemulsion with a surfactant system, especially, a single surfactant system. This is because most of them combine with long chain fatty acids with bulky structure and high hydrophobicity. Several research works have been reported for microemulsion with vegetable oil or triglyceride or triolein by mixture of surfactants (Alander and Warenheiem,1989; Chen et.al. (1997); and Tongcumpou et al., 2003) Alander and Warenheiem (1989) described the difference of phase behaviors of long chain triglycerides and hydrocarbons in details with both systems of nonionic and ionic surfactants. Their results showed that in the same surfactant system, replacing methyl ester with medium chain triglycerides or peanut oil affected both solubilization pattern and phase behavior significantly as a result of the large molecular size of triglycerides. While Tongcumpou et al. (2003) used mixture of nonionic secondary alcohol ethoxylate mixed with another two anionic surfactants to form microemulsion with triolein.

Table 4.1 Microemulsion Phase Transition for the Selected Surfactant Systems with Commercial Soybean Oil at Different NaCl Concentration

No	%NaCl	Mixed surfactants system of 3%Comperland KD and 0.1%Alfoterra 5PO	Mixed surfactants system of 3% Span 80 and 0.1% Alfoterra 5PO
		Phase Transition	Phase Transition
1	1.0	I	I
2	2.5	I	I
3	5.0	III	III
4	7.5	III	III
5	10.0	II	II
6	12.5	II	II
7	15.0	II	II
8	17.5	II	II
9	20.0	II	II

An extended surfactant is one of anionic surfactant that has been introduced to be a mixture of surfactant system to enhance solubilization and microemulsion formation with long chain hydrocarbon as well as vegetable oil. Minana-Perez et.al.(1995) introduced an extended surfactant and found that it could be able to form soybean oil-in-water microemulsion. Soybean oil was employed in another study examining the effect of oil type on phase behavior of oil-in-water microemulsion stabilized with nonionic surfactant. Long-chain triglycerides oil likes soybean oil is more difficult to solubilize in aqueous solution than single chain oils.

For this study, we selected Alfoterra 5PO to be an anionic surfactant to mix with another nonionic surfactant to form microemulsion with our commercial soybean oil and we found that microemulsion can be observed as mentioned earlier. Therefore, two selected systems; (1) the mixture of 3% Comperland KD and 0.1% Alfoterra 5 PO, and (2) the mixture of 4% Spand 80 and 0.1% Alfoterra 5 PO were selected for oil extraction study. For further experiment, these two surfactant systems were examined the property of interfacial tension since it is expected that to be one of key parameters to influence the oil extraction from soybean seeds (Tongcumpou et al., 2003).

4.1.2 Interfacial Tension between Mixed Surfactant Solution and Soybean Oil

Interfacial tension between oil and water is related to free energy difference of the interfacial film at the planar interface separating the bulk phase and the curve interface of microemulsion droplet. The microstructure of the middle phase is considered to be bicontinuous phase (Acosta et al, 2003) and as a consequence, it curvature is almost planar. Hence, the difference in free energy is very low, resulting in an ultra low interfacial tension value.

Due to its properties of microemulsion which provides ultralow interfacial tension (IFT), however, vegetable oil is one of the oils that hardly found to provide such a low IFT between the oil and aqueous phase microemulsion (Winsor Type I) or surfactant solution, it is expected that with an extended surfactant, IFT may be in the range of 10^{-1}-10^{-2} mN/m. Thus in this part of experiment, the IFT between soybean oil and the two selected surfactant systems which salinity scan that form Winsor Type I and Type III were measured by using a spinning drop tensiometer.

In this experiment, the IFT measurements were compared between the two sources of oil: commercial soybean oil and soybean oil (without any additive). In general, some additives such as vitamins are added into the commercial soybean oil. Since further experiment, commercial soybean oil would be used, this part of experiment was conducted to ensure that some additives added in commercial soybean insignificantly affected the result.

The NaCl scan was conducted for these two systems. Tables 4.2 and 4.3 show the values between the systems of mixed 3% Comperland KD and 0.1% Alfoterra 5 PO and mixed of 4% Span 80 and 0.1% Alfoterra 5 PO at different salinity with commercial soybean oil and soybean oil respectively. Figures 4.1 and 4.2 demonstrate the correlation between interfacial tension and salinity of the two mixed surfactant system for commercial soybean oil and soybean oil. The interfacial tension between commercial soybean oil and surfactant solution gradually decreased as the concentration of NaCl increases. Adding salt can promote the formation of middle phase microemulsion because salt can reduce the repulsive force between the ionic charges of surfactants at their head group (Tongcumpou et al., 2003).

Table 4.2 Interfacial Tension of Commercial Soybean oil and Soybean Oil with 0.1% Alfoterra 5PO and 3% Comperland KD at Different NaCl Concentrations

No	3% Comperland KD + 0.1%Alfoterral 5 PO with salinity scan					
	%NaCl	Commercial Soybean Oil		Soybean Oil		Microemulsion Winsor Type
		IFT(mN/m)	STDEV	IFT(mN/m)	STDEV	
1	1.0	0.170	0.00557	0.208	0.0065	I
2	2.5	0.176	0.00681	0.252	0.0476	I
3	5.0	0.275	0.00500	0.304	0.0060	III
4	7.5	0.116	0.00833	0.107	0.0053	III
5	10.0	0.167	0.00702	0.137	0.0119	II
6	12.5	0.365	0.01249	0.314	0.013	II

Table 4.3 Interfacial Tension of Commercial Soybean Oil and Soybean Oil with 0.1% Alfoterra 5PO and 4 % Span 80 at Different NaCl Concentrations

No	4% Span 80 + 0.1%Alfoterral 5 PO with salinity scan					
	%NaCl	Commercial Soybean Oil		Soybean Oil		Microemulsion Winsor Type
		IFT(mN/m)	STDEV	IFT(mN/m)	STDEV	
1	1.0	0.877	0.0215	2.747	0.4314	I
2	2.5	0.528	0.0266	0.444	0.0272	I
3	5.0	0.358	0.0141	0.331	0.0336	III
4	7.5	0.214	0.0254	0.163	0.0112	III
5	10.0	0.362	0.0154	0.288	0.0015	II
6	12.5	0.715	0.0087	0.613	0.0101	II

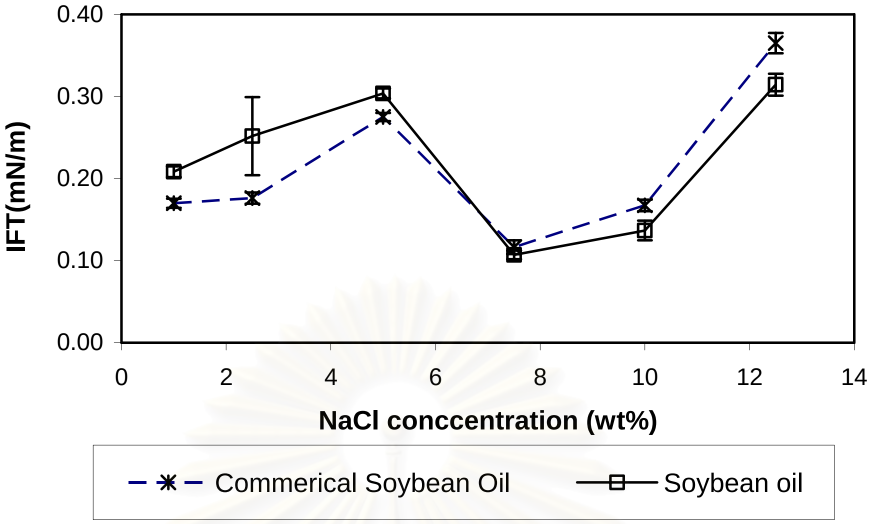


Figure 4.1 The Plots between Interfacial Tension and the System of 0.1% Alfoterra 5PO and 3% Comperland KD with Commercial Soybean Oil and Soybean Oil at Different NaCl Concentrations

