

Liquid – Liquid Hydrocyclone Design for Purification of Palm Biodiesel

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บทคัดย่อ

กระบวนการแยกกลีเซอรอลสำหรับกระบวนการผลิตไบโอคีเซลแบบคั้งเคิมนั้น ้คือ ระบบดีแคนเตอร์ ซึ่งใช้หลักการของแรงโน้มถ่วงในการแยกของเหลวสองชนิดที่มีความ ้หนาแน่นต่างกัน อย่างไรก็ตามพบว่าระบบดีแคนเตอร์ยังมีข้อเสียคือ ถังที่ใช้มีขนาดใหญ่, ใช้ เวลานานในการแยก และทำให้ระบบการผลิตไม่ต่อเนื่อง การศึกษาในครั้งนี้จึงนำเอาไฮโคร ์ ใซโคลนซึ่งเป็นเทคโนโลยีใหม่ในการแยกของเหลวมาใช้แทนระบบคั้งเคิม อีกทั้งยังเป็น เทคโนโลยีที่ติดตั้งง่าย, ค่าใช้จ่ายน้อย และใช้เวลาในการดำเนินงานต่ำ โดยการศึกษาในครั้งนี้จะใช้ ์ โปรแกรมสำเร็จรูป Aspen HYSYS ในการจำลองการกระบวนการ โดยการศึกษาครั้งนี้จะแบ่งเป็น ้สองกิจกรรมคือ การแยกน้ำออกจากน้ำมันในกระบวนการถ้างน้ำมันไบโอคีเซล และกระบวนการ .แยกน้ำมันออกจากลีเซอรอลในกระบวนการแยกกลีเซอรอล โดยตัวแปรที่จะศึกษาประกอบด้วย pressure differential ratio (PDR) และ flow split ratio, การกระจายขนาคของอนุภาค, อัตราส่วน ้ โดยน้ำหนัก, อัตราการไหลทางเข้า, ความดันที่ทางเข้าและอุณหภูมิที่ทางเข้า ผลที่ได้พบว่าสภาวะที่ เหมาะสมในการใช้ระบบไฮโครไซโกลนแยกน้ำจากน้ำมันไบโอดีเซลคือ PDR เท่ากับ 3.8. flow split ratio เท่ากับ 5.36%, ขนาดของอนภาคในช่วง 0.013 – 0.6 มิลลิเมตร อัตราการใหลทางเข้า ในช่วง 1.982.04 – 3.964.08 กิโลกรัมต่อชั่วโมง ความคันของสารป้อนเข้า 380 – 700 kPa และ อุณหภูมิของสารป้อนเข้า 30 - 70 องศาเซลเซียส สำหรับกิจกรรมที่สองจากการศึกษาเพิ่มเติมพบว่า ้กลีเซอรอลมีคุณสมบัติในการละลายน้ำ และการเติมน้ำเข้าไปในขั้นตอนการแยกกลีเซอรอลนั้นทำ ให้การแยกเกิดได้ดีขึ้น การศึกษาในครั้งนี้จึงใช้ระบบไฮโดรไซโคลนแยกกลีเซอรอลจากน้ำมันโดย มีการเติมน้ำร่วมด้วย ผลที่ได้พบว่าสภาวะที่เหมาะสมในการใช้ระบบไฮโครไซโคลนในการแยก ้กลีเซอรอลจากน้ำมันใบโอดีเซลคือ ที่อัตราส่วนของกลีเซอรอล: น้ำมัน: น้ำเท่ากับ 1:10:70, PDR เท่ากับ 3.9, flow split ratio เท่ากับ 5.35%, ขนาดของอนุภาคในช่วง 0.015 – 0.7 มิลลิเมตร อัตรา การใหลทางเข้าในช่วง 2,035.91 – 4,737.40 กิโลกรัมต่อชั่วโมง ความคันของสารป้อนเข้า 400 – 900 kPa และอุณหภูมิของสารป้อนเข้า 30 - 75 องศาเซลเซียส จากการศึกษาเพิ่มเติมยังพบว่าการ ้เพิ่มจำนวนของไฮโครไซโคลนที่ใช้ในระบบจะสามารถเพิ่มประสิทธิภาพในการแยกอีกด้วย

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Abstract

The conventional glycerol separation of biodiesel production process has been used a decanter system which used a set of gravity based vessel to separate glycerol. However, it has been found this process is bulky, requires considerable time consumption and effectuates discontinuous process. The objective of this study is to use a liquid-liquid hydrocyclone for separating glycerol from biodiesel which compact design, easy to install and short operate of time, via using a commercial simulation program such an ASPEN HYSYS. The activities of this study are two parts: using hydrocyclone for separating water in water washing step and for separating glycerol in glycerol separation step. Six variables have been investigated including of pressure differential ratio (PDR), oil droplet size, weight ratio of oil, inlet flow rate, inlet pressure, inlet temperature. The optimal condition for water - biodiesel separation obtained from hydrocyclone system was PDR of 3.8, flow split of 5.36%, oil droplet size range of 0.013 - 0.6mm. The feasible range of the inlet flow rate, inlet pressure and inlet temperature in this case is in range of 1,982.04 - 3,964.08 kg/hr, 380 - 700 kPa and 30 - 70°C respectively. Concerning about second activity is carried out with water added because of the better impact on separation. The optimal condition for glycerol separation obtained from hydrocyclone system was PDR of 3.9, flow split of 5.35%, oil droplet size range of 0.015 - 0.7 mm, which weight ratio as 1:10:70 of glycerol/biodiesel/water. The feasible range of the inlet flow rate, inlet pressure and inlet temperature in this case is in range of 2,035.91 - 4,737.40 kg/hr, 30-75 °C and 400 - 900 kPa respectively. Moreover, the number of hydrocyclone is investigated, this study found that the additional hydrocyclone also improving the separation efficiency.

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CHAPTER 1

INTRODUCTION

1.1 Rational/problem statement

The present moment, energy requirement current are increased while the energy resources are decreased and impact on energy prices is raised. It is necessary to find alternative energy resources to requite the demand for energy. Alternative Energy means energy used for fossil fuel substitution; allocate to 2 categories of theirs original resources; alternative energy from depleted resources such as coal, natural gas nuclear, peat and oil sand etc. and the other alternative energy from non-depleted resources which can be renewable such as solar, wind, biomass, hydro and hydrogen etc. In this article, it will only state about potential and status of alternative diesel energy. Biodiesel has physical properties similar to those of petroleum diesel.

Biodiesel is an alternative diesel energy made from vegetable oil such as palm oil products, waste cooking oil, jatropha, coconut and sunflower etc. As a result of, lower prices, biodegradable, clean burning and lower toxicity compared to petroleum diesel fuel, its performance is the same as diesel fuel from petroleum. A biodiesel process is commonly produced by an acid-transesterification reaction with alcohol and catalyst, yielding biodiesel and glycerol as a by-product. Biodiesel process (Figure 1-1) is include of the following steps:(1) a pretreatment of palm oil, (2) a mixing of alcohol and acid-catalyst, (3) transesterification reaction, (4) a separation of glycerol, (5) purification by washing, and (6) water removal (Tongurai *et al.*, 2006).

Concerning the separation of glycerol, the conventional glycerol separation uses a decanter system which uses a different of specific gravities. After the transesterification reaction, the product is fed into a settling vessel providing two phases of the glycerol and methylester (biodiesel). A glycerol phase is then further drawn off easily from the vessel bottom because it has higher density than another phase. Byproduct as glycerol usually sold to be used in soaps and other products. However, it has been found that this process is a bulky equipment, requires considerable time consumption (about 1-8 hours) (Palm Oil Research Center, Suratthani), and effectuates a discontinuous process.

Conventionally the biodiesel is further purified by gently warm water washing after the glycerol separation. The warm water is filled in a settling vessel tank to remove the remaining byproducts in the biodiesel including of soap, glycerol, catalyst, and unreacted excess methanol with a weight ratio of the water to the biodiesel, 4:1. It is noted that the amount of the wash water depends on the techniques used i.e. agitation washing, mist washing and bubble washing (Gerpen *et.al.*, 1996). After washing and settling, the water can be drained off from the vessel bottom because of its higher density. As a result of a water washing step, a biodiesel production process produces a large amount of highly polluted wastewater. Due to a requirement of long separation time, a conventional water washing causes a discontinuous biodiesel production. Furthermore, it requires a considerable space construction and additional water removal step.

Because a liquid-liquid hydrocyclone (LLHC) is an apparatus that can separate an immiscible liquid-liquid system, spends short operation time, and consequently gives a continuous process. In additional, it is a compact device with no moving part and requires low maintenance. This work then focuses on a using of LLHC for biodiesel purification purposes by considering two purification steps. Application of LLHC in the water washing step will be firstly focused since the assumption of a binary mixture followed by the application in the glycerol separation. Six variables have been investigated including of pressure differential ratio (PDR), oil droplet size, weight ratio of oil, inlet flow rate, inlet pressure, inlet temperature. All investigations have been achieved through a commercial simulation program such an Aspen HYSYS.



Figure 1-1 Schematic of a biodiesel production with hydrocyclone system

1.2 Research objectives

1) To use liquid-liquid hydrocyclone for methyl ester purification via using simulation software program (ASPEN HYSYS).

2) To design liquid-liquid hydrocyclone for separation process.

1.3 Scopes of research work

To achieve the above objectives, the following research scopes have been identified:

1) Separating glycerol from methyl ester after the transesterification reaction process.

2) Reactants for the transesterification reaction are crude palm oil with FFA content less than 0.1 %, methanol and Sodium Hydroxide.

3) Design and Simulate the Hydrocyclone system via using HYSYS.

4) Effect of pressure differential ratio and flow split ratio, inlet flow rate, inlet pressure, inlet temperature, oil droplet size distribution and weight ratios of glycerol to methylester to water on an efficiency of LLHC are investigated.

1.4 Expected benefits

1) The LLHC for separating the glycerol from methyl ester.

2) To decrease the operation time of glycerol separation and bring to increase production capacity.

CHAPTER 2

THEORIES AND LITERATURE REVIEWS

2.1 Theoretical background

2.1.1 Palm Biodiesel Production Process

Biodiesel is an alternative energy diesel fuel form natural. Biodiesel is typically made through a chemical process which converts vegetable oils and fats of natural origin into mono-alkyl esters of long chain fatty acid. The transesterification reaction is a common way to produce biodiesel, triglycerides as a main component of vegetable oils, react with alcohol (ethanol or methanol) and a catalyst (Sodium hydroxide), to produce fatty acid ethyl or methyl ester, and glycerol as a by-product. Biodiesel is intended to be used as a replacement for petroleum diesel fuel, which can be used alone or blended with petroleum diesel fuel in any proportion. Biodiesel can be used in existing diesel engines with little or no modification.

2.1.1.1 Biodiesel feedstock in Thailand

In Thailand has the agriculture of oil yielding crops among the six plant grown, i.e., oil palm, coconut, soybean, peanut, sesame and castor, In accordance with oil crops fact, palm oil has the highest annual yield and followed by coconut and soybean. Other plants have far less production. Another potential energy oil crop for biodiesel production is Jatropha, but it not planted for high consumption. Excepting these oil crops there are other feed stocks in Thailand such as animal fat and waste cooking oil. The appropriate main oilseed crops for biodiesel manufacture in Thailand are currently palm oil and waste cooking oil.

2.1.1.2 Biodiesel production process by transesterification reaction



Figure 2-1 Common Biodiesel Production Process

(1.) Pretreatment of palm oil

A used staple palm oil in palm biodiesel process is prepared properly before entering the reaction step, with the treatment process as giving below.

- Degumming process of palm oil. A step in the refining of oils and fats. The addition of phosphoric acid to the crude oil results in the separation of all or some of phospholipids which present.

- In case of a staple palm oil has the high content of free fatty acid, it is necessary to reduce the content of free fatty acid until less than 1% by weight, with through the esterification reaction.

(2.) Mixing of alcohol and catalyst

Methanol is used as alcohol in the transesterification reaction to produce methyl ester with to combine the separated ester. Methanol is chosen because of its relatively low price. The strong base catalyst to split the oil molecules in reaction is typically sodium hydroxide (NaOH) because of its available and relatively low cost. The specification of methanol used in reaction, must contaminate with water less than 1%. Thence, preparation of an alcohol solution with dissolves 2.5 - 5 parts of sodium hydroxide (NaOH) into 100 parts of methanol in proportion by weight. Besides the amount of sodium hydroxide (NaOH) is depended on the content of free fatty acid in palm oil feedstock.

(3.) Transesterification reaction

The main reaction for converting oil to biodiesel is called transesterification. Pretreatment of reaction, pretreated palm oil which removes the water content already, can go through the transesterification process. Water is removed because its presence causes the triglycerides to hydrolyze, giving salts of the fatty acids (soaps) instead of undergoing transesterification to give biodiesel. After that, palm oil is heated into the temperature about 80 $^{\circ}$ C in a closed reaction vessel, and then the alcohol/catalyst mix is charge slowly (within ten minutes) into the heated palm oil. The system from here on is totally closed to the atmosphere to prevent the loss of alcohol. Sufficient alcohol is added to make up three full equivalents of the triglyceride, and an excess of usually five parts alcohol to one part triglyceride is added to drive the reaction towards the right and ensure complete conversion. The reaction is carried out in vessel by stirring thoroughly for 15 minutes with medium stirring rate (500 rpm), the operating temperature is reduced approximately into 65 $^{\circ}$ C. At this stage, the reaction is proceeding rapidly and producing of methyl ester and glycerol, but the transesterification reaction is reversible therefore the stirring needed to cease. From there, allow the reaction to carry out for 3 – 4 hours without stirring, hereafter oil is reacted to more than 95% As soon as the reaction is complete, the

product mixture is allowed to separate by gravity and the methyl ester is separated from the glycerol.