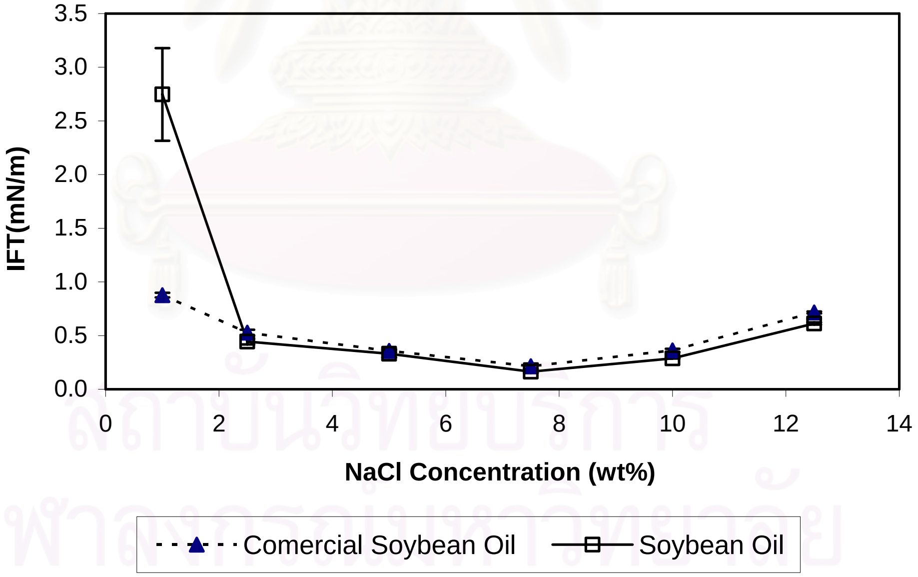


Figure 4.2 The Plots between Interfacial Tension and the System of 0.1% Alfoterra 5PO and 4% Span 80 with Concentration of Commercial Soybean Oil and Soybean Oil at Different NaCl Concentrations

From these results as shown in Figure 4.1 and 4.2, the IFT decreases as the salinity increases. The IFT reaches the minimum point at 7.5 wt% NaCl for both oils. The IFT of soybean oil is slightly lower than commercial soybean oil. The IFT decreasing is related to hydrophile-lipophile balance (HLB) of the system (Holmberg, 2003). The HLB is a parameter that shows partitioning of surfactant between oil and water phases relative to surfactant hydrophobicity. With decreasing HLB the surfactant moves from the water phase to the oil phase as the surfactant system becomes more hydrophobic. Added salt can promote the formation of middle phase microemulsion because salt can reduce the repulsive force between the ionic charges of surfactants at their head group (Rosen, 2004). A further increase in salinity results in increasing the IFT indicating that the system transforms form type III to type II microemulsion.

4.2 Soybean Oil Extraction

The mixed surfactants of 3% Comperland KD and 0.1% Alfoterra 5PO and the system of 4% Span 80 and 0.1% Alfoterra 5PO were selected to conduct the soybean oil extraction because it can form middle phase microemulsion and use the lowest concentration of surfactant and salinity. Microemulsion is a system proposed for this study since its properties are thermodynamically stable, and providing low interfacial tension. The experimental data and calculation methods of the soybean oil extraction are shown in Appendix C.

4.2.1 Oil Extraction by Selected Surfactant at Different Salinity Scan

For this part, in order to obtain optimum free oil extraction from the two selected systems at different salinities, soybean seeds were varied into 3 sizes namely; coarse size: larger than 35 Mesh (0.425 mm), fine size: Mesh 35 - Mesh 65 (0.212- 0.425 mm), and very fine size of less than Mesh 65 (0.21 mm) for this experiment. The extraction conditions was carried out by using 10 ml of the surfactant solution mixed with grinded soybean seeds and shake for 30 minutes and then centrifuged for 15 minutes to separate different phases of the system. The amount of soybean extraction was measured from the volume of oil free phase. The experimental results showed that only the system of mixed of 3% Comperland KD and 0.1% Alfoterra 5PO that was found to be able to extract and yield free soybean oil while the system of 4% Span 80 and 0.1% Alfoterra 5PO only yielded aqueous emulsion phase.

Figure 4.3 shows the percentage of oil extraction at different salinities. It was found that with decreasing grain size from larger than 35 Mesh to 35 Mesh-65 Mesh, the % extraction increased because the surface area is increased. A further decrease in grain size from 36 Mesh-65 Mesh to less than 65 Mesh, the % extraction slightly increased. The % extraction increased as the grain size decreased as the surfactant has higher capability to reduce the capillary force on soybean seed when the surface area of soybean seed is increased.

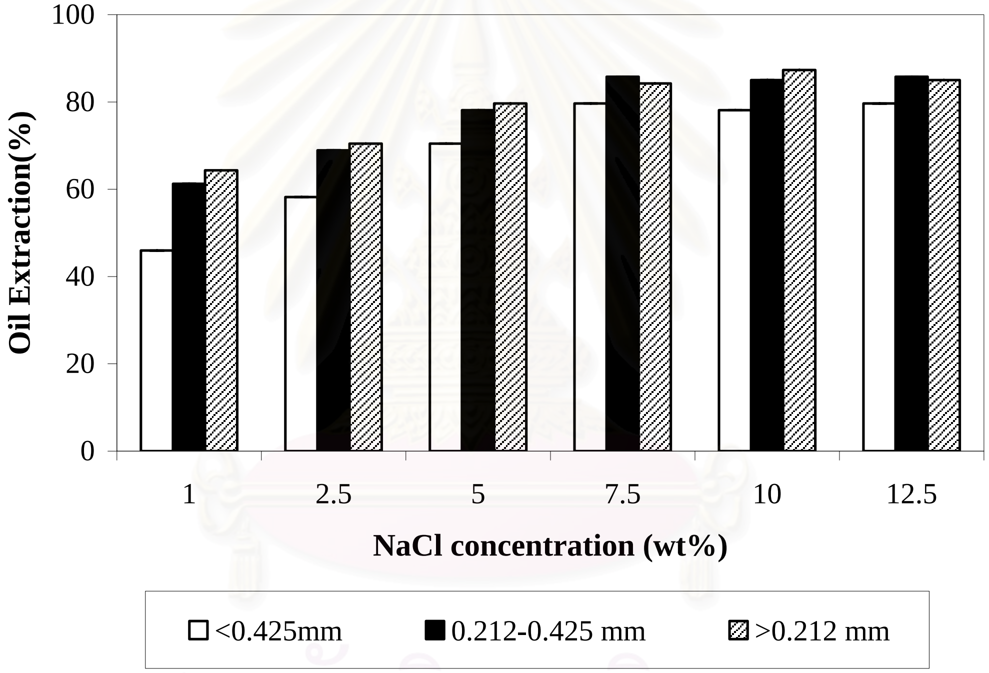


Figure 4.3 The Oil Extraction (%) from Soybean Seeds by Using with the System of Mixed 0.1% Alfoterra 5PO and 3% Comperland KD at Different Salinities and Grain Sizes

From this part of experiment, the middle range (35-65 Mesh) is the most appropriate diameter for soybean seed because if the size is smaller, there is insignificantly different in amount of oil extracted which is not worth-doing comparing with energy consumption in grinding. If the size is larger, the surface area is reduced thus decreasing the amount of oil extracted. Moreover, this range is the typical flake size in US industrial processes (Gunstone, 2002). So we decided to use this range for further study.

As can be seen in Figure 4.4, the results showed that the % oil extraction increases as the concentration of NaCl increases. At NaCl ≥7.5 wt%, there is no change in % extraction. If the NaCl concentration is higher, there is negligible increase in amount of oil extracted. Consequently, the optimum salinity is considered at 7.5 wt%.

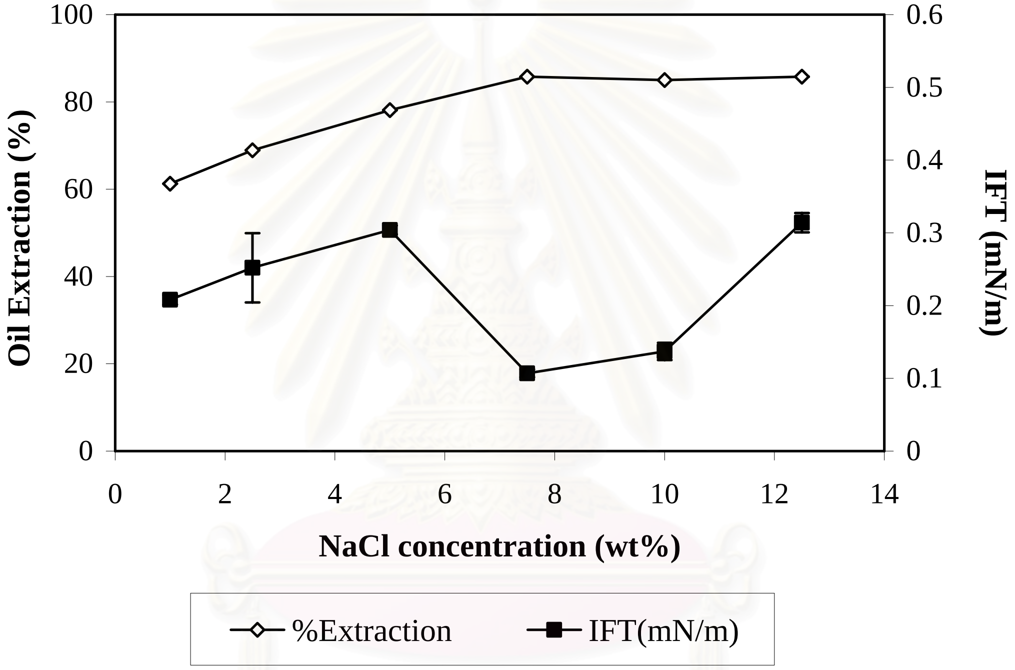


Figure 4.4 Correlation of the Percent Oil Extraction and NaCl Concentration in the System of 0.1% Alfoterra 5PO and 3% Comperland KD

4.2.2 Oil Extraction by a Single Surfactant at Different Salinity Scan

From comparison of IFT and oil extraction as shown in Figure 4.4, it indicates that even though IFT is an important parameter to reduce capillary force and detach the oil from soybean seeds, it is not directly proportional to % extraction. This means that at certain level of IFT that is low enough to overcome the force may already be good condition. However, for application of microemulsion on oil extraction from seeds may have to consider for solubilization as well. Generally, the optimum condition for the oil extraction should be the surfactant system that provides low IFT, but not high solubilization since oil free phase is desirable from the process. Consequently, microemulsion may not be the best system for the oil extraction. So, we introduced a single surfactant for oil extraction experiment in comparison.

For using a single surfactant systems for oil extraction, Comperland KD and Span 80 were varied in the range of 3-5% while Alfoterra 5 PO was varied in the range of 0.1-1% In the system of Alfoterra 5 PO, and Span 80 with salinity scan, free oil phase could not be obtained only the extraction with Comperland KD that oil free phase could be observed. Figure 4.5, 4.6 and 4.7 show the percent soybean oil extraction of from soybean seeds by Comperland KD at concentrations 3%, 4% and 5% with different salinities.

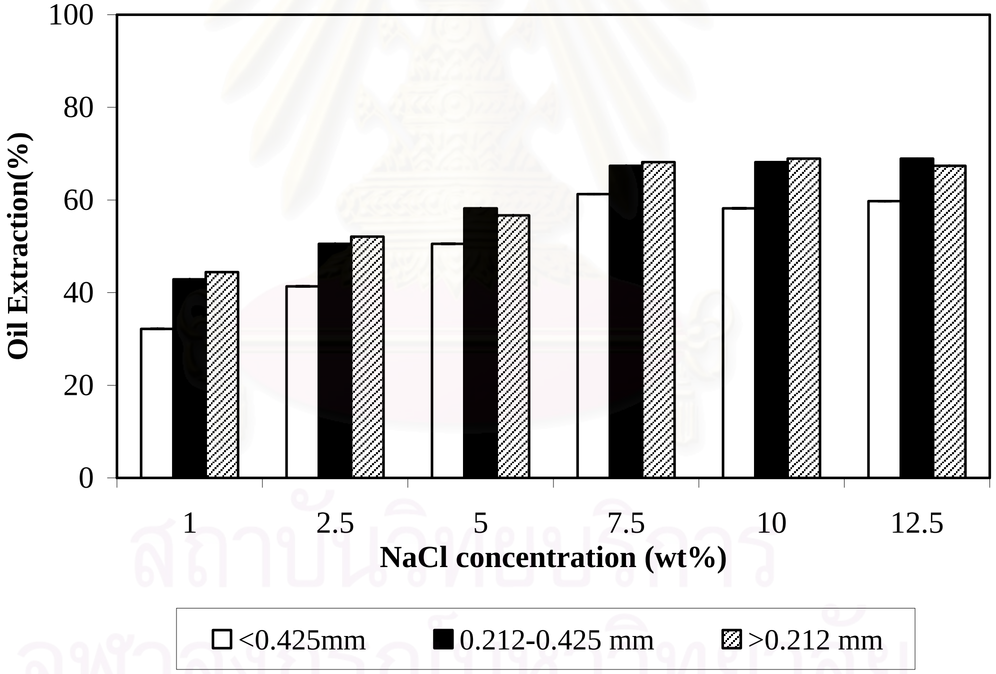


Figure 4.5 Comparison of Oil Extraction from Soybean Seeds by 3% Comperland KD with Different NaCl Concentrations

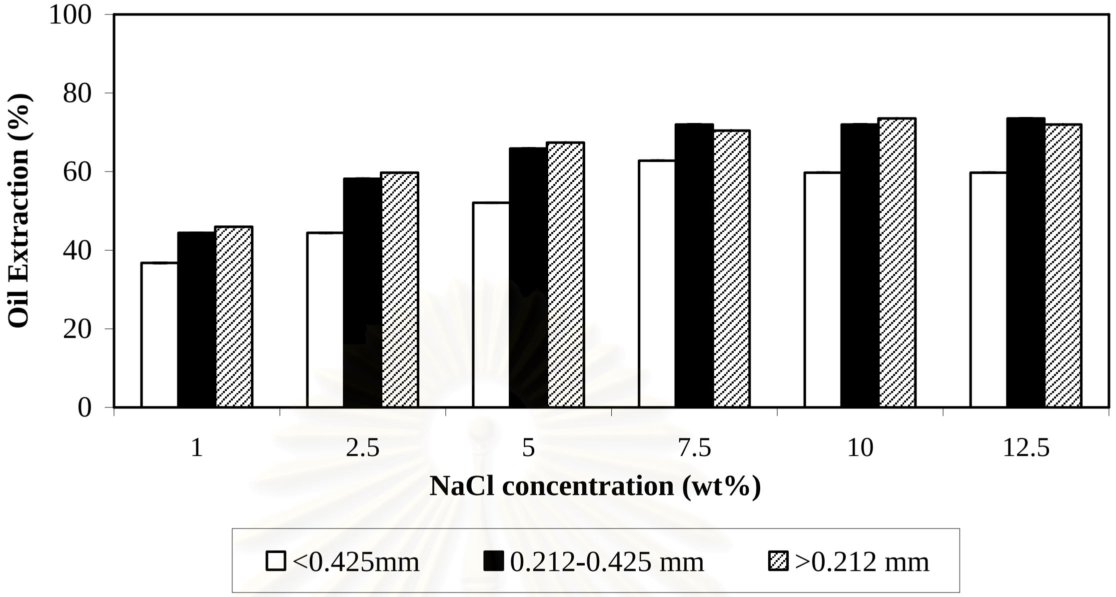


Figure 4.6 Comparison of Oil Extraction from Soybean Seeds by 4% Comperland KD with different NaCl Concentrations

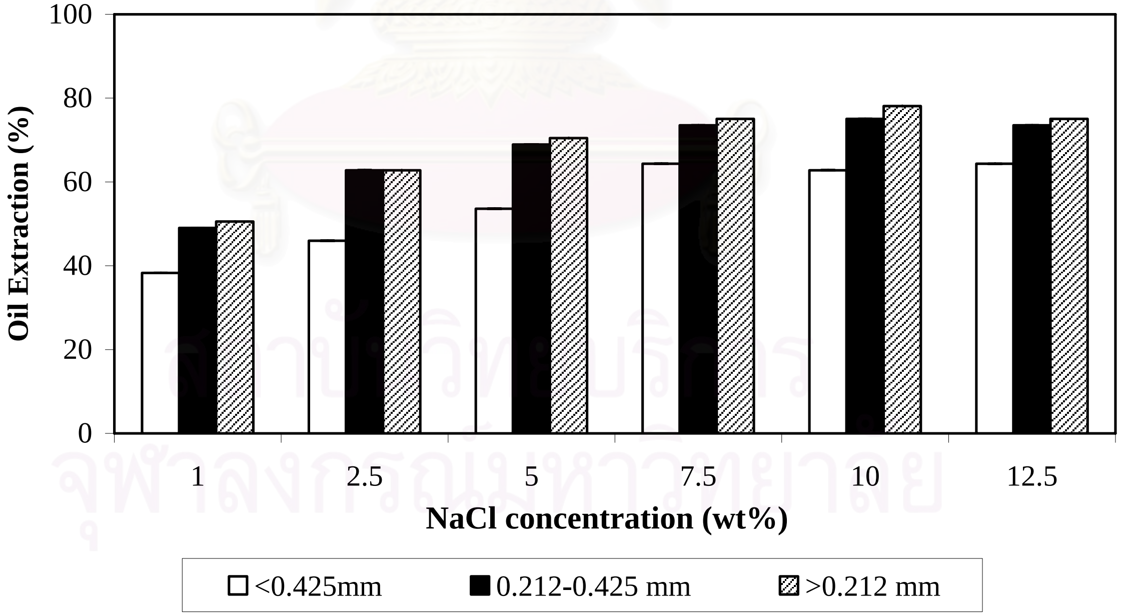


Figure 4.7 Comparison of Oil Extraction from Soybean Seeds by 5% Comperland KD with Different NaCl Concentrations