(4.) Glycerol separation

After the transesterification reaction step is completed, two major products are obtained including glycerol and methyl-ester (biodiesel). The products are fed into a settling vessel providing two phases of the glycerol and the methyl-ester. Since the glycerol phase is much denser than the biodiesel phase, it is simply drawn off from the bottom of the vessel. After the reaction, the glycerol is commonly separated at the reaction temperature (about $60 \forall c$) (Tongurai *et al.*, 2006) because it be solidified at low temperature (its melting point is $19 \forall c$) (Sciencelab.com Inc, 2005). The practical techniques and equipment for separating the glycerol from the biodiesel are as following.

a) Decanter system is a system for separating biodiesel and glycerol, by using the difference of specific gravity. The capacity of separated product is depended on the dormancy time period (about 1-8 hours). For example, 700 gallon decanter can separate biodiesel 5,000,000 gallon/year, with the dormancy time of 1 hour. Howsoever, providing that the reaction is occurring slowly, that mean to the longer dormancy time. The optimal decanter dimension is the ratio L/D (height/diameter) of 1:2, which reach the best performance (Palm Oil Research Center, Suratthani). Furthermore, the temperature within decanter has affect on the solubility of alcohol in biodiesel-glycerol and viscosity of biodiesel and glycerol. Thus, the increase of temperature has an effect on flashpoint. On the other side, the decrease of temperature is producing the higher viscosity, the coalescence take slow down and the emulsion phenomenal of two phases within decanter may take place.

b) Centrifuge system is commonly used to separate biodiesel from glycerol in continuous biodiesel production process. The advantages of centrifuge system are complete separation unit, more expedition and more effectiveness. Nevertheless, the defects of centrifuge technology are high cost of imported centrifuge machinery and need high elaborate maintenance in application.

c) Hydrocyclone system, the principle of hydrocyclone system is used the centrifugal force to separate immiscible continuous liquid. Hydrocyclone is an interesting contemporary technology however, it found some disadvantage that the vapor of biodiesel production may causing the trouble to the mechanism of hydrocyclone separation, owing to the reducing pressure immediately that to induce in the flashpoint of alcohol, for this reason to begin first dispose of methanol is all right well.

(5.) Purification biodiesel

Products of the reaction are included not only biodiesel, but also byproducts and residues i.e., soap, glycerol, remaining catalyst, and unreacted methanol etc. After the glycerol separation, the biodiesel is further purified by gently warm water washing. The amount of water used depends on the techniques used i.e. agitation washing, mist washing and bubble washing (Gerpen, *et.al.*, 1996). The warm water is filled in a settling vessel tank to remove the impurities in the biodiesel, after that the settling water is drained off from the vessel bottom because of its higher density.

(6.) Water removal

After the washing step, the water removal step is then proceeded to achieve ASTM D1796 (maximum water content = 0.05% by vol.). Oil with contaminating water is heated at the temperature of 120° C for 20 minutes, and then the purified oil is cooled down and stored for further use. Other method than heater is filtration by salt filter (Tongurai *et al.*, 2006).

2.1.1.3 Transesterification reaction

The transesterification reaction, the vegetable oil (triglyceride) is reacted in the presence of a strong base catalyst (sodium hydroxide) with an alcohol (usually methanol) to produce the mono alkyl ester (methyl ester) and glycerol.

Biodiesel production process has developed from a one step of transesterification reaction into a two step method of esterification and transesterification reaction. The primary step is esterification reaction to produce primary product and followed by transesterification reaction to from biodiesel production.

(1.) Esterification reaction is the primary process that using the acid catalyst to reduce the free fatty acid content to less than 1% by weight. The esterification reaction of the acid with alcohol is shown in Eq. 2-1



Providing the alcohol is methanol, Eq.2-1 be rewritten in

RCOOH + $CH_{3}OH$ \leftarrow $RCOOCH_{3}$ + $H_{2}O_{2}$ (2-2) acid methanol methyl ester water

(2.) Transesterification reaction is a process using a strong base catalyst to produce biodiesel, which shown in Eq. 2-3, usually provide sodium hydroxide (NaOH) or potassium hydroxide (KOH) as base catalyst. In addition, saponification reaction is occurring be

side reaction, it is a process that produces soap as a production. Saponification reaction is shown in Eq. 2-5.



The transesterification process is the reaction of a triglyceride with a methanol and catalyst to produce methyl ester and glycerol, which shown in Eq. 2-4



Important parameters effect obtained from biodiesel production process are five

parameters.

- The temperature in the reaction, reaction is commonly an endothermic reaction, when the temperature reach higher causing the reaction arise better.

- The weight ratio between oil and alcohol.
- Genre and concentration of catalyst.
- Effect of agitation rate.

2.1.2 Liquid-liquid Hydrocyclone

2.1.2.1 Liquid-Liquid Hydrocyclone principles



Figure 2-2 Hydrocyclone flow behavior.

A liquid-liquid hydrocyclone (LLHC) is utilized centrifugal force to separate glycerol (dispersed phase) from methyl ester (continuous fluid phase). In consequence of immiscible fluid generally have low relative densities that intensive swirling motion are required to separate fine dispersed liquid droplet from continuous liquid phase. The pressurized fluid is injected into the hydrocyclone body in a tangential direction producing the intensive swirling flow within and also to archive high acceleration velocities and pressure drop. The fluid consequently develops a flow pattern consists of inner and outer spiral moving with the same circular direction (Gomez *et al.*, 2001) as shown in Figure 2-2. The inner forced vortex is produced in the region close to LLHC axis delivering the reverse flow of the methyl ester through the overflow outlet. While the water flow moves downward to the underflow outlet resulting of the outer forced vortex appearance in the wall region. The flow movement in hydrocyclone is

complicated. And the two-folded vortex system is essential with the gravity as strong as 2000-3000g, and then some disturbance in the steadiness of the vortex system may detrimental to the separation (Husveg *et al.*, 2007). It has been estimated that particles within the flow field of a 10mm or mini-hydrocyclone experience local accelerations as high as 10,000 gravitation units. (Grady *et al.*, 2003)

The particularity of reverse flow in the liquid-liquid hydrocyclone is explained. The high pressurized fluid is injected and producing the high swirling in the inlet region. The pressure is high close the wall region and decreasing toward the axis region. As a result of that, the pressure gradient profile is occurring across the diameter. While the downstream position decreasing, and the pressure at the downstream end of the core is greater than the upstream, then finally causing the flow reversal. While the fluid moves to the underflow port, the diminishing hydrocyclone cross-sectional area is increasing the angular velocity and the centrifugal force. Because of this force and the differential density between two liquid phases, which the lighter phase migrates to the axis region, where it is caught by the reverse flow and then eventually separated, moving to the overflow port. In contrary, the heaver phase will migrate to the wall region and finally emerging through the underflow port. (Gomez *et al.*, 2001)

Another phenomenon in the hydrocyclone is the occurring of a gas core. An insignificant amount of gas can be tolerated however the excessive amounts will disturb the vortex system. (Gomez *et al.*, 2001)

The movement of droplet under the centrifugal force is estimated by the aggregated forces that acted to the droplet, can be expressed by Eq. 2-6.

$$F_E + F_D + F_B = m \frac{av_d}{dt}$$
(2-6)

in which F_E is The external forced (N) to droplet such as Gravitational force and centrifugal force, F_D is drag force (N), F_B is buoyant force (N) and v_d is droplet velocity (m/s). The maximum velocity of droplet under the centrifugal force without another external force is expressed in Eq. 2-7.

$$\nu_R = \frac{r\omega^2 d_d^2(\rho_d - \rho)}{18\mu} \tag{2-7}$$

in which d_d is diameter of droplet (m), ρ_d is density of droplet (kg/m³), r is position of droplet in radial (m), ω is angular velocity of droplet and μ is viscosity of fluid (kg/m.s).

The maximum velocity of droplet under the gravitational force without another external force is expressed in Eq. 2-8.

$$v_t = \frac{gd_d^2(\rho_d - \rho)}{18\mu} \tag{2-8}$$

The flow inside a hydrocyclone are complex three dimensional velocity flow patterns such as tangential velocity, axial velocity and radial velocity. The tangential velocity is the important velocity component inside a hydrocyclone. The tangential velocity increases from the hydrocyclone wall towards to the center, till reaches the maximum velocity and eventually, rapidly decreases. The axial velocity increases from the hydrocyclone wall towards to the center. (Bai *et al.*, 2009) The radial velocity in the continuous is very small, and has been neglected in many studies. (Gomez *et al.*, 2001) Furthermore, the literature data can be concluded that the tangential and axial velocity mostly affect to the hydrocyclone performance while the radial velocity barely affect. (Srirahong *et al.*, 2009)

2.1.2.2 Liquid-Liquid Hydrocyclone Geometry



Figure 2-3 Hydrocyclone Geometry

The LLHC geometry composes of a set of cylindrical and conical sections and also has the sub four sections as shown in Figure 2-3. The inlet chamber and the reducing section are designed to reach higher tangential acceleration of the fluid, reducing the pressure drop and the shear stress to an acceptable level. The latter section, tapered section is where most of the separation is occurred. The last section, an integrated part of the design is a long tail pipe cylindrical section where the smallest separated droplets, migrate to the reversed flow core at the axis region and eventually being separated flowing into the overflow exit. (Colman and Thew, 1983) Another literature concerned about the hydrocyclone is studied by Schütz. The effect of different cyclone geometries on the separation behavior was investigated. A double-cone cyclone has smoother velocity gradients in its flow field than a standard cylindrical–conical hydrocyclone and also shows lower breakup rates improving the separation behavior. (Schütz *et al.*, 2009).

2.1.2.3 Liquid-Liquid Hydrocyclone separation parameters

The considerable parameters of liquid-liquid hydrocyclone are used to define the total separation efficiency. The following are the important parameters.

(1) Flow Split Ratio

In order to effectuate oil-water separation and maintain the internal flow structure of hydrocyclone, a flow split is exposed (Colman and Thew., 1983). A flow split ratio is defined as the ratio of overflow flow rate to the inlet flow rate, as given in Eq. 2-9.

$$F \mid \frac{q_{overflow}}{q_{inlet}} \Delta 100 \tag{2-9}$$

where, F is the flow split ratio, $q_{overflow}$ is the overflow volumetric flow rate, and q_{inlet} is the inlet volumetric flow rate.

(2) Pressure Differential Ratio (PDR)

Pressure differential ratio (*PDR*) is a fundamental ratio defined as the ratio of overflow to underflow pressure drops as shown in Eq. 2-10.

$$PDR \mid \frac{Inlet \ Pr \ essure \ 4 \ Overflow \ Pr \ essure}{Inlet \ Pr \ essure \ 4 \ Underflow \ Pr \ essure}$$
(2-10)

Hydrocyclone have a characteristic flow rate and pressure drop relationship. (Meldrum, 1988) Pressure differential ratio or pressure drop ratio is defined as dP_o/dP_u . Where dP_o is an inlet to overflow pressure drop when $dP_o = P_i - P_o$, as a function of inlet flow rate for a constant flow split, while an inlet to underflow pressure, $dPu = P_i - P_u$.

(3) Separation Efficiency

.

The separation efficiency of the simulation data is considered with the purity of individual separated streams, calculated by the ratio as given in Eq. 2-11: Many references quantify the relative phase composition of the separated streams in the form of a percentage by volume measurement.

$$\varepsilon \mid \frac{F_o X_{mo}}{F_i X_{mi}} \Delta 100\% \tag{2-11}$$

where F_o is the overflow mass flow rate and F_i is the inlet mass flow rate respectively, while X_{mo} is the mass fraction of methyl ester in overflow stream and C_i are the mass fraction of methyl ester in inlet stream

CHAPTER 3

RESEARCH METHODLOGY

3.1 Biodiesel purification in a water washing step

Conventionally the biodiesel is further purified by gently warm water washing after the glycerol separation using a decanter. The warm water is filled in a settling vessel tank to remove the remaining byproducts in the biodiesel including of soap, glycerol, catalyst, and unreacted excess methanol with a weight ratio of the water to the biodiesel, 4:1. It is noted that the amount of the wash water depends on the techniques used i.e. agitation washing, mist washing and bubble washing (Gerpen, et.al., 1996). After washing and settling, the water can be drained off from the vessel bottom because of its higher density. The washed product is further removed the remaining water by evaporation at the temperature of 120° C for 20 minutes to achieve ASTM D1796 (maximum water content = 0.05% by vol.).

As a result of a water washing step, a biodiesel production process produces a large amount of highly polluted wastewater. Due to a requirement of long separation time, a conventional water washing causes a discontinuous biodiesel production. Furthermore, it requires a considerable space construction and additional water removal step. To overcome these problems, a liquid-liquid hydrocyclone (LLHC) is then investigated in this chapter to separate water from the biodiesel product. It is assumed that a transesterification reaction is complete with no other residues remaining in the products, and insignificant amount of glycerol is considered. By those assumptions, only two components in wastewater are regarded including of the water and the biodiesel.

In this work, the biodiesel is represented by methyl-oleate (MO) which is a major component in the palm biodiesel, 42.93% by weight (Tongurai *et al.*, 2006) and all results have been obtained by using a commercial simulation program such an Aspen HYSYS. This chapter contains of two major parts: (I) single hydrocyclone effect, and (II) double hydrocyclone effect.

3.2 Biodiesel purification in glycerol separation step

In a palm biodiesel production, triglyceride reacts with methanol in the presence of a base catalyst to produce methyl ester (biodiesel) and glycerol (byproduct) as seen in Eq. 2-4. The products are conventionally fed into a decanter providing two phases of the glycerol and the methyl-ester. Since the glycerol phase is much denser than the biodiesel phase, the two can be gravity separated by simply drawing off the glycerol from the bottom of the decanter. However, it has been found that it is a bulky equipment, requires considerable time consumption for separating the glycerol (about 1-8 hours) (Palm Oil Research Center, Suratthani), and effectuates a discontinuous process.