In order to get better understanding for single oil surfactant system, IFT values between surfactant solution and soybean oil were measured. For most cases, the IFT values were found suitably low enough for extraction which was in the range from 0.1 to 0.9 mN/m. Figure 4.8 is the plot to compare IFT values and % oil extraction for the system of Comperland KD at different concentrations of surfactants and NaCl. The result showed that the % oil extraction is increased as the amount of Comperland KD increase. However, the addition of Comperland KD from 4% to 5% shows only a slight increase in % extraction. For the discussion of salinity, the results showed that the % oil extraction is increased as the concentration of NaCl increases. At NaCl ≥7.5 wt%, there is no change in % extraction. If the NaCl concentration is higher, there is negligible increase in amount of oil extracted. Thus, the optimum salinity for oil extraction is considered at 7.5 wt%.

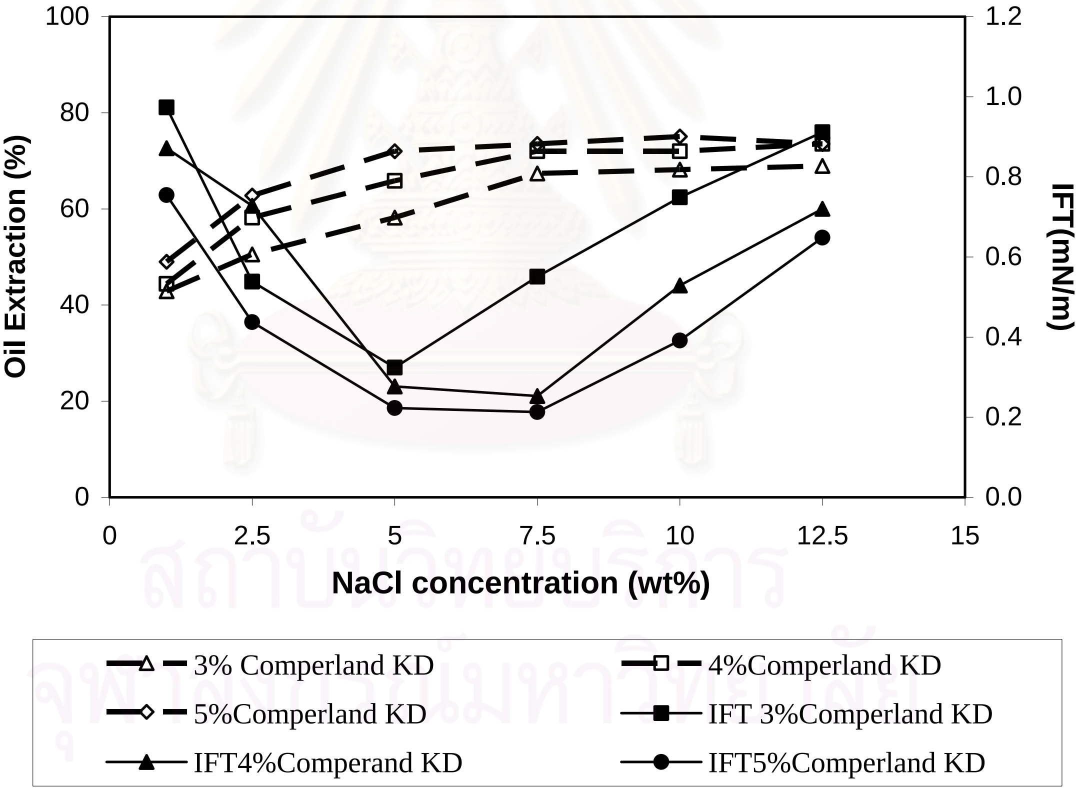


Figure 4.8 Comparison of Oil Extraction from Soybean Seeds by Comperland KD with Different NaCl Concentrations

To summarize the efficiency of extraction of the mixed surfactant system and single surfactant systems, the % oil extraction grom the fine grain grinded soybean seeds which were obtained from same condition (1 gm of grinded soybean seeds, 10 ml surfactant, 30 min shaking and centrifuge for 10 min), are compared as shown in Table 4.4. The result shows that the mixed surfactant is still more efficient for oil extraction as compared to a single one. Therefore, systems of mixed surfactant were used to determine for other parameters on different contact times and soybean seeds load.

Table 4.4 Comparison of % Oil Extraction by a Mixed Surfactant and Single Surfactant Systems for 1 g of Grinded Soybean Seeds at Size 0.212-0.425 mm

Surfactant solution used for extraction	**% Soybean Oil Extraction**					
	1.0% NaCl	**2.5% NaCl**	**5.0% NaCl**	**7.5% NaCl**	**10% NaCl**	**12.5% NaCl**
Mixed of 3% Comperland KD and 0.1% Alfoterra 5PO	61.27	68.93	78.12	85.77	85.01	85.77
3% Comperland KD	42.89	50.55	58.2	67.39	68.16	68.93
4% Comperland KD	44.42	58.2	65.86	71.99	71.99	73.52
5% Comperland KD	49.01	62.8	68.93	73.52	75.05	73.52

4.2.3 Efficiency of Soybean Oil Extraction at Varied Contact Time

The contact time is a parameter that influences the efficiency of extraction as it is the time to allow surfactant to coalesce oil in the grinded soybean seeds and detached it from solid phase to surfactant solution. Figure 4.9 shows the plot of % oil extraction at different contact time for the mixed surfactant at different salinities. As it can be seen in Figure 4.9, the % oil extraction increased as the shaking time increased and allowed the surfactant solution to contact with oil. However, at time ≥30 min, the plot is became plateau. So, the optimum contact time is 30 min.

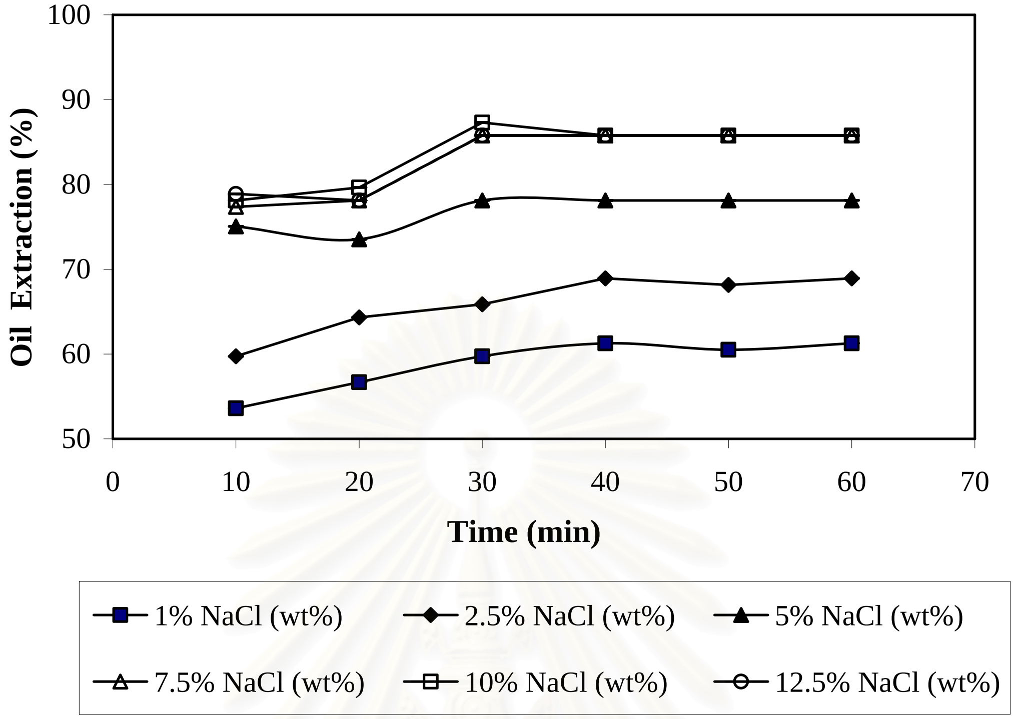


Figure 4.9 Comparison of the Oil Extraction and Contact Times of Soybean Seed and 0.1% Alfoterra 5PO and 3% Comperland KD