Because a liquid-liquid hydrocyclone (LLHC) is an apparatus that can separate an immiscible liquid-liquid system, spends short operation time, and consequently gives a continuous process. In this chapter, an improvement of biodiesel purification in a glycerol separation step has been investigated by applying the LLHC through a commercial simulation program such an Aspen HYSYS. It is assumed that a transesterification reaction is complete with no other residues remaining in the products, thus a binary mixture of the glycerol and the biodiesel is considered.

In this work, the biodiesel is represented by methyl-oleate (MO) which is a major component in the biodiesel. This chapter contains of two major parts: (I) single hydrocyclone effect, and (II) multiple hydrocyclone effect.



Figure 3-1 Gravity separation of (a) biodiesel-glycerol, and (b) biodiesel-glycerol-water systems

Initially, the separation of glycerol-biodiesel is simulated, and then the result shown that essentially no glycerol removed by the hydrocyclone unit which the separation efficiency is almost 0%. Then study to find the material or substance which is assisted the separation of glycerol. Eventually found that, the addition of water to the glycerol-biodiesel is yielded the positive result, the removal of glycerol began. Then the experiment for separating glycerol-biodiesel with water added is occurred in the lab scale.

Figure 3-1 shows characteristics of the gravity separation in both cases of (a) glycerol-biodiesel, and (b) glycerol-biodiesel-water systems. In case 1, the biodiesel amount of 200 ml has been mixed with 50 ml of glycerol as shown in Table 3-1 with one minute stirring. The glycerol is settling significantly with a remarkable separation time, 14 minutes. Nevertheless the settling time is improved by adding 200 ml of water under the same condition. The aqueous phase is separated from the liquid phase within only 4 minutes. These results are supported by Saleh et al. (2010) who has found that droplet size of glycerol tends to increase with the

additional of water amount. Then the considered biodiesel production in this chapter is shown in Figures 3-2.

	Volume (ml)			Settling time
	Biodiesel	Glycerol	Water	(min)
Case 1:	200	50	_	14
Biodiesel :Glycerol	200	50		17
Case 2:	200	50	200	4
Biodiesel :Glycerol :Water	200	50	200	4

Table 3-1 Settling time in gravity separation of biodiesel-glycerol and biodiesel-glycerol-water



Figure 3-2 The Schematic of biodiesel production process with hydrocyclone system

In this section, a nominal simulation has been carried out with inlet flow rate (F_i) , inlet pressure (P_i) and inlet temperature (T_i) of 3,964.088 kg/hr (4 m³/hr), 500 kPa and 30°C respectively. Six variables have been investigated including of pressure differential ratio (PDR), oil droplet size, weight ratio of oil, inlet flow rate, inlet pressure, inlet temperature.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Biodiesel purification in a water washing step

4.1.1 Part I: Single hydrocyclone effect

In this section, a nominal simulation has been carried out with an inlet flow rate (F_i) and an inlet pressure (P_i) of 3,964.08 kg/hr (4 m³/hr), and 500 kPa respectively. The studied concentration of methyl-oleate (MO) is 5% by weight of feed with feed temperature is 30 $\forall C$. Six variables have been investigated including of pressure differential ratio (PDR), oil droplet size, weight ratio of oil, inlet flow rate, inlet pressure, inlet temperature.

4.1.1.1 Effect of Pressure Differential Ratio (PDR)

In this case, the effect of the pressure differential ratio (PDR) or pressure drop $(\div P)$ ratio has been studied in which PDR is the ratio of overflow pressure drop $(\div P_o)$ to underflow pressure drop $(\div P_u)$ as shown in Eq. 4-1.

$$PDR \mid \frac{dP_o}{dP_u} \circ \frac{\Delta P_o}{\Delta P_u} \tag{4-1}$$

where $\div P_o$ is an inlet to overflow pressure drop, which $\div P_o = P_i - P_o$ $\Rightarrow P_u$ is an inlet to underflow pressure drop, which $\Rightarrow P_u = P_i - P_u$ P_i, P_o and P_u are inlet, overflow and underflow pressure respectively Figure 4-1 demonstrates the effect of PDR on a separation efficiency of the hydrocyclone by varying between 1.6 to 6. It can be seen that the hydrocyclone efficiency primarily increases from 7.5 % to 91.3 % by increasing PDR from 1.6 to 3.8. After that, the efficiency drops dramatically during PDR = 3.8 to 6. For the proposed hydrocyclone a PDR of 3.8 resulted in the maximum efficiency.



Figure 4-1 Effect of Pressure Differential Ratio (PDR) on separation efficiency


Figure 4-2 Pressure drop (÷P) profiles according to PDR variation

Concerning about the efficiency increased as a function of pressure differential ratio, one explanation shown in Figure 4-2. Increasing the pressure differential ratio was directly to decrease the underflow pressure drop continuously while the overflow pressure drop got essentially constant. Another explanation of the effect of pressure differential ratio is increasing of the axial pressure gradient, there corresponding with previous study, increasing PDR means increasing the axial pressure gradient to the overflow (Husveg *et al.*, 2007).

However, the result also shown that, the efficiency gradually decreases after reached the maximum efficiency, this due to the insufficient of underflow pressure drop, and finally causing the disappear of the inner forced vortex.

In this case, the performance of the liquid-liquid hydrocyclone is determined under pressure differential ratio effect on the separation efficiency. As can be seen, the maximum separation efficiency obtained at PDR around 3.8 and a split ratio around 5.36%. The new simulation obtained data is corresponds with the previous literatures review. The flow split data obtained from this simulation correspond with Colman stated that flow split of deoiling hydrocyclone could be operated in range from 0.2% to 10 % (Colman and Thew, 1983). According to this simulation, it is demonstrated how a relationship of PDR and flow split, as described by Figure 4-3. There is an approximate non liner relationship between PDR and flow split (%) (Husveg *et al.*, 2007).



Figure 4-3 Relationship of Pressure Differential Ratio as a function of flow split



Figure 4-4 Oil Concentration profiles according to Pressure Differential Ratio variation

The simulation also shows the purity of overflow stream as given in Figure 4-4, that methyl oleate is reach purified around 99% in all range of Pressure Differential Ratio (PDR). The performance of the hydrocyclone in the test rig was achieving well result.

4.1.1.2 Effect of Droplet Size Distribution

Oil droplet distribution is greatest impact on efficiency separation of LLHC (Gomez *et al.*, 2001). In this case, the system is operated under the inlet pressure and flow rate of 500 kPa and 3,964.08 kg/hr, flow split ratio of 5.36%, and PDR 3.8. The median droplet size range used during the experiments in the test rig is from 0.013 to 0.6 mm. Figure 4-5 shows the separation efficiency of hydrocyclone performance for several droplet size distributions. The simulation result has shown that the efficiency increases by increasing in median droplet size (d50). That can be intuitively expects as larger droplet size coalesce faster than smaller ones. The

proposed LLHC can reach the maximum efficiency, 91.3% with the valid range of the droplet size 0.013 - 0.6 mm.

This can be intuitively expected as the larger oil droplets coalesce faster than the smaller ones. Also, the underflow stream contains smaller droplets sizes, as compared to the inlet stream, due to breakup of droplets in the LLHC. (Gomez *et al.*, 2001)



Figure 4-5 Effect of droplet size distribution on efficiency



Figure 4-6 Effect of weight ratio of oil on efficiency

In this experiment, the performance of the hydrocyclone in the test rig is determined under the weight ratio variation. The purpose of these experiments is how the weight ratios affect the separation efficiency. The inlet pressure and inlet flow rate used during the experiment is 500 kPa and 3,964.08 kg/hr, flow split ratio of 5.36%, and PDR around 3.8. Weight ratios range used during the experiment is around from 4 to 6%. Figure 4-6 shows how efficiency decreased as the weight ratios increasing. This hydrocyclone can reach the higher the maximum efficiency (close to 100%) of weight ratio around 4%. The experimental data is in range of optimal weight ratio in which reviewed from literatures. The performance of the LLHC is best for very low oil concentrations at the inlet, below 1%. For low concentrations, no emulsification of the mixture occurs in the hydrocyclone. However, high inlet concentrations, up to 10%, promote emulsification posing a separation problem in the overflow stream. (Gomez *et al.*, 2001)



Figure 4-7 Effect of inlet flow rate on efficiency

The performance of hydrocyclone affected by the flow rate, lower flow rates mean longer residence times but lower acceleration forces. Conversely higher flow rates result in higher acceleration forces and smaller residence times. (Young *et al.*, 1994) Effect of inlet flow rate is further evaluated on the efficiency of methyl oleate separation. In this experiment hydrocyclone performance was studied as flow rate varation. The propose of of this experiment is to define the optimal operational range of this hydrocyclone. In this simulation is carried out under the inlet pressure kept constant of 500 kPa, flow split ratio of 5.36%, and PDR 3.8 through the experiment. The results are presented in Figure 4-7, it has been found that the feasible range of the inlet flow rate in this case is in range of 1,982.04 - 3,964.08 kg/hr.

As flow increases, the centrifugal forces get stronger and hence increase separation. This increase continues until the flow rate reaches Q_{min} where the efficiency plateaus. The efficiency remains essentially constant until the flow rate reaches Q_{max} . (Husveg *et al.*, 2007)

In this experiment, the inlet flow rate change has slightly effects on the seperation efficiency which is about 91.3%. The characteristic efficiency decrease at flow rates above Q_{max} is explained as a result of either (1) a dramatic increase in droplet break-up due to excessive shear-forces and turbulence, and/or (2) a lack of sufficient pressure gradients to drive the separated oil-core through the overflow as the pressure at the hydrocyclone axis is reduced at high flow rates (Husveg *et al.*, 2007).

Figure 4-8 also shows that the efficiency and product purity insignificantly decreases by increasing the inlet flow rate. The maximum oil purity is 99.99 % under the minimum inlet flow rate, 1,982.04 kg/hr and the minimum purity is 99% under the maximum inlet flow rate, 3,964.08 kg/hr. The overflow as well as underflow flowrates and pressure drops $(\div P)$ increases by increasing the inlet flow rate. The results present that higher flow rate provides higher centrifugal force and $\div P$ but lower efficiency due to lower product purity. It is noted that the results corresponds to ones obtained by Husveg, 2007.



Figure 4-8 Oil purity profile according to variation of inlet flow rate

4.1.1.5 Effect of Inlet Pressure

Similarly with the previous considered variable, the performance of hydrocyclone in the test rig was determined initially under the defining inlet pressure at 500 kPa. In this simulation is carried out under the inlet flow rate kept constant of 3,964.08 kg/hr, flow split ratio of 5.36%, and PDR 3.8 through the experiment. Subsequently, through this test section the performance of hydrocyclone was exposed to several of inlet pressures. The objective of these simulations was to determine, how the inlet pressure affect to separation efficiency with keep constant other variables through the simulation. The result was shown that, inlet pressure with the feasible range of 380 - 700 kPa have barely effects on separation efficiency same as the previous variable in let temperature, and also illustrate a function between inlet pressure and efficiency as liner trend line as shown in Figure 4-9.



Figure 4 - 9 Effect of inlet pressure of efficiency

4.1.1.6 Effect of Inlet Temperature

Initially, the performance of hydrocyclone in the test rig was determined under the defining inlet temperature at 30 °C. Consequently, through this test section the performance of hydrocyclone was exposed to increasing of inlet temperature. In this simulation is carried out under the inlet pressure kept constant of 500 kPa, inlet flow rate 3,964.08 kg/hr flow split ratio of 5.36%, and PDR 3.8 through the experiment. The purpose of these simulations was to determine, how the inlet temperature affect to separation efficiency as a constant of other variables. The result was shown that, the feasible range of the inlet temperature in this case is in range of 30 - 70°C, inlet temperature have barely effects on separation efficiency, and also illustrate a function between inlet temperature and efficiency as liner trend line, can be seen in Figure 4-10.



Figure 4 - 10 Effect of inlet temperature of efficiency

The inlet temperature is also effect to the density of inlet stream as shown in Figure 4-11, increasing of inlet temperature is gradually decreased density.



Figure 4 - 11 The density profile of inlet stream with increasing inlet temperature

4.1.2. Part II: Double hydrocyclone effect

According to the part I, studies the performance and separation efficiency of the hydrocyclone during the variations of important variables, in which of using single hydrocyclone for separation of water – biodiesel. From the previous section propose that, the optimal variables for higher performance in separation is obtained, including flow parameters (inlet flow rate range, flow split), pressure parameter (inlet pressure, PDR), ranges of droplet size distribution and ranges of weight ratios.

In accordance with existent data, this section is study the performance and separation efficiency of the double hydrocyclones in series, with the same dimension of hydrocyclone. The purpose of this study is to increase weight ratio of oil in the process, but keep maintaining the higher separation efficiency. The performance of the double hydrocyclones in the test rig is determined under the condition of weight ratio oil 10%, as demonstrated in Figure 4-12. Another variables used in the experiment is get from the previous section, the inlet flow rate used

is 3,964.08 kg/hr, inlet pressure 500 kPa, PDR and flow split around 3.8 and 5.36% respectively, by kept constant all of the variables throughout the experiment.

The acquired result has intended as the anticipation and the gratifying result. The first hydrocyclone performed at efficiency of 43.9% however, it can reach purity of oil at 99.95%. Following with the second hydrocyclone performed at high efficiency of 82.8%, and present the purity of oil at 99.95%, furthermore the result also shown the purity of water is 99.05%. Moreover the obtained higher efficiency and the increment of oil weight ratio which satisfying, it also the water that reached a higher purity can use to recycle to the next washing time.