4.2.4 Efficiency of Soybean Oil Extraction at Varied Soybean Load

The system of mixed surfactant of 0.1% Alfotera 5PO and 3% Comperland KD at 7.5%, 10.0% and 12.5% NaCl were selected for this experiment. It was carried out by varied soybean loads from 0.5 to 2.5g with the condition of 10 ml surfactant solution, 30 min shaking, and 10 min centrifuge. This experiment aimed to seek the optimum load that yielded highest free oil from the selected surfactant system. As it can be seen Figure 4.10, the results showed that the optimum soybean load is 1 g since at the soybean load was higher than 1 g, soybean load started to decrease the efficiency of extraction. This may be because higher mass of soybean load lead to less coalesce between surfactant solution and the oil.

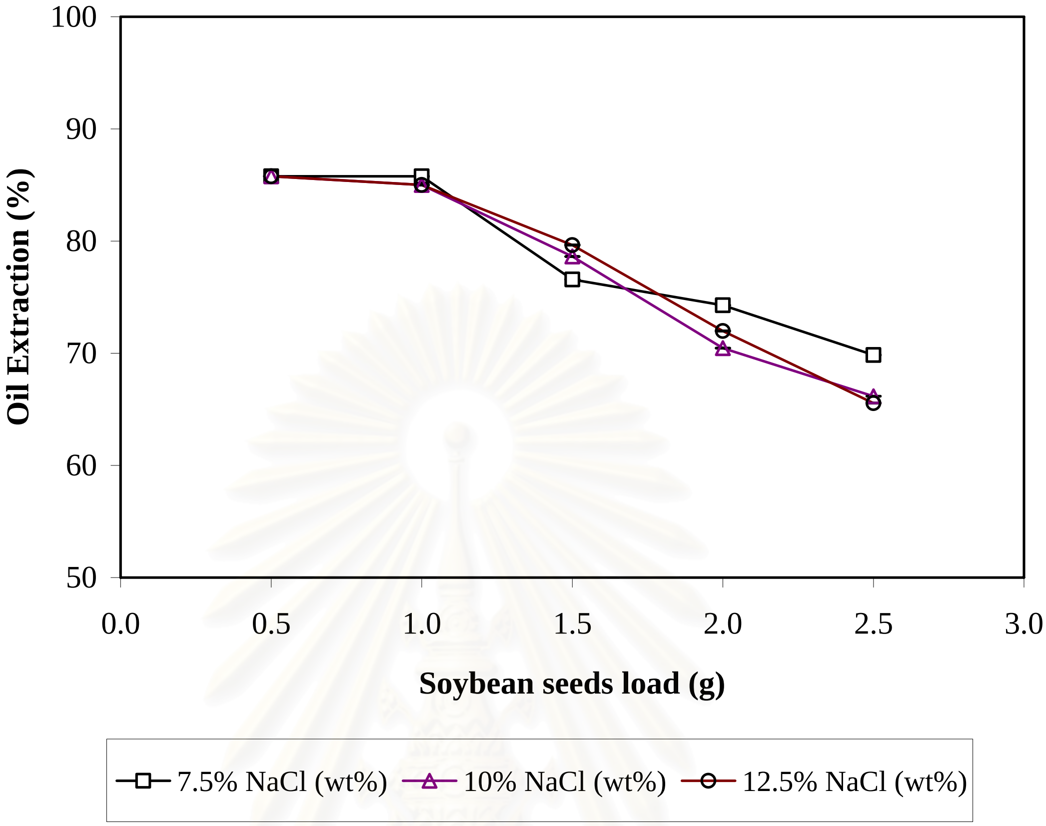


Figure 4.10 Comparison of Relationship between Oil Extraction and Soybean Load of Soybean Seed with 0.1% Alfoterra 5PO and 3% Comperland KD

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CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

This study was conducted to extract oil from soybean seeds using the surfactant systems approach. The objective of this study was to apply the microemulsion formation for soybean oil extraction. The specific application was to develop surfactant-based aqueous solvents in order to replace organic solvents in cleaning processes and soybean oil extraction. This alternative can be categorized as one of Green Chemistry. This study was introduced to maximize soybean oil mobilization while minimizing soybean oil solubilization.

For this study, the result showed that the surfactant mixture of 3%Comperland KD and 0.1% Alfoterra 5PO and the surfactant mixture of 4%Span 80 and 0.1%Alfoterra 5PO were able to form microemulsion with soybean oil by using the lowest concentration of surfactant and salinity, so these two systems were selected to soybean oil extraction. For further study, the experimental results showed that only the system of mixture of 3% Comperland KD and 0.1% Alfoterra 5PO that is found to be able to extract and yields free soybean oil while the system of 4% Span 80 and 0.1% Alfoterra 5PO only yields aqueous emulsion phase.

The optimum condition for this study is to obtain the surfactant system that provides low IFT, but not high solubilization since only oil free phase is desirable from the process. Microemulsion thus may not be the best system for the oil extraction. So, we introduced a single surfactant for oil extraction experiment. However, the result still shows that the mixed surfactant is more efficient for oil extraction. The soybean oil can be extracted more than 85% at soybean seeds size 0.212 - 0.425 mm. In additions, the contact time and soybean load are parameters that influence the efficiency of extraction so we found that the optimum contact time is 30 minutes at soy bean load 1 g.

5.2 Recommendations

Because the microemulsion formation approaches to vegetable oil extraction are new approach and not well understood, the further studies in this topic are necessary. Based on the experimental results, the following recommendations are suggested for future studies.

1. The surfactant systems from this study should be further investigation for varying the temperature in soybean oil extraction process.
2. The properties of free oil phase from the extraction should be studied in more details such as water and surfactant content in this phase
3. The parameter affected to the kinetic of the extraction process should be studied more in details in order to determine the optimum condition in the dimensions of time and energy consideration
4. The properties of emulsion phase as well as soybean meal from the extraction should be further studied

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APPENDICES

Appendix A Experimental Data of Phase Behavior Study.

Table A-1 Phase Behavior Study Results for Different Surfactant System.

No	System of surfactant	Salinity scan	Temperature (°C)	Comercial Soybean Oil	Result
		%NaCl			
1	3%Tergitol 15-S 5	0-20	30	x	Neg
2	4%Tergitol 15-S 5	0-20	30	x	Neg
3	5%Tergitol 15-S 5	0-20	30	x	Neg
4	3%Tergitol 15-S 3	0-20	30	x	Neg
5	4%Tergitol 15-S 3	0-20	30	x	Neg
6	5%Tergitol 15-S 5	0-20	30	x	Neg
7	3%Comperland KD	0-20	30	x	Neg
8	4%Comperland KD	0-20	30	x	Neg
9	5%Comperland KD	0-20	30	x	Neg
10	3%SDS	0-20	30	x	Neg

Table A-1 (continue).

No	System of surfactant	Salinity scan	Temperature (°C)	Comercial Soybean Oil	Result
		%NaCl			
11	4%SDS	0-20	30	x	Neg
12	5%SDS	0-20	30	x	Neg
13	3%SDS+3%Comperland KD	0-20	30	x	Neg
14	3%SDS+4%Comperland KD	0-20	30	x	Neg
15	3%SDS+5%Comperland KD	0-20	30	x	Neg
16	4%SDS+3%Comperland KD	0-20	30	x	Neg
17	4%SDS+4%Comperland KD	0-20	30	x	Neg
18	4%SDS+5%Comperland KD	0-20	30	x	Neg
19	5%SDS+3%Comperland KD	0-20	30	x	Neg
20	5%SDS+4%Comperland KD	0-20	30	x	Neg

Table A-1 (continue).