The improvement of double hydrocyclone with increasing weight ratio of oil can expand the production capacity, in case of the weight ratio of oil get higher that can produce more capacity of oil as well.



Figure 4-12 Purpose scheme of using hydrocyclone for washing biodiesel

Stream	Mixer	Overflow1	Underflow1	Overflow2	Underflow2
Temperature (°C)	30	30	30.01	30.01	30.02
Pressure (kPa)	500	230.77	429.15	187.24	365.49
Mass Flow (kg/hr)	3,937.44	185.33	3,752.10	175.38	3,576.72

Table 4-1 Detail of each stream from the simulation of double hydrocyclone effect

This research is instead of the conventional washing unit by the hydrocyclone as shown in Figure 4-12. Usefulness of using hydrocyclone for washing biodiesel are combine of many steps of washing unit to a one step (water washing, separation and water removal), that also cause the continuous washing step, the device require short residence time also short operations time compared with traditional gravity separator and in addition that can leading to produce more production capacity as well. Furthermore, the biodiesel purity obtains from hydrocyclone (up to 99.95%) process reach higher than the biodiesel purity of biodiesel standard of Thailand (above 96.5%). (Department of Energy Business, Ministry of energy, Thailand)

4.2 Biodiesel purification in a glycerol separation step

4.2.1 Part I: Single hydrocyclone effect

In this section, a nominal simulation has been carried out with inlet flow rate (F_i) , inlet pressure (P_i) and inlet temperature (T_i) of 3,915.21 kg/hr (4 m³/hr), 500 kPa and 30°C respectively. Six variables have been investigated including of PDR, oil droplet size, weight ratio of oil, inlet flow rate, inlet pressure, inlet temperature.

4.2.1.1 Effect of pressure differential ratio (PDR)

In this case, the simulation was carried out with the inlet flow rate kept constant of 3,915.21 kg/hr, inlet temperature of 30 °C and inlet pressure of 500 kPa as throughput varies PDR. The weight ratio of glycerol to methyl ester to water was defined as 1:10:50 which are the nominal ratio of the considered unit. The pressure differential ratio was adjusted in the feasible range of 1.6 to 6.

Figure 4-13 demonstrated the effect of various pressure differential ratios (PDR) on separation efficiency of hydrocyclone. It can be seen that the efficiency of hydrocyclone was primarily increase from 2.18 % to 28.6 % by increasing pressure differential ratio from 1.6 to 6. The result also shown that, efficiency was drop dramatically after the PDR of 3.9. For the proposed hydrocyclone a PDR of 3.9 resulted in the maximum efficiency.



Figure 4-13 Effect of Pressure differential ratios on oil separation efficiency

Regarding about the efficiency increased as a function of pressure differential ratio, one explanation shown in Figure 4-14. Increasing the pressure differential ratio with keep constant inlet pressure was directly to decrease the underflow pressure drop continuously (increasing of underflow pressure) while the overflow pressure drop got essentially constant. Another explanation of the effect of pressure differential ratio is increasing of the axial pressure gradient, there corresponding with previous study, increasing PDR means increasing the axial pressure gradient to the overflow (Husveg *et al.*, 2007).

However, the result also shown that, the efficiency gradually decreases after reached the maximum efficiency, this due to the insufficient of underflow pressure drop as underflow pressure increasing continuously while inlet pressure keep constant, and finally causing the disappear of the inner forced vortex.



Figure 4-14 (Above) Overflow and Underflow pressure drop profiles (Below) Overflow and Underflow pressure profile according to various PDR, by inlet flow rate kept constant of 3,915.21 kg/hr, inlet temperature 30 °C and inlet pressure of 500 kPa



Figure 4-15 (Above) Overflow and Underflow flow rate profiles (Below) Overflow and Underflow oil flow rate profile according to various PDR, by inlet flow rate kept constant of 3,915.21 kg/hr, inlet temperature 30 °C and inlet pressure of 500 kPa

The simulation also shows in Figure 4-15 that, the overflow stream is riched oil stream by oil purity of overflow stream, while the underflow stream is less content of oil with respect to the underflow flow rate.

In this case, the performance of the liquid-liquid hydrocyclone is determined under pressure differential ratio effect on the separation efficiency. As can be seen, the maximum separation efficiency is 28.60 % with purity of 99.95% oil, obtained at PDR around 3.9 and a split ratio around 5.35%.



Figure 4-16 Density of overflow and underflow stream by increasing pressure differential ratio

The density profile of overflow and underflow stream is shown in Figure 4-16. Through the experiments it is demonstrated how a relationship of PDR and flow split, described by Figure 4-17. The new experimental data obtained is corresponds well with the previous study case of biodiesel – water separation.



Figure 4-17 Relationship of pressure differential ratio as a function of flow split

4.2.1.2 Effect of droplet size distribution

In this case, the system is operated under the inlet pressure and flow rate of 500 kPa and 3,915.21 kg/hr, inlet temperature 30°C flow split ratio of 5.3%, and PDR 3.9 which kept constant through the simulation. The median droplet size range used during the experiments in the test rig is from 0.005 to 0.70 mm. Figure 4-18 shown that the separation efficiency of hydrocyclone performance for several droplet size distributions. The simulation result has shown that the efficiency increases by increasing in median droplet size (d50) until reached the maximum value and essentially constant. The proposed LLHC can reach the maximum efficiency 28.6% with the valid range of the droplet size 0.013 - 0.7 mm.



Figure 4-18 Effect of droplet size distribution on efficiency



Figure 4-19 Overflow and underflow flow rate profile by increasing d50, mean droplet size

According to Figure 4-19, it claimed that efficiency is primarily increased until reached maximum value and essentially constant, this figure is illustrated that overflow rate is also initially increased and reached constant value too.



Figure 4-20 Overflow and underflow pressure profile by increasing d50, mean droplet size



Figure 4-21 The change of density in overflow and underflow stream profile by increasing d50, mean droplet size



Figure 4-22 The oil flow rate in overflow and underflow stream profile by increasing d50, mean

droplet size

4.2.1.3 Effect of weight ratio

In this case, weight ratio of water was investigated along with weight ratio of glycerol, in which biodiesel got constant weight ratio through the simulations. The normally weight ratio of Glycerol: Biodiesel: Water was 1:10:50 as typical of water washing unit.

The main component weight ratios are glycerol and water, which divided weight ratio of glycerol into 1, 5, and 7 respectively, then divided of glycerol weight ratio again with water as 1 - 100 of water weight ratio. The separation efficiency of each weight ratio was observed. In this case, the simulation was carried out with the inlet flow rate kept constant of 3,915.21 kg/hr, inlet temperature 30 °C and inlet pressure of 500 kPa and PDR of 3.9.

	Weight Ratio		Separation Efficiency (%)	Purity (%)	
Glycerol	Biodiesel	Water			
		10	9.13	83.28	
		20	15.01	99.94	
		30	19.57	99.95	
		40	24.12	99.95	
1	10	50	25.45	82.42	
I		60	29.54	82.39	
		70	33.63	82.37	
		80	37.72	82.35	
		90	41.81	82.34	
		100	45.90	82.33	

Table 4-2 Separation efficiency of single effect with Glycerol: Biodiesel (1:10)

Weight Ratio				$\mathbf{D}_{\mathbf{r}}$	
Glycerol	Biodiesel	Water	Separation Efficiency (%)	Fully (76)	
		10	0.00	0.002	
		20	17.20	99.93	
		30	19.46	83.77	
		40	23.53	83.40	
5	10	50	27.60	83.17	
5		60	31.69	83.02	
		70	39.98	99.94	
		78	43.62	99.94	
		92	44.76	82.74	
		100	53.64	99.94	

Table 4-3 Separation efficiency of single effect with Glycerol: Biodiesel (5:10)

Table 4-4 Separation efficiency of single effect with Glycerol: Biodiesel (7:10)

Weight Ratio				$\mathbf{D}_{\mathbf{r}}$	
Glycerol	Biodiesel	Water	Water Separation Efficiency (%)		
		10	0.00	0.00	
		20	18.29	99.93	
		30	20.55	84.33	
		40	24.61	83.83	
7	10	50	28.68	83.53	
/		60	32.76	83.32	
		70	36.84	83.17	
		80	40.92	83.05	
		90	45.01	82.96	
				100	49.10



Figure 4-23 Effect of weight ratio of glycerol and water on efficiency

According to the results, insist that trend of hydrocyclone efficiency get better with increasing glycerol and water weight ratio. Regarding to first considered weight ratio of glycerol to biodiesel of 1:10, and also give consideration to water weight ratio of 1-100. The result in Table 4-2 displayed that, efficiency was improved with increasing water weight ratio, and then efficiency primary was 9.14% as weight ratio of 1:10:10 and reached maximum efficiency of 45.9% as weight ratio of 1:10:100. The latter ratios were weight ratio of glycerol to biodiesel of 5:10, and also give consideration to water weight ratio of 1-100. The result in Table 4-3 maintain that, the efficiency is increased along with increasing weight ratio of glycerol and water. The purposed hydrocyclone give maximum efficiency in weight ratio of 5:10:100 as 43.6%. The last ratios were weight ratio of glycerol to biodiesel of 7:10, and also give consideration to water weight ratio of 1-100. The result in Table 4-4 suggest that, the efficiency raising along with increased weight ratio of glycerol and water until the maximum ratio of glycerol weight ratio, excessive increasing of glycerol weight ratio causing the efficiency drop. The comparison of each ratio is illustrated in Figure 4-23 and Table 4-5 also shown that The details of inlet, overflow and underflow stream in case of weight ratio of glycerol: biodiesel: water as 1: 10: 100, 5:10: 100 and 7:10: 100.

The result data in this study is corresponded with many previous researches, the important reason of the increasing is droplet size distribution. The additional of amounts of water was found to improve the removal of glycerol from biodiesel (Saleh *et al.*, 2010). Concerning to the miscibility of glycerol and water, that improve droplet size of glycerol, a large droplet is affect to the separation and eventually improve efficiency.

Glycerol: Biodiesel: Water	Flow Rate (kg/hr)				
1:10:100	Inlet	Overflow	Underflow		
Glycerol	35.145	0.124	35.022		
Methyl Oleate	361.378	165.904	195.474		
Water	3552.834	35.483	3517.351		
Separation Efficient	ency	45.90	%		
Purity		82.33	%		
Glycerol: Biodiesel: Water	Flow Rate (kg/hr)				
5:10:100	Inlet	Overflow	Underflow		
Glycerol	170.962	0.008	170.954		
Methyl Oleate	351.577	188.597	162.980		
Water	3456.481	0.088	3456.394		
Separation Efficient	ency	53.64%			
Purity		99.94%			
Glycerol: Biodiesel: Water		Flow Rate (kg/hr)			
7:10:100	Inlet	Overflow	Underflow		
Glycerol	236.144	0.811	235.334		
Methyl Oleate	346.874	170.319	176.555		
Water	3410.239	34.339 3375.900			
Separation Efficie	ency	49.10%			
Purity		82.89%			

Table 4-5 The details of inlet, overflow and underflow stream in case of weight ratio of glycerol: biodiesel: water as 1: 10: 100, 5:10: 100 and 7:10: 100.



Figure 4-24 The feasible inlet flow rate plot with separation efficiency

The propose of of this experiment is to define the optimal operational range of this hydrocyclone. In this simulation is carried out under the inlet pressure kept constant of 500 kPa, inlet temperature of 30 °C, flow split ratio of 5.35%, and PDR 3.9 through the experiment. The results are presented in Figure 4-24, it has been found that the feasible range of the inlet flow rate in this case is in range of 2,035.91 – 4,737.40 kg/hr which extended to the increasing and decreasing inlet flow rate, along with separation efficiency. In this experiment, the inlet flow rate change has slightly effects on the seperation efficiency which is about 25-28 % and remained essentially constant. To conclude that, flow rate is less effect on efficiency.



Figure 4-25 The Overflow and Underflow pressure profile with keep constant inlet pressure along with various inlet flow rate increasing

As seen in Figure 4-25 with keep constant of inlet pressure at 500 kPa, overflow and underflow pressure profile is decreased with increasing inlet flow rate. At minimum inlet flow rate at 2,035.91 kg/hr is reached the overflow pressure at 423.15 kPa and underflow pressure at 480.29 kPa, while the maximum inlet flow rate of 4,737.40 kg/hr is raised with the overflow pressure at 103.89 kPa and underflow pressure at 398.43 kPa. Overflow pressure is gradually decreased while underflow pressure is dramatically decreased. It is noticed that, the discrepancy of overflow and underflow pressure at high inlet is larger which can concluded that the higher inlet flow rate causes the larger pressure drop.



Figure 4-26 The Overflow and Underflow flow rate of oil profile with various inlet flow rate

According to Figure 4-26, increasing of inlet flow rate is produced higher overflow flow rate and also presented more oil flow rate.



Figure 4-27 The Overflow and Underflow density profile with various inlet flow rate

4.2.1.5 Effect of inlet Pressure

In this case, the simulation was carried out with the inlet flow rate of 3,915.21 kg/hr, inlet temperature of 30° C flow split ratio of 5.35%, and PDR 3.9 The weight ratio of glycerol to methyl ester to water was defined as 1:10:70 which are the optimum ratio from the previous considerate effect. The inlet pressures are adjusted in the feasible range of 400 - 900 kPa.

Figure 4-28 and Table 4-6 are shown that the separation efficiency of hydrocyclone performance slightly increased along with the increasing of inlet pressure. The proposed LLHC can reach the efficiency around 35.5 % under this condition. It can be conclude that, the inlet pressure is barely effect to the efficiency.