No	System of surfactant	Salinity scan	Temperature (^oC)	Comercial Soybean Oil	Result
		%NaCl			
21	5%SDS+5%Comperland KD	0-20	30	x	Neg
22	3%SDS+3%Comperland KD	0-20	30	x	Neg
23	3%SDS+4%Comperland KD	0-20	30	x	Neg
24	3%SDS+5%Comperland KD	0-20	30	x	Neg
25	4%SDS+3%Comperland KD	0-20	30	x	Neg
26	4%SDS+4%Comperland KD	0-20	30	x	Neg
27	4%SDS+5%Comperland KD	0-20	30	x	Neg
28	5%SDS+3%Comperland KD	0-20	30	x	Neg
29	5%SDS+4%Comperland KD	0-20	30	x	Neg
30	5%SDS+5%Comperland KD	0-20	30	x	Neg

Table A-1 (continue).

No	System of surfactant	Salinity scan	Temperature (^{o}C)	Comercial Soybean Oil	Result
		%NaCl			
31	3%Comperland KD+0.1%Alfoterra 5PO	0-20	30	x	Middle phase at 5%NaCl
32	3%Comperland KD+0.5%Alfoterra 5PO	0-20	30	x	Middle phase at 15%NaCl
33	3%Comperland KD+1%Alfoterra 5PO	0-20	30	x	Middle phase at 15%NaCl
34	4%Comperland KD+0.1%Alfoterra 5PO	0-20	30	x	Neg
35	4%Comperland KD+0.5%Alfoterra 5PO	0-20	30	x	Middle phase at 15%NaCl
36	4%Comperland KD+1%Alfoterra 5PO	0-20	30	x	Middle phase at 15%NaCl
37	5%Comperland KD+0.1%Alfoterra 5PO	0-20	30	x	Neg
38	5%Comperland KD+0.5%Alfoterra 5PO	0-20	30	x	Neg
39	5%Comperland KD+1%Alfoterra 5PO	0-20	30	x	Middle phase at 5%NaCl
40	3%Spand80+0.1%Alfoterra 5PO	0-20	30	x	Neg

Table A-1 (continue).

No	System of surfactant	Salinity scan	Temperature (^oC)	Comercial Soybean Oil	Result
		%NaCl			
41	3%Spand80+0.5%Alfoterra 5PO	0-20	30	x	Middle phase at 5%,10%NaCl
42	3%Spand80+1%Alfoterra 5PO	0-20	30	x	Middle phase at 5%,10%NaCl
43	4%Spand80+0.1%Alfoterra 5PO	0-20	30	x	Neg
44	4%Spand803+0.5%Alfoterra 5PO	0-20	30	x	Middle phase at 5%,10%NaCl
45	4%Spand80+1%Alfoterra 5PO	0-20	30	x	Middle phase at 5%,10%NaCl
46	5%Spand80+0.1%Alfoterra 5PO	0-20	30	x	Neg
47	5%Spand80+0.5%Alfoterra 5PO	0-20	30	x	Middle phase at 5%NaCl
48	5%Spand80+1%Alfoterra 5PO	0-20	30	x	Middle phase at 5%,10%NaCl
49	3%Tergitol S3+0.1%Alfo	0-20	30	x	Neg
50	3%Tergitol S3+0.5%Alfo	0-20	30	x	Neg

Table A-1 (continue).

No	System of surfactant	Salinity scan	Temperature (^{o}C)	Comercial Soybean Oil	Result
		%NaCl			
51	3%Tergitol S3+1%Alfo	0-20	30	x	Neg
52	4%Tergitol S3+0.1%Alfo	0-20	30	x	Neg
53	4%Tergitol S3+0.5%Alfo	0-20	30	x	Neg
54	4%Tergitol S3+1%Alfo	0-20	30	x	Neg
55	5%Tergitol S3+0.1%Alfo	0-20	30	x	Neg
56	5%Tergitol S3+0.5%Alfo	0-20	30	x	Neg
57	5%Tergitol S5+1%Alfo	0-20	30	x	Neg
58	3%Tergitol S5+0.1%Alfo	0-20	30	x	Neg
59	3%Tergitol S5+0.5%Alfo	0-20	30	x	Neg
60	3%Tergitol S5+1%Alfo	0-20	30	x	Neg

Appendix B Experimental Data of Interfacial Tension Measurement in Batch Study.

Single Surfactant Systems

Table B-1 Interfacial tension of commercial soybean oil with 3% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	AverageIFT(mN/m)	STDEV	%RSD
1	1	0.996	0.986	0.977	0.986	0.0095	0.9636
2	2.5	0.805	0.823	0.846	0.825	0.0206	2.4920
3	5	0.668	0.631	0.653	0.651	0.0186	2.8601
4	7.5	0.466	0.433	0.450	0.450	0.0165	3.6699
5	10	0.698	0.702	0.723	0.708	0.0134	1.8976
6	12.5	1.012	0.989	0.998	1.000	0.0116	1.1594

Table B-2 Interfacial tension of commercial soybean oil with 4% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.889	0.866	0.846	0.867	0.0215	2.4818
2	2.5	0.524	0.555	0.536	0.538	0.0156	2.9036
3	5	0.368	0.321	0.303	0.331	0.0336	10.1495
4	7.5	0.298	0.263	0.276	0.279	0.0177	6.3411
5	10	0.543	0.597	0.556	0.565	0.0282	4.9854
6	12.5	0.801	0.827	0.858	0.829	0.0285	3.4437

Table B-3 Interfacial tension of commercial soybean oil with 5% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.809	0.818	0.836	0.821	0.0137	1.6745
2	2.5	0.504	0.525	0.516	0.515	0.0105	2.0458
3	5	0.368	0.321	0.303	0.331	0.0336	10.1495
4	7.5	0.256	0.233	0.240	0.243	0.0118	4.8518
5	10	0.475	0.487	0.496	0.486	0.0105	2.1678
6	12.5	0.725	0.754	0.736	0.738	0.0146	1.9829

Table B-4 Interfacial tension of soybean oil with 3% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.989	0.976	0.956	0.974	0.0166	1.7073
2	2.5	0.524	0.555	0.536	0.538	0.0156	2.9036
3	5	0.543	0.576	0.533	0.551	0.0225	4.0863
4	7.5	0.348	0.321	0.303	0.324	0.0226	6.9906
5	10	0.725	0.756	0.766	0.749	0.0214	2.8541
6	12.5	0.989	0.996	0.971	0.985	0.0129	1.3089

Table B-5 Interfacial tension of soybean oil with 4% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.888	0.876	0.851	0.872	0.0189	2.1656
2	2.5	0.723	0.755	0.706	0.728	0.0249	3.4175
3	5	0.298	0.276	0.256	0.277	0.0210	7.5932
4	7.5	0.255	0.246	0.257	0.253	0.0059	2.3190
5	10	0.525	0.556	0.506	0.529	0.0252	4.7711
6	12.5	0.701	0.729	0.759	0.730	0.0290	3.9752

Table B-6 Interfacial tension of soybean oil with 5% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.724	0.756	0.784	0.755	0.0300	3.9782
2	2.5	0.474	0.405	0.433	0.437	0.0347	7.9353
3	5	0.233	0.220	0.216	0.223	0.0089	3.9857
4	7.5	0.211	0.206	0.222	0.213	0.0082	3.8429
5	10	0.403	0.376	0.396	0.392	0.0140	3.5775
6	12.5	0.624	0.656	0.639	0.640	0.016	2.503

Mixed Surfactant Systems

Table B-7 Interfacial tension of commercial soybean oil with 0.1% Alfoterra 5PO and 3% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.171	0.175	0.164	0.170	0.00557	3.2752
2	2.5	0.174	0.171	0.184	0.176	0.00681	3.8602
3	5	0.275	0.28	0.27	0.275	0.00500	1.8182
4	7.5	0.107	0.123	0.119	0.116	0.00833	7.1576
5	10	0.160	0.168	0.174	0.167	0.00702	4.1975
6	12.5	0.375	0.351	0.369	0.365	0.01249	3.4219

Table B-8 Interfacial tension of soybean oil with 0.1% Alfoterra 5PO and 3% Comperland KD at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	0.215	0.202	0.208	0.208	0.0065	3.1231
2	2.5	0.297	0.202	0.256	0.252	0.0476	18.9330
3	5	0.31	0.298	0.303	0.304	0.0060	1.9850
4	7.5	0.101	0.109	0.111	0.107	0.0053	4.9453
5	10	0.127	0.150	0.133	0.137	0.0119	8.7295
6	12.5	0.303	0.311	0.329	0.314	0.013	4.236

Table B-9 Interfacial tension of commercial soybean oil with 0.1% Alfoterra 5PO and 4% Span 80 at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	AverageIFT(mN/m)	STDEV	%RSD
1	1	0.876	0.899	0.856	0.877	0.0215	2.4535
2	2.5	0.525	0.503	0.556	0.528	0.0266	5.0430
3	5	0.345	0.373	0.356	0.358	0.0141	3.9404
4	7.5	0.243	0.203	0.196	0.214	0.0254	11.8493
5	10	0.369	0.344	0.372	0.362	0.0154	4.2506
6	12.5	0.725	0.709	0.711	0.715	0.0087	1.2193

Table B-10 Interfacial tension of commercial soybean oil with 0.1% Alfoterra 5PO and 4% Span 80 at different NaCl concentration.