Figure 4-28 Effect of inlet pressure on efficiency

Inlet Pressure (kPa)	Efficiency (%)
400	33.543
450	33.545
500	33.547
550	33.549
600	33.551
650	33.553
700	33.555
750	33.557
800	33.559
850	33.561
900	33.564

Table 4-6 Efficiency of increasing inlet pressure

4.2.1.6 Effect of inlet temperature

In this case, the simulation was carried out with the the inlet pressure and flow rate of 500 kPa and 3,915.21 kg/hr flow split ratio of 5.35%, and PDR 3.9 The weight ratio of glycerol to methyl ester to water was defined as 1:10:70 which are the optimum ratio from the previous considerate effect. The inlet temperatures are adjusted in the feasible range of 30-75 °C.

Figure 4-29 and Table 4-7 are shown that the separation efficiency of hydrocyclone performance increased along with the increasing of temperatures. The proposed LLHC can reach the maximum efficiency 37.59% with the inlet temperature of 70 °C, under this condition.

The temperature is once important effect of fluid properties, the viscosity of water drops as the temperature rises, and this allowed oil droplets to move more easily through

the water phase, thereby producing higher separation performance. Note that for some separators, higher temperatures can reduce separation efficiency. (GHD Pty Limited, 2003)



Figure 4-29 Effect of temperature on efficiency

Table 4-7 Efficiency of increasing inlet temperature

Inlet Temperature (°C)	Efficiency (%)
30	33.54
40	34.32
50	35.01
60	35.65
70	37.59
75	37.58



Figure 4-30The density profile of streams by increasing inlet temperature

According to Figure 4-30, increasing inlet temperature is decreased density of stream, corresponded with Cheng, 2008 which to study the relation of glycerol – water density with temperature in Eq. 3-1. Furthermore, the density profile of glycerol and water is shown in Eq. 3-2 and 3-3 respectively.

$$\rho = \rho_g C_m + \rho_w (1 - C_m) \tag{3-1}$$

$$\rho_g = 1277 - 0.654T \tag{3-2}$$

$$\rho_{\rm w} = 1000 \left(1 - \left| \frac{T-4}{622} \right|^{1.7} \right) \tag{3-3}$$

4.2.2 Part II: Multiple hydrocyclone effect

According to latter section Single hydrocyclone, the separation efficiency is insufficient for the separation process, although the purity of biodiesel reached the standard. In this part the second and third hydrocyclone units have been supplemented, to improve the separation efficiency.

4.2.2.1 Effect of double hydrocyclone

In this case, the simulation was carried out with the the inlet flow rate of $3,915.21 \text{ kg/hr} (4 \text{ m}^3/\text{hr})$, inlet temperature of 30° C flow split ratio of 5.3%, and PDR 3.9 The weight ratio of glycerol to methyl ester to water was defined as 1:10:70 which are the optimum ratio from the previous considerate effect. The inlet pressures are adjusted in the feasible range of 400-900 kPa.

Weight Ratio		First Hydrocyclone		Second hydrocyclone		
Glycerol	Biodiesel	Water	Efficiency (%)	Purity (%)	Efficiency (%)	Purity (%)
		10	9.13	83.28	9.51	83.28
		20	15.01	99.94	16.72	29.17
1 10		30	19.57	99.95	23.03	99.95
		40	24.12	99.95	30.09	99.95
	50	25.45	82.42	32.32	82.42	
		60	29.54	82.39	39.69	82.39
		70	33.63	82.37	47.96	82.37
		80	37.72	82.35	57.33	82.35
		90	41.81	82.34	68.01	82.34
		100	45.90	82.33	89.71	99.95

Table 4-8 Separation efficiency of double effect with Glycerol: Biodiesel (1:10)



Figure 4-31 Effect of weight ratio of glycerol and water on efficiency

Using of two hydrocyclone units, the simulation was carried out with identical weight ratio in the previous section. Consideration to first considered weight ratio of glycerol to biodiesel of 1:10 as shown in Table 4-8, the second hydrocyclone improving separation and also reached maximum efficiency of 89.7% as weight ratio of 1:10:100, while first hydrocyclone only reached maximum efficiency of 45.9%, as shown in Figure 4-31.

Weight Ratio		First Hydrocyclone		Second hydrocyclone		
Glycerol	Biodiesel	Water	Efficiency (%)	Purity (%)	Efficiency (%)	Purity (%)
		10	0.00	0.002	3.27	24.06
		20	17.20	99.93	17.66	84.48
		30	19.46	83.77	22.86	83.77
	10	40	23.53	83.40	29.12	83.40
5		50	27.60	83.17	36.09	83.17
		60	31.69	83.02	43.90	83.02
		70	39.98	99.94	56.60	82.94
		78	43.62	99.94	73.23	99.94
		92	44.76	82.74	85.39	99.94
		100	53.64	99.94	99.84	89.96

Table 4-9 Separation efficiency of double effect with Glycerol: Biodiesel (5:10)



Figure 4-32 Effect of weight ratio of glycerol and water on efficiency

Weight Ratio		First Hydrocyclone		Second hydrocyclone		
Glycerol	Biodiesel	Water	Efficiency (%)	Purity (%)	Efficiency (%)	Purity (%)
		10	0.00	0.00	0.00	0.00
		20	18.29	99.93	21.18	99.93
		30	20.55	84.33	24.48	84.33
		40	24.61	83.83	30.89	83.83
7	10	50	28.68	83.53	38.06	83.53
		60	32.76	83.32	46.11	83.32
		70	36.84	83.17	61.36	99.95
		80	40.92	83.05	72.87	99.94
		90	45.01	82.96	86.10	99.94
		100	49.10	82.89	99.80	98.10

Table 4-10 Separation efficiency of double effect with Glycerol: Biodiesel (7:10)



Figure 4-33 Effect of weight ratio of glycerol and water on efficiency

The latter ratios were weight ratio of glycerol to biodiesel of 5:10. The result in Table 4-9 maintain that, the efficiency is increased along with increasing weight ratio of glycerol and water and also shown that additional hydrocyclone unit can improving efficiency. The purposed second hydrocyclone perfectly reached maximum efficiency in weight ratio of 5:10:100 as 99.8% as illustrated in Figure 4-32, which better than the first hydrocyclone.

The last ratios were weight ratio of glycerol to biodiesel of 7:10. The result in Table 4-10 remaining suggest that, the efficiency raising along with increased weight ratio of glycerol and water until the maximum ratio of glycerol weight ratio, excessive increasing of glycerol weight ratio causing the efficiency drop, as shown in Figure 4-33.

The result data in this part I: second hydrocyclone, is shown that the addition of two hydrocyclone units can improving efficiency as shown in Table 4-11.
Table 4-11 The details of inlet, overflow and underflow stream in case of weight ratio of glycerol: biodiesel: water as 1: 10: 100, 5:10: 100 and 7:10: 100 of

first and second hydrocyclone

Glycerol: Biodiesel: Water	Flow Rate (kg/	hr) of First hyc	rocyclone	Flow Rate (kg/h	ır) of Second hy	drocyclone
1:10:100	Inlet	Overflow	Underflow	Inlet	Overflow	Underflow
Glycerol	35.14	0.12	35.02	35.022	0.002	35.020
Methyl Oleate	361.37	165.90	195.474	195.474	175.367	20.107
Water	3552.83	35.48	3517.35	3517.351	0.082	3517.269
	Separation Efficiency 45.90	%	Purity 82.33 %	Separation Efficiency 89.71	%	Purity 99.95 %
Glycerol: Biodiesel: Water	Flow Rate (kg/	hr) of First hyc	rocyclone	Flow Rate (kg/ł	ır) of Second hy	drocyclone
5:10:100	Inlet	Overflow	Underflow	Inlet	Overflow	Underflow
Glycerol	170.962	0.008	170.954	170.954	0.858	170.096
Methyl Oleate	351.577	188.597	162.980	162.980	162.721	0.259
Water	3456.481	0.088	3456.394	3456.394	17.292	3439.101
	Separation Efficiency 53.64	%	Purity 99.94 %	Separation Efficiency 99.	.84%	Purity 89.96 %
Glycerol: Biodiesel: Water	Flow Rate (kg/	hr) of First hyc	rocyclone	Flow Rate (kg/h	ır) of Second hy	drocyclone
7:10:100	Inlet	Overflow	Underflow	Inlet	Overflow	Underflow
Glycerol	236.144	0.811	235.334	235.334	0.226	235.108
Methyl Oleate	346.874	170.319	176.555	176.555	176.216	0.339
Water	3410.239	34.339	3375.900	3375.900	3.174	3372.726
	Separation Efficiency 49	.10%	Purity 82.89%	Separation Efficiency 99.	80%	Purity 98.10 %

60

4.2.2.2 Effect of triple hydrocyclone

According to latter section Second hydrocyclone, the separation efficiency is insufficient for the separation in some case of weight ratio, although the purity of biodiesel reached the standard. In this part the third hydrocyclone unit was supplemented, to reach the better separation efficiency along with the optimal weight ratio.

In this case, the simulation was carried out with the inlet flow rate of $3,915.21 \text{ kg/hr} (4 \text{ m}^3/\text{hr})$, inlet pressure of 500 kPa, inlet temperature of 30°C flow split ratio of 5.35%, and PDR 3.9.

Weight Ratio			Third Hydrocyclone	Purity (%)
Glycerol	Biodiesel	Water	Separation Efficiency (%)	
		10	9.93	83.28
		20	19.00	99.94
		30	28.31	99.95
		40	36.32	82.47
1	10	50	45.19	82.42
1	10	60	62.28	82.39
		70	97.38	99.95
		80	99.86	67.31
		90	99.86	40.99
		100	99.87	10.64

Table 4-12 Separation efficiency of triple effect with Glycerol: Biodiesel (1:10)



Figure 4-34 Effect of weight ratio of glycerol: biodiesel (1:10) on efficiency

Weight Ratio		Third Hydrocyclone	Purity (%)	
Glycerol	Biodiesel	Water	Separation Efficiency (%)	
		10	5.36544	44.60
		20	20.30121449	84.48
		30	28.05944663	83.77
		40	38.88223488	83.40
5	10	50	53.44490725	83.17
5 10	60	82.29052708	99.94	
	70	99.77665374	70.08	
	80	99.79305244	35.41	
		90	99.80776052	16.11
		100	99.81590219	0.13

Table 4-13 Separation efficiency of triple effect with Glycerol: Biodiesel (5:10)



Figure 4-35 Effect of weight ratio of glycerol and water on efficiency

Weight Ratio			Third Hydrocyclone	Purity (%)
Glycerol	Biodiesel	Water	Separation Efficiency (%)	
		10	2.584039624	18.27
		20	25.4448064	99.93
		30	30.6806923	84.32
		40	42.31643722	83.83
7	10	50	64.4400875	99.94
	10	60	89.73000376	99.94
		70	99.71548108	63.41
		80	99.74393382	36.17
		90	99.76425366	15.23
		100	99.77795635	0.17

Table 4-14 Separation efficiency of triple effect with Glycerol: Biodiesel (7:10)



Figure 4-36 Effect of weight ratio of glycerol and water on efficiency

In this section the performance of third hydrocyclone unit, was displayed the best efficiency. Consideration to first considered weight ratio of glycerol to biodiesel of 1:10 as shown in Table 4-12, the third hydrocyclone improving separation and also reached maximum efficiency of 97.4% as weight ratio of 1:10:70, while first and second hydrocyclone only reached maximum efficiency of 33.6% and 47.9% respectively, as shown in Figure 4-34. Moreover, the maximum efficiency that purposed hydrocyclone reached was 99.8% as weight ratio of 1:10:100.

The latter ratios were weight ratio of glycerol to biodiesel of 5:10. The result in Table 4-13 maintain that, the purposed third hydrocyclone perfectly reached maximum efficiency in weight ratio of 5:10:70 as 99.7% as illustrated in Figure 4-35, while first and second hydrocyclone only reached maximum efficiency of 39.9% and 56.6% respectively. Moreover, the maximum efficiency that purposed hydrocyclone reached was 99.8% as weight ratio of 5:10:100.

The last ratios were weight ratio of glycerol to biodiesel of 7:10. The result in Table 4-14 shown that, the purposed third hydrocyclone reached maximum efficiency of 99.7% as weight ratio of 7:10:100, which less than the second hydrocyclone, as shown in Figure 4-36. Regarding to the result, conclude that weight ratio of glycerol to biodiesel of 7:10 is only appropriated with two hydrocyclone.

The result data in this part II: third hydrocyclone is shown that the addition of three hydrocyclone units can improve efficiency.

Table 4-15 The details of inlet, overflow and underflow stream in case of weight ratio of glycerol: biodiesel: water as 1: 10: 100, 5:10: 100 and 7:10: 100 of

first, second and third hydrocyclone

Flow Rate (kg/hr) of First hydrocyclone Flow Rate (kg/hr) of S	r) of First hydrocyclone Flow Rate (kg/hr) of S	rocyclone Flow Rate (kg/hr) of S	Flow Rate (kg/hr) of So	/hr) of S	econd	hydrocyclone	Flow Rate (kg	t/hr) of Third h	ydrocyclone
Inlet Overflow Underflow Inlet	Overflow Underflow Inlet	Underflow Inlet	Inlet		Overflow	Underflow	Inlet	Overflow	Underflow
35.145 0.124 35.022 35.022	0.124 35.022 35.022	35.022 35.022	35.022		0.002	35.020	35.020	1.663	33.358
361.378 165.904 195.474 195.474	165.904 195.474 195.474	195.474 195.474	195.474		175.367	20.107	20.107	20.081	0.026
3552.834 35.483 3517.351 3517.351	35.483 3517.351 3517.351	3517.351 3517.351	3517.351		0.082	3517.269	3517.269	166.976	3350.293
Efficiency 45.90% Purity 82.33 % Efficiency 89	0% Purity 82.33 % Efficiency 89	Purity 82.33 % Efficiency 89	Efficiency 89	<u> </u>	71%	Purity 99.95 %	Efficiency 99.	.87% Pui	ity 10.64 %
Flow Rate (kg/hr) of First hydrocyclone	r) of First hydrocyclone	rocyclone Flow Rate (k	Flow Rate (k	60	/hr) of Second	hydrocyclone	Flow Rate (kg	t/hr) of Third h	ydrocyclone
Inlet Overflow Underflow Inlet	Overflow Inlet	Underflow Inlet	Inlet		Overflow	Underflow	Inlet	Overflow	Underflow
170.962 0.008 170.954 170.954	0.008 170.954 170.954	170.954 170.954	170.954		0.858	170.096	170.096	9.113	160.983
351.577 188.597 162.980 162.980	188.597 162.980 162.980	162.980 162.980	162.980		162.721	0.259	0.259	0.259	0.000
3456.481 0.088 3456.394 3456.394	0.088 3456.394 3456.394	3456.394 3456.394	3456.394		17.292	3439.101	3439.101	184.256	3254.846
Efficiency 53.64% Purity 99.94 % Efficiency 99	Purity 99.94 % Efficiency 99	Purity 99.94 % Efficiency 99	Efficiency 99	<u> </u>	84%	Purity 89.96 %	Efficiency 99.	.81% Pu	ity 0.13 %
Flow Rate (kg/hr) of First hydrocyclone Flow Rate (k	r) of First hydrocyclone	rocyclone Flow Rate (k	Flow Rate (k	60	/hr) of Second	hydrocyclone	Flow Rate (kg	t/hr) of Third h	ydrocyclone
inlet Overflow Underflow Inlet	Overflow Inlet	Underflow Inlet	Inlet		Overflow	Underflow	Inlet	Overflow	Underflow
236.144 0.811 235.334 235.334	0.811 235.334 235.34	235.334 235.334	235.334		0.226	235.108	235.108	12.591	222.517
346.874 170.319 176.555 176.555	170.319 176.555 176.555	176.555 176.555	176.555		176.216	0.339	0.339	0.338	0.001
3410.239 34.339 3375.900 3375.900	34.339 3375.900 3375.900	3375.900 3375.900	3375.900		3.174	3372.726	3372.726	180.620	3192.105
Efficiency 49.10% Purity 99.94 % Efficiency 9	1% Purity 99.94 % Efficiency 9	Purity 99.94 % Efficiency 9	Efficiency 9	9.	80%	Purity 89.96 %	Efficiency 99.	T7% Pu	ity 89.96 %

The best conditions obtained from part II: third hydrocyclone is weight ratio of glycerol: biodiesel: water as 1:10:70, is illustrated in Figure 4-37. The details of each stream such as inlet, overflow and underflow stream is given in Table 4-16.

Table 4-16 The details of inlet, overflow and underflow stream in case of weight ratio of glycerol: biodiesel: water as 1: 10: 70 of first, second and third hydrocyclone

		Overflow stream	
	First hydrocyclone	Second hydrocyclone	Third hydrocyclone
Capacity (kg/hr)	201.3271671	190.5268955	165.8566694
Purity (%)	82.37	82.37	99.95



Figure 4-37 The schematic of streams in case of weight ratio of glycerol: biodiesel: water as 1: 10: 70 of first, second and third hydrocyclone

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APPENDIX A

MATERIAL SAFETY DATA SHEET (MSDS) OF GLYCERIN

The MSDS data of glycerin are as follows:

Section 1) Chemical Product and Company Identification

Product name: Glycerin

CAS number: 56-81-5

RTECS: MA8050000

TSCA: TSCA 8(b) inventory: Glycerin

CI#: Not available.

Synonym: 1, 2, 3-Propanetriol; Glycerol

Chemical name: Glycerin

Chemical Formula: C_3H_5 (OH) $_3$

Section 2) Composition and Information on Ingredients

Product name: Glycerin

CAS number: 56-81-5

100% by weight

Section 3) Hazards Identification

Potential Acute Health Effects: Slightly hazardous in case of skin contact

(irritant, permeator), of eye contact (irritant), of ingestions, of inhalation.

Potential Chronic Health Effects:

CARCINOGENIC EFFECTS: Not available.

MUTAGENIC EFFECTS: Not available.

TERATOGENIC EFFECTS: Not available.

DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to

kidneys. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4) First Aid Measures

Eye Contact: Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention if irritation occurs.

Skin Contact: Wash with soap and water. Cover the irritated skin with an emollient. Get medical attention if irritation develops. Cold water may be used.

Serious Skin Contact: Not available.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation: Not available.

Ingestion: Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention if symptoms appear.

Serious Ingestion: Not available.

Section 5) Fire and Explosion Data

Flammability: May be combustible at high temperature.

Auto-Ignition Temperature: 370°C (698°F)

Flash Points: CLOSED CUP: 160°C (320°F).

OPEN CUP: 177°C (350.6°F)

Flammable Limits: LOWER: 0.9%

Products of Combustion: These products are carbon oxides (CO, CO2), irritating and toxic fumes.

Fire Hazards in Presence of Various Substances: Slightly flammable to flammable in presence of open flames and sparks, of heat, of oxidizing materials. Non-flammable in presence of shocks.

Explosion Hazards in Presence of Various Substances: Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available. Explosive in presence of oxidizing materials.

Fire Fighting Media and Instructions: SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Glycerin is incompatible with strong oxidizers such as chromium trioxide, potassium chlorate, or potassium permanganate and may explode on contact with these compounds. Explosive glyceryl nitrate is formed from a mixture of glycerin and nitric and sulfuric acids. Perchloric acid, lead oxide + glycerin form perchloric esters which may be explosive. Glycerin and chlorine may explode if heated and confined.

Section 6) Accidental Release Measures

Small Spill: Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. Finish cleaning by spreading water on the contaminated surface and dispose of according to local and regional authority requirements.

Large Spill: Stop leak if without risk. If the product is in its solid form: Use a shovel to put the material into a convenient waste disposal container. If the product is in its liquid form: Do not get water inside container. Absorb with an inert material and put the spilled material in an appropriate waste disposal. Do not touch spilled material. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Eliminate all ignition sources. Call for assistance on disposal. Finish cleaning by spreading water on the contaminated surface and allow to evacuate through the sanitary system. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities. Section 7) Handling and Storage

Precautions: Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area. Hygroscopic

Section 8) Exposure Controls/Personal Protection

Engineering Controls: Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location. Personal Protection: Safety glasses. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill: Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits: TWA: 10 (mg/m3) from ACGIH (TLV) [United States] [1999] Inhalation Total. TWA: 15 (mg/m3) from OSHA (PEL) [United States] Inhalation Total. TWA: 10 STEL: 20 (mg/m3) [Canada] TWA: 5 (mg/m3) from OSHA (PEL) [United States] Inhalation Respirable. Consult local authorities for acceptable exposure limits.

Section 9) Physical and Chemical Properties

Physical state and appearance: Liquid. (Viscous (Syrupy) liquid)
Odor: Mild
Taste: Sweet.
Molecular Weight: 92.09 g/mole
Color: Clear Colorless.
pH (1% soln/water): Not available.
Boiling Point: 290°C (554°F)
Melting Point: 19°C (66.2°F)
Critical Temperature: Not available.
Specific Gravity: 1.2636 (Water = 1)
Vapor Pressure: 0 kPa (@ 20°C)
Vapor Density: 3.17 (Air = 1)
Volatility: Not available.
Odor Threshold: Not available.
Water/Oil Dist. Coefficient: The product is more soluble in water; log (oil/water)

= -1.8

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, acetone.

Solubility: Miscible in cold water, hot water and alcohol. Partially soluble in

acetone. Very slightly soluble in diethyl ether (ethyl ether). Limited solubility in ethyl acetate.

Insoluble in carbon tetrachloride, benzene, chloroform, petroleum ethers, and oils

Section 10) Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Avoid contact with incompatible materials, excess heat and ignition, sources, moisture.

Incompatibility with various substances: Highly reactive with oxidizing agents.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:

Hygroscopic. Glycerin is incompatible with strong oxidizers such as chromium trioxide, potassium chlorate, or potassium

permanganate. Glycerin may react violently with acetic anhydride, aniline and

nitrobenzene, chromic oxide, lead oxide and

fluorine, phosphorous triiodide, ethylene oxide and heat, silver perchlorate, sodium peroxide, sodium hydride.

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

Section 11) Toxicological Information

Routes of Entry: Absorbed through skin. Eye contact.

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Toxicity to Animals: WARNING: THE LC50 VALUES HEREUNDER ARE
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ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 4090

mg/kg [Mouse]. Acute dermal toxicity (LD50): 10000 mg/kg [Rabbit]. Acute toxicity of the mist

(LC50): >570 mg/m3 1 hours [Rat].

Chronic Effects on Humans: May cause damage to the following organs:

kidneys.

Other Toxic Effects on Humans: Slightly hazardous in case of skin contact (irritant), of ingestion, of inhalation.

Special Remarks on Toxicity to Animals: TDL (rat) - Route: Oral; Dose: 100 mg/kg 1 day prior to mating. TDL (human) - Route: Oral; Dose: 1428 mg/kg

Special Remarks on Chronic Effects on Humans: Glycerin is transferred across the plancenta in small amounts. May cause adverse reproductive effects based on animal data (Paternal Effects (Rat): Spermatogenesis (including genetic material, sperm morphology, motility, and count), Testes, epididymis, sperm duct). May affect genetic material. S

pecial Remarks on other Toxic Effects on Humans: Acute Potential Health Effects: Low hazard for normal industrial handling or normal workplace conditions. Skin: May cause skin irritation. May be absorbed through skin Eyes: May cause eye irritation with stinging, redness, burning sensation, and tearing, but no eye injury. Ingestion: Low hazard. Low toxicity except with very large doses. When large doses are ingested, it can cause gastrointestinal tract irritation with thirst (dehydration), nausea or vomiting diarrhea. It may also affect behavior/central nervous system/nervous system (central nervous system depression, general anesthetic, headache, dizziness, confusion, insomnia, toxic psychosis, muscle weakness, paralysisconvulsions), urinary system/kidneys(renal failure, hemoglobinuria), cardiovascular system (cardiac arrhythmias), liver. It may also cause elevated blood sugar. Inhalation: Due to low vapor pressure, inhalation of the vapors at room temperature is unlikely. Inhalation of mist may cause respiratory tract irritation. Chronic Potential Health Effects: Ingestion: Prolonged or repeated ingestion may affect the blood(hemolysis, changes in white blood cell count), endocrine system (changes in adrenal weight), respiratory system, and may cause kidney injury.

Section 12) Ecological Information

Ecotoxicity: Ecotoxicity in water (LC50): 58.5 ppm 96 hours [Trout].

BOD5 and COD: Not available.

Products of Biodegradation: Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise. Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13) Disposal Considerations

Waste Disposal: Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14) Transport Information

DOT Classification: Not a DOT controlled material (United States).

Identification: Not applicable.

Special Provisions for Transport: Not applicable.

Section 15) Other Regulatory Information

Federal and State Regulations: Illinois toxic substances disclosure to employee act: Glycerin Rhode Island RTK hazardous substances: Glycerin Pennsylvania RTK: Glycerin Minnesota: Glycerin Massachusetts RTK: Glycerin Tennessee - Hazardous Right to Know: Glycerin TSCA 8(b) inventory: Glycerin

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

> Other Classifications: WHMIS (Canada): Not controlled under WHMIS (Canada). DSCL (EEC): Not available S24/25- Avoid contact with skin and eyes. HMIS (U.S.A.): Health Hazard: 1 Fire Hazard: 1 Reactivity: 0 Personal Protection: g National Fire Protection Association (U.S.A.): Health: 1 Flammability: 1 Reactivity: 0 Specific hazard:

Protective Equipment: Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Safety glasses.

Section 16) Other Information

References: Not available.

Other Special Considerations: Not available.

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall ScienceLab.com be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if ScienceLab.com has been advised of the possibility of such damages.

APPENDIX B

1. HYSYS PROCESS SIMULATION

HYSYS was selected as a process simulator for both its simulation capabilities and its ability to incorporate calculations using the Microsoft Excel. The first steps in developing the process simulation were selecting the chemical components for the process, as well as a thermodynamic model. The unit operation for the base cases is liquid-liquid hydrocyclone separator.

HYSYS library contained information for the following components used in the simulation: palm biodiesel and water. Palm biodiesel was represented by methyl oleate. Accordingly, methyl-oleate, available in the HYSYS component library, was taken as the product of the transesterification reaction. In consequence of the properties of methyl oleate have almost same properties as palm biodiesel.

Thermodynamic analysis of models of liquid solutions is carried out on the basis of the concentration dependences of excess functions. Using the energy balance plane, the applicability region of is determined and constraints on the equation parameters are imposed.

The nonrandom two-liquid (NRTL) was selected for use as the property package for the simulation which used to calculate the equilibrium compositions in two-phase and multiphase systems. However some binary interaction parameters were not available in the simulation databanks, they were estimated using the UNIFAC liquid–liquid equilibrium models for calculation a set of binary coefficient parameters, along with NRTL model.

Simulate software program has assisted the design process for a chemical engineering process. The simulation can provide the process to closer to the reality operating condition, because there is data in the Library's program both physical and thermodynamic of

substance. Simulate software program is well-known and widely used commonly in industry. Furthermore, the considerable specific substance which without the databank in library, where the user can enter basic information of substance such as structure formula, density data, boiling point and other data into the program. In order that, to render the program calculate physical and thermodynamic of specific substance, for apply to process designing. Usability of program, the user assigns requisite unit and some requiring parameters or coefficients in the simulation from there program solver will calculate the other needed parameters.