No	%NaCl	IFT(mN/m)	IFT(mN/m)	IFT(mN/m)	Average IFT(mN/m)	STDEV	%RSD
1	1	3.24	2.44	2.56	2.747	0.4314	15.7075
2	2.5	0.424	0.475	0.433	0.444	0.0272	6.1309
3	5	0.368	0.321	0.303	0.331	0.0336	10.1495
4	7.5	0.151	0.173	0.166	0.163	0.0112	6.8815
5	10	0.287	0.290	0.288	0.288	0.0015	0.5298
6	12.5	0.624	0.604	0.611	0.613	0.0101	1.6556

Appendix C Experimental Data of Soybean Oil Extraction.

%Oil Extraction = V measured/ (wt of oil in soy bean seed/d $_{\text{soybean oil}}$)*100

Effected of Grain Sizes on Soybean Oil Extraction

Single surfactant systems

Table C-1 Oil extraction (%) oil with 3% Comperland KD at soybean seed size larger than 35 Mesh (0.425 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	3	1	0.070	0.060	0.080	0.070	0.010	14.286	32.165
2	0	3	2.5	0.080	0.090	0.100	0.090	0.010	11.111	41.355
3	0	3	5	0.110	0.100	0.120	0.110	0.010	9.091	50.545
4	0	3	7.5	0.130	0.140	0.130	0.133	0.006	4.330	61.267
5	0	3	10	0.120	0.120	0.140	0.127	0.012	9.116	58.203
6	0	3	12.5	0.130	0.130	0.130	0.130	0.000	0.000	59.735

Table C-2 Oil extraction (%) oil with 3% Comperland KD at soybean seed size 35 Mesh (0.425 mm)-65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	3	1	0.090	0.090	0.100	0.093	0.006	6.186	42.887
2	0	3	2.5	0.120	0.110	0.100	0.110	0.010	9.091	50.545
3	0	3	5	0.130	0.130	0.120	0.127	0.006	4.558	58.203
4	0	3	7.5	0.150	0.140	0.150	0.147	0.006	3.936	67.393
5	0	3	10	0.160	0.140	0.145	0.148	0.010	7.017	68.159
6	0	3	12.5	0.150	0.140	0.160	0.150	0.010	6.667	68.925

Table C-3 Oil extraction (%) oil with 3% Comperland KD at soybean seed size less than 65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3))	V(cm^3)	V(cm^3))	AverageV(cm3)	STDEV	%RSD	%Extraction
1	0	3	1	0.090	0.100	0.100	0.097	0.006	5.973	44.418
2	0	3	2.5	0.120	0.110	0.110	0.113	0.006	5.094	52.077
3	0	3	5	0.130	0.120	0.120	0.123	0.006	4.681	56.672
4	0	3	7.5	0.150	0.145	0.150	0.148	0.003	1.946	68.159
5	0	3	10	0.160	0.140	0.150	0.150	0.010	6.667	68.925
6	0	3	12.5	0.150	0.140	0.150	0.147	0.006	3.936	67.393

Table C-4 Oil extraction (%) oil with 4% Comperland KD at soybean seed size larger than 35 Mesh (0.425 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	4	1	0.070	0.090	0.080	0.080	0.010	12.500	36.760
2	0	4	2.5	0.090	0.100	0.100	0.097	0.006	5.973	44.418
3	0	4	5	0.120	0.100	0.120	0.113	0.012	10.189	52.077
4	0	4	7.5	0.130	0.140	0.140	0.137	0.006	4.225	62.798
5	0	4	10	0.120	0.130	0.140	0.130	0.010	7.692	59.735
6	0	4	12.5	0.130	0.130	0.130	0.130	0.000	0.000	59.735

Table C-5 Oil extraction (%) oil with 4% Comperland KD at soybean seed size 35 Mesh (0.425 mm)-65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	4	1	0.090	0.100	0.100	0.097	0.006	5.973	44.418
2	0	4	2.5	0.130	0.130	0.120	0.127	0.006	4.558	58.203
3	0	4	5	0.150	0.150	0.130	0.143	0.012	8.056	65.862
4	0	4	7.5	0.150	0.170	0.150	0.157	0.012	7.370	71.988
5	0	4	10	0.160	0.160	0.150	0.157	0.006	3.685	71.988
6	0	4	12.5	0.160	0.170	0.150	0.160	0.010	6.250	73.520

Table C-6 Oil extraction (%) oil with 4% Comperland KD at soybean seed size less than 65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	4	1	0.090	0.100	0.110	0.100	0.010	10.000	45.950
2	0	4	2.5	0.130	0.130	0.130	0.130	0.000	0.000	59.735
3	0	4	5	0.150	0.150	0.140	0.147	0.006	3.936	67.393
4	0	4	7.5	0.150	0.160	0.150	0.153	0.006	3.765	70.457
5	0	4	10	0.160	0.170	0.150	0.160	0.010	6.250	73.520
6	0	4	12.5	0.160	0.160	0.150	0.157	0.006	3.685	71.988

Table C-7 Oil extraction (%) oil with 5% Comperland KD at soybean seed size larger than 35 Mesh (0.425 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	5	1	0.080	0.090	0.080	0.083	0.006	6.928	38.292
2	0	5	2.5	0.100	0.100	0.100	0.100	0.000	0.000	45.950
3	0	5	5	0.120	0.110	0.120	0.117	0.006	4.949	53.608
4	0	5	7.5	0.140	0.140	0.140	0.140	0.000	0.000	64.330
5	0	5	10	0.140	0.130	0.140	0.137	0.006	4.225	62.798
6	0	5	12.5	0.140	0.130	0.150	0.140	0.010	7.143	64.330

Table C-8 Oil extraction (%) oil with 5% Comperland KD at soybean seed size 35 Mesh (0.425 mm)-65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	5	1	0.120	0.100	0.100	0.107	0.012	10.825	49.013
2	0	5	2.5	0.130	0.150	0.130	0.137	0.012	8.449	62.798
3	0	5	5	0.150	0.140	0.160	0.150	0.010	6.667	68.925
4	0	5	7.5	0.150	0.170	0.160	0.160	0.010	6.250	73.520
5	0	5	10	0.160	0.180	0.150	0.163	0.015	9.352	75.052
6	0	5	12.5	0.160	0.170	0.150	0.160	0.010	6.250	73.520

Table C-9 Oil extraction (%) oil with 5% Comperland KD at soybean seed size less than 65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0	5	1	0.120	0.110	0.100	0.110	0.010	9.091	50.545
2	0	5	2.5	0.140	0.140	0.130	0.137	0.006	4.225	62.798
3	0	5	5	0.150	0.160	0.150	0.153	0.006	3.765	70.457
4	0	5	7.5	0.160	0.170	0.160	0.163	0.006	3.535	75.052
5	0	5	10	0.160	0.180	0.170	0.170	0.010	5.882	78.115
6	0	5	12.5	0.160	0.160	0.170	0.163	0.006	3.535	75.052

Mixed surfactant systems

Table C-10 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at soybean seed size larger than 35 Mesh (0.425 mm)

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	1	0.050	0.040	0.060	0.050	0.010	20.000	45.950
2	0.1	3	2.5	0.060	0.060	0.070	0.063	0.006	9.116	58.203
3	0.1	3	5	0.070	0.080	0.080	0.077	0.006	7.531	70.457
4	0.1	3	7.5	0.080	0.090	0.090	0.087	0.006	6.662	79.647
5	0.1	3	10	0.090	0.090	0.075	0.085	0.009	10.189	78.115
6	0.1	3	12.5	0.070	0.090	0.100	0.087	0.015	17.625	79.647

Table C-11 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at soybean seed size 35 Mesh (0.425 mm)-65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	1	0.120	0.150	0.130	0.133	0.015	11.456	61.267
2	0.1	3	2.5	0.140	0.150	0.160	0.150	0.010	6.667	68.925
3	0.1	3	5	0.170	0.160	0.180	0.170	0.010	5.882	78.115
4	0.1	3	7.5	0.190	0.170	0.200	0.187	0.015	8.183	85.773
5	0.1	3	10	0.190	0.180	0.185	0.185	0.005	2.703	85.008
6	0.1	3	12.5	0.180	0.190	0.190	0.187	0.006	3.093	85.773

Table C-12 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at soybean seed size less than 65 Mesh (0.212 mm).