HYSYS (HYprotech SYStem) ASPEN Tech. Plant Simulation Software V 7.1 (ASPEN Tech, Burlington MA) is used to conduct the simulation and designing. Aspen HYSYS is a market-leading process modeling tool for conceptual design, optimization, business planning, asset management, and performnce monitoring for any application. There are many features of Aspen HYSYS as followed.

2. Liquid-Liquid Hydrocyclone in HYSYS

The HYSYS Liquid-liquid Hydrocyclone generates the results based on the migration probability theory. Besides, the oil droplet size distribution based on sauter mean diameter is applied.

The hydrocyclone tab is designed to be easy to use with a single input tab giving liner details and the oil droplet size distribution. Moreover, the process details, hydrocyclone liner dimensionless parameters and separation performance are calculated. The option of modeling three different types of liner:

- Vortoil G-Liners
- Serck Baker Oilspin liners
- Custom user liners

The fundamental calculation methods are similar for all liners.

2.1 Oil Droplet Distribution

The HYSYS hydrocyclone uses a Rosin Rammler Oil Droplet Distribution to describe the dispersion at the inlet. Two parameters cumulative distributions are defined. The cumulative distribution is defined by the following Eq. B-1.

$$F(d) = 1 - exp\left(-\left(\frac{d}{d_{rm}}\right)^n\right)$$
(B-1)

where F(d) is the cumulative distribution, d is the droplet diameter, d_{rm} is the Rosin Rammler modal diameter and n is the exponential power index.

The Rosin Rammler modal diameter $d_{\rm rm}$ can be related to another mean diameter $d_{\rm m}$ by the following Eq. B-2.

$$d_{\rm M} = d_{\rm rm} \times \left[-\ln(1 - f(d)) \right]^{1/n}$$
 (B-2)

where f(d) is the fraction undersize at diameter d_M

2.2 HYSYS hydrocyclone liner dimensions



Figure B-1 Hydrocyclone liner dimensions

The hydrocyclone dimensions are based on the following variables as shown in Figure B-1.

- D, Characteristic diameter is defined by the user.
- D_{IN} , Inlet diameter is set at 0.35D.
- D_U, Underflow diameter is set at 0.5D.
- D_0 , Overflow diameter is defined by the user.
- The taper angles θ_1 and θ_2 define the separation section geometry.
- L, length of each section is calculated from the taper angles (θ_1 and θ_2) and the characteristic diameter (D).

- The length from the end of taper section to the liner tip taken as 20D. All of these lengths are then summed to give the total liner length of hydrocyclone.

2.3 Hydrocyclone Hydraulics

The HYSYS hydrocyclone can be modeled hydraulically in a dimensionless manner assuming geometrically similar the criteria. A Reynolds number and hydrocyclone number can be defines using dimensions, fluid parameters and operating conditions. Split ratio and maximum flow rate are also determined from the operating data.

Reynolds studied the conditions under which one type of flow changes to the other found that the critical velocity, at which laminar flow change to turbulent flow, depends on the diameter of tube, velocity, density and viscosity of the liquid. (Warren *et al.*, 2005)

Additional observations have shown that the transition from laminar to turbulent flow actually may occur over a wide range of Reynolds numbers. In a pipe, flow is always laminar at Reynolds numbers below 2,100, but laminar flow can persist in smooth tubes up to Reynolds numbers well above 2,100 by eliminating all disturbances at the inlet. If the laminar flow at such high Reynolds numbers is disturbed, however say by a fluctuation in velocity, the flow quickly becomes turbulent. Disturbances under these conditions are amplified, whereas at Reynolds numbers below 2,100 all disturbances are damped and the flow remains laminar. At some flow rates a disturbance may be neither damped nor amplified; the flow is then said to be neutrally stable. Under ordinary conditions, the flow in a pipe or tube is turbulent at Reynolds numbers above about 4,000. Between 2,100 and 4,000 a transition region is found may the flow are be either laminar or turbulent, depending upon conditions at the entrance of the tube and on the distance from the entrance. (Warren *et al.*, 2005) Reynolds number Re_D is expressed as Eq. B-3.

$$Re_D = \frac{Q_T \times \rho_c}{900 \times \pi \times D_H \times \mu_c} \tag{B-3}$$

where Q_T is the volumetric flow rate, ρ_c is the density of continuous phase, D_H is the hydrocyclone characteristic diameter and μ_c is the viscosity of continuous phase.

Hydrocyclone number, Hy_{75} is related to an oil droplet diameter, d'_{75} may be defined as Eq. B-4.

$$Hy_{75} = \frac{Q_T \times \Delta \rho \times d{r_{75}}^2}{3600 \times D_H^3 \times \mu_c}$$
(B-4)

where Q_T is the volumetric flow rate, Δ_{ρ} is an oil and water density difference, d'_{75} is the 75% migration probability droplet diameter, D_H is the hydrocyclone characteristic diameter and μ_c is viscosity of the continuous phase.

The hydrocyclone number can also be related to the Reynolds number for similar geometric units by means of the following general equation, Eq. B-5.

$$Hy_{75} = a(Re_D)^b \tag{B-5}$$

Experiment or production performance data can be used to establish the values of a and b. Theses constants are liner specific.

Split ratio is calculated from the user defined pressure difference ratio (PDR) by means of a quadratic expression as shown in Eq. B-6.

$$F = \propto (PDR)^2 + \beta (PDR) + \gamma \tag{B-6}$$

where \propto , β and γ are the parameter values established from a curve fit to operating data.

Maximum flow rate, Q_{MAX} for the system is related to the pressure differential between the inlet and reject streams, which expressed in Eq. B-7.

$$Q_{MAX} = n_L \times k \left(P_{IN} - P_{REI} \right)^n \tag{B-7}$$

where n_L is the number of liners, P_{IN} is the inlet pressure, P_{REJ} is the overflow pressure and k, n are the constant values established from hydraulic data.

2.4 Oil Droplet migration probability

The method of dense dispersion hydrocyclone is applied to predict the volume of oil separated from the inlet stream. A migration probability for the droplet distribution is derived from statistical theory and a reduced migration probability.

Migration probability, for a given inlet oil droplet distribution the migration probability (MP) of a droplet of diameter d microns is defined as the chance that it will be separated in the oil overhead stream. The migration probability can be related to the reduced migration probability (RMP) and the split ratio (F) by following the Eq. B-8.

$$MP(d) = RMP(d) \times (1 - F) + F \tag{B-8}$$

Reduced migration probabilities, analytical function may be fitted to represent the center of an envelope of experimental curves for a particular liner. This reduced migration probability (RMP) can be represented generally in terms of a normalized droplet diameter Δ_{75} as shown in Eq. B-9.

$$RMP = 1 - exp(a[\Delta_{75} - b]^c)$$
 (B-9)

where *a*, *b*, *c* are the constants determined by experiment, Δ_{75} is $\frac{d}{d_{75}}$ which be the dimensionless droplet diameter, d'_{75} is determined from the hydrocyclone number and d is the droplet diameter from the distribution.

The migration probability for an oil droplet distribution is represented graphically as shown in Figure B-2.





Figure B-2 The oil droplet migration probability

3 Liquid-liquid hydrocyclone operation approach

There are two methods to add a liquid-liquid hydrocyclone to the simulation.

The first method is usually by the following.

1. From the Flow sheet menu, click Add Operation. The Unit Operations view

appears.

- 2. Click the Upstream Ops radio button.
- 3. From the list of available unit operations, select liquid-liquid hydrocyclone.
- 4. Click the Add button.

The second method is usually by the following.

1. From the Flow sheet menu, select Palette (or press F4). The Object Palette

appears.

2. In the Object Palette, click the liquid-liquid hydrocyclone icon, as shown in

Figure B-3.





Liquid-liquid Hydrocyclone Icon

Figure B-3 The objective palette and liquid-liquid hydrocyclone icon

3.1 The liquid-liquid hydrocyclone property view



Figure B-4 The property view page for operation design tab

To delete the liquid-liquid hydrocyclone operation, click the delete button. HYSYS will ask the user to confirm the deletion. Besides, also delete a liquid-liquid hydrocyclone by clicking on the liquid-liquid hydrocyclone icon on the PFD and pressing DELETE. To ignore the liquid-liquid hydrocyclone during calculations, select the Ignored checkbox. HYSYS completely disregards the operation (and cannot calculate the outlet stream) until the user restore it to an active state by clearing the checkbox.

3.1.1 Design tab

The Design tab consists of the following pages:

- a) Connections
- b) Parameters
- c) Liner Details
- d) Droplet Distribution
- e) User Variables
- f) Notes

a) Connections page

The Connections page is used to define all of the connections to the liquid-liquid hydrocyclone. User can specify the inlet stream, overflow outlet stream, and underflow outlet stream attached to the operation. The name of the operation can be changed in the Name field as shown in Figure B-5.

Design	Name LHC-1	<u>O</u> verflow
Connections		Overflow1
Parameters	Inlet	
Liner Details Droplet Distribution	Mixer	121
User Variables	i de la constante de	
Notes		
	Fluid <u>P</u> ackage	
	Basis-1	Underflow Underflow1
		<u>لا ، ہ</u>

Figure B-5 The connection page for operation design tab

b) Parameters Page

The Parameters page allows you to specify the liquid-liquid hydrocyclone operation parameters as shown in Figure B-6.



Figure B-6 The parameters page for operation design tab

In regard to the parameters page, the list and describing of the parameters available in the parameters page is following the Table B-1.

Objects	Description
Liner Type dropdown list	Allows the user choose between three types of Vessel liner:
	- Vortoil G-Liners
	- Serck Baker Oil Spin
	- Custom liner
	Hydraulic parameters and physical dimensions change
	between the three types of Liner.
Number of Liners cell	Allows you the user specify the number of active vessel liners.
Min. Flow rate cell	Displays the minimum flow rate per liner depending on the

Table B-1 The lists and description of the parameters available in the Parameters page

	selected Liner type.
	- Vortoil recommends a minimum value of 2 m ³ /hr for the
	G-Liner.
	- Serck Baker recommends a minimum value of 4 m^3/hr
	for the OilSpin Liner.
	- Custom liner a minimum value of 2 m ³ /hr
Min. Reject Pressure cell	Allows you to specify the minimum Oil Overflow (Reject)
	downstream pressure.
PDR cell	Allows you to specify the Pressure Differential Ratio. The
	PDR is the ratio of the following stream pressure drops:
	PDR Inlet Pr essure 4 Overflow Pr essure
	Inlet Pr essure 4 Underflow Pr essure
Split Ratio cell	Allows you to specify the volume percent of the total inlet
	stream that passes to the overflow stream.
Underflow DP cell	Allows you to specify the pressure difference between the
	inlet stream and the underflow stream.
Underflow Pressure cell	Displays the pressure of the underflow stream.

c) Liner Details page

The Liner details page allows the user to manipulate the selected Liner type as shown in Figure B-7.

•	
200 - FL	
e-002	
e-002	
20.0	
1.5	
e-003	
e-002	
2.636	

Figure B-7 The liner details page for operation design tab

Regarding the liner details page, the list and describing of the parameters available for modification in the liner details page is following the Table B-2.

Objects	Description
Liner Type dropdown list	Allows the user choose between three types of Vessel
	liner:
	- Vortoil G-Liners
	- Serck Baker Oil Spin
	- Custom liner
	Hydraulic parameters and physical dimensions change
	between the three types of Liner.
Characteristic Diameter cell	Allows the user to specify the liner characteristic
	diameter, which is used to determine the diameter for the Inlet

Table B-2 The lists and description of the parameters available in the Liner details page

	and Underflow.
Inlet Diameter cell	Displays the calculated inlet diameter value.
Upper Taper cell	Displays the upper taper angle.
Lower Taper cell	Displays the lower taper angle.
Overflow Diameter cell	Allows you to specify the Overflow diameter.
Underflow Diameter cell	Displays the calculated Underflow diameter based on the
	selected Liner type and the specified characteristic diameter.
Total Length cell	Displays the Liner overall length of the selected Liner
	type's hydrocyclone geometry.

d) Droplet Distribution Page

The Droplet Distribution page allows the user to manipulate the Liquid-liquid Hydrocyclone performance, by modifying the dispersed oil droplet distribution as shown in Figure B-8.

Design	<u>O</u> il Droplet Distribution		
Connections	Droplet Sauter Mean [mm]	0.091	
)	Droplet d50 [mm]	0.130	
rarameters	Droplet d95 [mm]	0.270	
iner Details	Rosin-Rammler Index	2.00	
lotes			

Figure B-8 The droplet distribution page for operation design tab
The size distribution of oil droplets at the Hydrocyclone inlet is calculated using two parameters of the Rosin Rammler distribution. The Rosin Rammler distribution calculation is based on a mean droplet diameter and an exponential term power index.

Concerning the droplet distribution page, the list and describing of the parameters available in the droplet distribution page is following the Table B-3.

Table B-3 The lists and description of the parameters available in the droplet distribution page

Parameter	Description				
Droplet Sauter Mean	This is the droplet diameter whose volume to surface area				
	ratio is the same as that of the distribution as a whole and s				
	represents the surface area mean diameter.				
Droplet d50	This is the diameter of droplet at the 50% undersize point on				
	a cumulative volume distribution curve.				
Droplet d95	This is the diameter of droplet at the 95% undersize point on				
	a cumulative volume distribution curve.				
Rosin Rammler Index	This is the power term to which the exponential part of the				
	Rosin-Rammler Distribution is raised. Usually the value is between				
	1 and 2.5.				

e) User Variables Page

The user variables page allows you to create and implement variables in the HYSYS simulation case as shown in Figure B-9.

Design	▼ x x × A ₂ 121 ↔ ♥ ✓	
Connections Parameters Liner Details Droplet Distribution User Variables Notes		
Design Performance	Worksheet Dynamics	

Figure B-9 The user variables page for operation design tab

f) Notes Page

The notes page provides a text editor that allows you to record any comments or information regarding the specific unit operation, or the simulation case in general as shown in Figure B-10.

Design	Arial ▼ 10 ▼ 🕲 B I U 🗐 🗃 🗄 🗐 😭
Connections	
^D arameters	
_iner Details	
Droplet Distribution	
Jser Variables	
Notes	

Figure B-10 The note page for operation design tab

3.1.2 Performance tab

The performance tab display the calculated performance results of the liquidliquid hydrocyclone consists of the following pages:

- a) General
- b) Geometric
- c) Overflow
- d) Underflow
- e) Tables
- f) Plots

a) General Page

The General page displays the calculated general liner performance results as shown in Figure B-11.

Performance	<u>G</u> eneral		
General	Inlet Oil Conc. [ppm]	109273	
	Inlet Oil Conc. [mg/l]	114520	
ieometric	Max. Flowrate [m3/h]	4.88	
)verflow	d75 [microns]	76	
Indorflow	dP Overflow [kPa]	279	
JUTCHIOM	dP Underflow [kPa]	73.3	
ables	Reject Ratio [%]	5.37	
Plots	Efficiency [%]	46.4	

Figure B-11 The general page for performance tab

In regard to the general page, the description of the parameters available in the

general page is described by the following.

- Inlet oil concentration in parts per million (ppm) by volume and mg/l
- Maximum flow rate for the vessel. This value is calculated from the Liner

hydraulic characteristics.

- Droplet diameter separated with 75% efficiency at operating conditions
- Pressure drops at Overflow and Underflow relative to the Inlet
- System Reject Ratio
- System separation efficiency

b) Geometric Page

The geometric page displays the calculated geometric liner performance results as shown in Figure B-12.

Performance	<u>G</u> eometric Similarity	
General Geometric Overflow Underflow Tables Plots	Reynolds Number Hydrocyclone Number (Hy75)	28654 2.36e-003

Figure B-12 The geometric page in the performance tab

Regarding the geometric page, the description of the parameters available in the

geometric page is described by the following.

- Hydrocyclone Reynolds Number based on the Characteristic diameter
- Hydrocyclone Number (Hy75)

c) Overflow Page

The overflow page displays the calculated Overflow results as shown in Figure B-13.

Performance		1
General	Pressure [kPa] 221	
Geometric	Dil Concentration [ppm] 995884	
) verflow		
Inderflow		
ables		
Plots		
Design Performa	nce Worksheet Dynamics	
·		

Figure B-13 The overflow page in the performance tab

Concerning the overflow page, the description of the parameters available in the overflow page is described by the following.

- Overflow pressure
- Volumetric flow rate
- Oil concentration in parts per million (ppm)

d) Underflow Page

The underflow page displays the calculated Underflow results as shown in Figure B-14.

Performance	<u>U</u> nderflow	2	
General	Pressure [kPa]	427	
aeneiai	Flowrate [m3/h]	3.853	
Geometric	Oil Concentration [ppm]	58549	
) verflow	Oil Concentration [mg/l]	61361	
Inderflow			
ables			
00100			
Note			
Plots			

Figure B-14 The underflow page in the performance tab

About the underflow page, the description of the parameters available in the underflow page is described by the following.

- Underflow pressure
- Volumetric flow rate
- Oil concentration in parts per million (ppm) and mg/l

e) Tables Page

The tables page displays the tabulated results of the Oil Droplet Distribution or the Migration Probability. To view either result selects the appropriate radio button as shown in Figure B-15.

Performance	Droplet Dis	stribution	
General	Diameter [microns]	Vol. Undersize [%]	Iable View
achora	4.778	1.94e-006	Oroplet Distribution
Geometric	4.902	2.05e-006	C Migration Probability
Overflow	5.030	2.18e-006	
11	5.160	2.32e-006	
Undernow	5.295	2.48e-006	
Tables	5.433	2.67e-006	
Plots	5.574	2.88e-006	
1 1000	5.719	3.12e-006	
	5.868	3.39e-006	
	6.021	3.70e-006	
	6.178	4.05e-006	
	6.338	4.45e-006	
	6.503	4.90e-006	
	6.673	5.42e-006	
	6.846	6.00e-006	
	7.025	6.67e-006	
	7.207	7.43e-006	
	7.395	8.29e-006	
	7.588	9.27e-006	
	7.785	1.04e-005	_
Di D (- Northelese Domes		

Figure B-15 The tables page in the performance tab

f) Plots Page

The Plots page displays in graph format the results of the Oil Droplet Distribution or the Migration Probability. To view either plot selects the appropriate radio button as shown in Figure B-16.



Figure B-16 The plots page in the performance tab

3.3.3 Worksheet Tab

The Worksheet tab contains a summary of the information contained in the stream property view for all the streams attached to the operation.

3.3.4 Dynamics Tab

This unit operation is currently not available for dynamic simulation.

3.3.5 Nomenclature

The following Nomenclature has been adopted for the liquid-liquid hydrocyclone calculations following the Table B-4.

Table B-4 The lists and description of the variables available in the HYSYS

Variables	Symbol	Units
Volumetric Flow rate	Q_T	m ³ /hr
Maximum Volumetric Flow rate	$\mathcal{Q}_{\scriptscriptstyle MAX}$	m ³ /hr
Inlet Pressure	P _{IN}	Bar
Overflow Pressure	P _{REJ}	Bar
Underflow Pressure	P _{OUT}	Bar
Continuous Phase Density	$ ho_{c}$	kg/m ³
Oil Droplet Density	$ ho_{_o}$	kg/m ³
Hydrocyclone Characteristic Diameter	D	m
Continuous Phase Viscosity	μ_{c}	Pa.s
Droplet Diameter	d	microns
Sauter Mean Droplet Diameter	<i>d</i> _{3,2}	microns
50% Droplet Diameter	<i>d</i> ₅₀	microns

75% Droplet Diameter	<i>d</i> ₇₅	microns
95% Droplet Diameter	d_{g_5}	microns
75% Migration Probability Droplet	<i>d</i> ['] ₇₅	microns
Diameter		
Dimensionless Droplet Diameter	\varDelta_{75}	
Pressure Differential	ΔP	Bar
Separation Efficiency	З	%
Inlet Oil Concentration	C_i	ppm Vol.
Underflow Oil Concentration	C_o	ppm Vol.
Split Ratio	F	
Hydrocyclone Reynolds Number	$Re_{_D}$	
Hydrocyclone Number	Hy ₇₅	
Number of Liners	n_L	
Total Liner Length	L	m
Upper Taper Angle	θ_{i}	degrees
Lower Taper Angle	$ heta_{2}$	degrees

APPENDIX C

COMPOSITION OF PALM BIODIESEL

	RBD Palm Oil
Methyl Myristate, %wt	1.19
Methyl Palmitate %wt	27.10
Methyl Palmitoleate %wt	1.78
Methyl Stearate, %wt	7.25
Methyl Oleate, %wt	42.93
Methyl Linoleate, %wt	18.68
Methyl Linolenate, %wt	1.07

Table C-1 Composition of Methyl Esters from RBD Palm Oil (Tongurai et al., 2006)

According to the Table C-1, methyl oleate is the highest composition of methyl ester from palm oil, and then using of methyl oleate to represented palm biodiesel in the simulation is agreement.

APPENDIX D

STANDARD SPECIFICATION OF PALM BIODIESEL

Table D-1 Standard Specification of palm biodiesel (B100), Department of Energy Business, Ministry of Energy

1	Methyl Ester, % wt.	min	96.5	EN 14103	
2	Density at 15 °C kg/m ³	min	860	ASTM D 1209	
2	Density at 15°C, kg/m	max	900	ASTM D 1298	
2	Viscosity at 40 °C CSt	min	3.5	A STM D 445	
5	Viscosity at 40°C, CSt	max	5.0	AS1M D 445	
4	Flash Point, °C	min	120	ASTM D 93	
5	Sulphur, %wt.	max	0.0010	ASTM D 2622	
6	Carbon Residue, on 10 % distillation residue, %wt)	max	0.30	ASTM D 4530	
7	Cetane Number	min	51	ASTM D 613	
8	Sulphated Ash, %wt.	max	0.02	ASTM D 874	
9	Water, %wt.	max	0.050	EN ISO 12937	
10	Total Contaminate, %wt.	max	0.0024	EN 12662	
11	Copper Strip Corrosion	max	No. 1	ASTM D 130	
12	Oxidation Stability at 110 °C, hours	max	6	EN 14112	
13	Acid Value, mg KOH/g	max	0.50	ASTM D 664	
14	Iodine Value, g Iodine / 100 g	max	120	EN 14111	
15	Linolenic Acid Methyl Ester, %wt	max	12.0	EN 14103	
16	Methanol, %wt	max	0.20	EN 14110	
17	Monoglyceride %wt.	max	0.80	EN 14105	
18	Diglyceride, %wt.	max	0.20	EN 14105	

19	Triglyceride, %wt.	max	0.20	EN 14105		
20	Free glycerin, %wt.	%wt. max 0.02				
21	Total glycerin, %wt.	max 0.25				
22	Group I metals (Na+K), mg/kg	max	5.0	EN 14108 and EN 14109		
	Group II metals (Ca+Mg), mg/kg	max	5.0	pr EN 14538		
23	Phosphorus, %wt.	max	0.0010	ASTM D 4951		
24	Additive	Approved by DG of DOEB				

APPENDIX E

1.1 Study and design liquid-liquid hydrocyclone

1.1.1 Introduction

The liquid-liquid hydrocyclone performance is relatively complicated then the study of basic principle and ordinarily designing are required. The separation is effected by many section of liquid-liquid hydrocyclone, and then the character of each section was investigated. And there are many previous literature data about the fundamental designing of liquid-liquid hydrocyclone. Despite of the accurate hydrocyclone geometry platform not available and then designing of individual dimension in this study was developed.

1.1.2 The impact of characteristic section of hydrocyclone

The liquid-liquid hydrocyclone configuration based has two discrepant types as classic cone and double cone. The complicated of geometry and dimension designing due to the pressure drop, three-dimensional and high turbulent swirling flow.

Regarding the double cone geometry, the viscous stresses acting on droplets in the inlet chamber region and the cyclone wall region was decreased, as a result of the tangential velocity is reduced with compared to the classic cone geometry.

A double cone geometry hydrocyclone also shown smoother velocity gradient in flow field than a standard classic cone geometry and also shown the lower breakup rates of droplet, which improving the separation performance. As shown in Figure E-1.

Concerning the flow behavior within hydrocyclone, in which the amount of fluid going through the different outlets which variously with heavy and light density. It means that for these two different separation cases then two different geometries are needed.



Figure E-1 The discrepancy of cone geometry, (a) double cone, (b) standard classic cone

The hydrocyclone configuration is comprised of two different geometries as cylindrical section and conical section, and also divided into four subsections as shown in Figure E-2. The first and second parts are inlet chamber and reducing section respectively, which designed to produce higher tangential acceleration of the fluid and also reducing the pressure drop and the shear stress reach to an acceptable level. The latter section, reducing section is designed to minimized and avoid droplet breakup and it also leading to the reduction in separation efficiency. The following section, taper section is where the most of the separation achieved. Concerning the low angle of this segment is carrying high swirl intensity along with high residence time. The last section, long tail cylindrical section is an integrated design part in which the smallest droplets are separated. Another individual part of hydrocyclone is the overflow port, the separated droplet be migrate to the reversed flow core nearly the axis region and being separated. So this configuration of overflow exit is utilizing a very stable small diameter because of small diameter reversed flow core. And most of commercial hydrocyclone change the overflow diameter which depending on the range of operating conditions.



Figure E-2 The illustration of each sub-sectional part of hydrocyclone

1.1.3 The hydrocyclone dimensional design

(a) The hydrocyclone dimensional from HYSYS user guide



Figure E-3 The illustration of hydrocyclone dimension detail

Table E-1 Geometric parameters for HYSYS hydrocyclone

HYSYS	D _c	D _n	D _{in}	D _o	D _u	θ_{1}	θ_{2}	L_4
Hydrocyclone	Define	0.5D _c	0.35D _c	Define	0.25D _c	20	1.5	20D _c

(b) The hydrocyclone dimensional from literature data (Colman and Thew., 1983)

Table E-2 Geometric parameters for literature data

Literature	D _c	D _n	D _{in}	D _o	D _u	θ_{1}	θ_{2}	L ₄
Hydrocyclone	Define	0.5D _c	Define	0.05D _c	0.25 D _c	20	0.75	15D _c

There are the same in proportion of D_n and D_u of both hydrocyclone. The

difference dimension between hydrocyclone of HYSYS user guide and literature is the angle, length and inlet diameter.

(c) The hydrocyclone dimensional in this study

Table E-3 Geometric parameters in this study

Literature	D _c	D _n	D _{in}	D _o	D _u	θ_{1}	θ_{2}	L ₄
Hydrocyclone	Define	0.5D _c	0.35D _c	0.05D _c	0.25 D _c	20	1.5	8.55D _c

The proposed hydrocyclone was similar with HYSYS dimension. The once difference of proposed hydrocyclone was total length, which represent by L_4 that proposed hydrocyclone have total length shorter that HYSYS hydrocyclone.

APPENDIX F

HYDROCYCLONE DIMENSION DETAILS



UNIT : mm.

Figure F-1 The proposed hydrocyclone dimension details

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- Sawangpon, W., Sukmanee, S., and Keawpradit, P. 2011. Preliminary study of water-methyl ester separation via Aspen-HYSYS. Proceedings of 21st Thai Institute of Chemical Engineering And Applied Chemistry (TICHE-2011) Thailand. November 10-11: 114.
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