No	wt% Alfoterra 5PO	wt% Comperland KD	%NaCl	V(cm^3)	V(cm^3)	V(cm^3)	AverageV V(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	1	0.140	0.150	0.130	0.140	0.010	7.143	64.330
2	0.1	3	2.5	0.150	0.150	0.160	0.153	0.006	3.765	70.457
3	0.1	3	5	0.170	0.170	0.180	0.173	0.006	3.331	79.647
4	0.1	3	7.5	0.190	0.170	0.190	0.183	0.012	6.298	84.242
5	0.1	3	10	0.190	0.190	0.190	0.190	0.000	0.000	87.305
6	0.1	3	12.5	0.180	0.190	0.185	0.185	0.005	2.703	85.008

Effect of Contact Times on Soybean Oil Extraction

Table C-13 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 1%NaCl concentration (wt %)

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.120	0.130	0.100	0.117	0.015	13.093	53.608
2	0.1	3	20	0.120	0.130	0.120	0.123	0.006	4.681	56.672
3	0.1	3	30	0.120	0.140	0.130	0.130	0.010	7.692	59.735
4	0.1	3	40	0.120	0.150	0.130	0.133	0.015	11.456	61.267
5	0.1	3	50	0.130	0.130	0.135	0.132	0.003	2.192	60.501
6	0.1	3	60	0.130	0.140	0.130	0.133	0.006	4.330	61.267

Table C-14 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 2.5%NaCl concentration (wt %)

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.140	0.120	0.130	0.130	0.010	7.692	59.735
2	0.1	3	20	0.130	0.140	0.150	0.140	0.010	7.143	64.330
3	0.1	3	30	0.140	0.140	0.150	0.143	0.006	4.028	65.862
4	0.1	3	40	0.130	0.160	0.160	0.150	0.017	11.547	68.925
5	0.1	3	50	0.140	0.150	0.155	0.148	0.008	5.149	68.159
6	0.1	3	60	0.140	0.150	0.160	0.150	0.010	6.667	68.925

Table C-15 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 5%NaCl concentration (wt %)

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.150	0.180	0.160	0.163	0.015	9.352	75.052
2	0.1	3	20	0.150	0.170	0.160	0.160	0.010	6.250	73.520
3	0.1	3	30	0.170	0.160	0.180	0.170	0.010	5.882	78.115
4	0.1	3	40	0.170	0.160	0.180	0.170	0.010	5.882	78.115
5	0.1	3	50	0.170	0.160	0.180	0.170	0.010	5.882	78.115
6	0.1	3	60	0.170	0.160	0.180	0.170	0.010	5.882	78.115

Table C-16 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 7.5%NaCl concentration (wt %)

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.155	0.180	0.170	0.168	0.013	7.475	77.349
2	0.1	3	20	0.160	0.180	0.170	0.170	0.010	5.882	78.115
3	0.1	3	30	0.190	0.190	0.180	0.187	0.006	3.093	85.773
4	0.1	3	40	0.190	0.180	0.190	0.187	0.006	3.093	85.773
5	0.1	3	50	0.190	0.180	0.190	0.187	0.006	3.093	85.773
6	0.1	3	60	0.190	0.180	0.190	0.187	0.006	3.093	85.773

Table C-17 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 10%NaCl concentration (wt %)

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.160	0.180	0.170	0.170	0.010	5.882	78.115
2	0.1	3	20	0.160	0.180	0.180	0.173	0.012	6.662	79.647
3	0.1	3	30	0.190	0.190	0.190	0.190	0.000	0.000	87.305
4	0.1	3	40	0.190	0.190	0.180	0.187	0.006	3.093	85.773
5	0.1	3	50	0.190	0.190	0.180	0.187	0.006	3.093	85.773
6	0.1	3	60	0.190	0.190	0.180	0.187	0.006	3.093	85.773

Table C-18 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 12.5%NaCl concentration (wt %).

No	wt% Alfoterra 5PO	wt% Comperland KD	Time	V(cm^3)	V(cm^3)	V(cm^3)	AverageV(cm^3)	STDEV	%RSD	%Extraction
1	0.1	3	10	0.170	0.170	0.175	0.172	0.003	1.682	78.881
2	0.1	3	20	0.170	0.170	0.170	0.170	0.000	0.000	78.115
3	0.1	3	30	0.190	0.180	0.190	0.187	0.006	3.093	85.773
4	0.1	3	40	0.200	0.190	0.170	0.187	0.015	8.183	85.773
5	0.1	3	50	0.200	0.190	0.170	0.187	0.015	8.183	85.773
6	0.1	3	60	0.190	0.190	0.180	0.187	0.006	3.093	85.773

Effected of soybean load on soybean oil extraction

Table C-19 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 7.5%NaCl concentration (wt %).

No	wt% Alfoterra 5PO	wt% Comperland KD	Load (g)	V(cm3)	V(cm3)	V(cm3)	AverageV(cm3)	STDEV	%RSD	%Extraction
1	0.1	3	0.5	0.100	0.090	0.090	0.093	0.006	6.186	85.773
2	0.1	3	1.0	0.190	0.190	0.180	0.187	0.006	3.093	85.773
3	0.1	3	1.5	0.250	0.260	0.240	0.250	0.010	4.000	76.583
4	0.1	3	2.0	0.320	0.330	0.320	0.323	0.006	1.786	74.286
5	0.1	3	2.5	0.380	0.390	0.370	0.380	0.010	2.632	69.844

Table C-20 Oil extraction (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 10%NaCl concentration (wt %).

No	wt% Alfoterra 5PO	wt% Comperland KD	Load	V(cm3)	V(cm3)	V(cm3)	AverageV(cm3)	STDEV	%RSD	%Extraction
1	0.1	3	0.5	0.100	0.100	0.080	0.093	0.012	12.372	85.773
2	0.1	3	1.0	0.190	0.180	0.185	0.185	0.005	2.703	85.008
3	0.1	3	1.5	0.250	0.260	0.260	0.257	0.006	2.249	78.626
4	0.1	3	2.0	0.300	0.320	0.300	0.307	0.012	3.765	70.457
5	0.1	3	2.5	0.360	0.370	0.350	0.360	0.010	2.778	66.168

Table C-21 Oil extr action (%) oil with 3% Comperland KD and 0.1% Alfoterra 5PO at 12.5%NaCl concentration (wt %).

No	wt% Alfoterra 5PO	wt% Comperland KD	Load (g)	V(cm3)	V(cm3)	V(cm3)	AverageV(cm3)	STDEV	%RSD	%Extraction
1	0.1	3	0.5	0.090	0.100	0.090	0.093	0.006	6.186	85.773
2	0.1	3	1.0	0.190	0.180	0.185	0.185	0.005	2.703	85.008
3	0.1	3	1.5	0.250	0.270	0.260	0.260	0.010	3.846	79.647
4	0.1	3	2.0	0.310	0.330	0.300	0.313	0.015	4.875	71.988
5	0.1	3	2.5	0.370	0.350	0.350	0.357	0.012	3.237	65.555

BIOGHAPHY

Mr. Noravee Klongklaew was born on January 6, 1982 in Lopburi, Thailand. He graduated his Bachelor's degree in Chemical Technology from faculty of Science, Chulalongkorn University in 2004. At Chulalongkorn University, he has studied in the topic of "Effect of alkaline activation on properties of activated carbon prepared by antracites" as his senior project.

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