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THESIS

DEVELOPMENT OF CONTINUOUS BIODIESEL PRODUCTION WITH STATIC MIXER, ULTRSONIC AND MICROWAVE

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A Thesis Submitted in Partial Fulfillment of
the Requirements for the Degree of
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Even through most of biodiesel productions are batch processes, a combination of static mixer, microwave, ultrasonic, centrifugal and washing are proposed as the continuous biodiesel production. A computational fluid dynamics (CFD) is used to design of the static mixer and the continuous washing equipment. Both palm oil with 6:1 methanol to oil ratio and 1% wt potassium hydroxide are experimental used. In this part, the conventional stirred tank temperature is controlled at 60 °C, the static mixer, the microwave and the ultrasonic flow rate are set at 3 L/min. As a result, the static mixer showed the optimal condition for continuous biodiesel production. Washing operation includes the centrifugal separators suitable for separate between biodiesel and glycerol with the flow rate 1.5 L/min. The flow rates of biodiesel and water at 1:1 ratio varied from 1.5, 3.0 and 4.5 L/min, and the washing cycle from 3, 4 and 5 times. The result shows that the washing flow rate does not significantly affect to the purity of biodiesel and the cycle at 3 times is the optimal for continuous washing biodiesel production.

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Atinudda Ratchatasot
April 2011

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LIST OF ABBREVIATIONS

Abbreviations

A = Area
B = Beta function
cP = Centipoi
CFD = Computational Fluid Dynamics
D = Diameter of mixer element
F_c = Centrifugal acceleration
FAME = Fatty Acid Methyl Ester
g = gram
GC = Gas chromatography
kg = kilogram
kg/s = kilogram per second
L/min = liter per minutes
MW_p = Molecular weight of palm oil
MW_m = Molecular weight of methanol
m. = meter
min = minutes
mg = milligram
ml. = milliliter
$\dot{m}_m$ = Mass flow rate of methanol
$\dot{m}_p$ = Mass flow rate of palm oil
N = Angular speed
n = Number of observations in the sample
R = Radius
r_l = density of interstitial liquid
r_s = density of particle
rpm = round per minutes

LIST OF ABBREVIATIONS **(Continued)**

s = Second

S^2 = Variance of the sample

t = Test statistic

T = Temperature

u = Fluid velocity

V = Volume

$V_{reactor}$ = Volume of reactor

$V_{thickness}$ = Volume of thickness

X_p = Ratio of palm oil in fluid

X_m = Ratio of methanol in fluid

$\bar{X}$ = Mean of the sample

μ_m = Viscosity of methanol

μ_p = Viscosity of palm oil

ρ = Density

ρ_m = Density of methanol

ρ_p = Density of palm oil

υ = Degree of freedom

DEVELOPMENT OF CONTINUOUS BIODIESEL PRODUCTION WITH STATIC MIXER, ULTRSONIC AND MICROWAVE

INTRODUCTION

At present, energy is one of important factors for human life and the manufacture industries. The energy demands are significantly increased but petroleum based fuel is limited that may cause the increasing in the price of the energy. Therefore, we have to explore other alternative energy resources which can compensate petroleum based fuel, have enough quantity, and low price such as wind power, solar energy and biodiesel etc (Nezihe and Aysegul, 2007a). One of the alternative fuels that get interested from the Thai government is biodiesel because biodiesel is renewable, biodegradable and non-toxic, produces lower carbon monoxide (CO) and hydrocarbon emissions than petroleum based diesel fuel when burned. Moreover, it contributes a minimum amount of greenhouse gases or sulfur to atmosphere. (Nicholas and Lauren, 2006; Nezihe and Aysegul, 2007b)

Biodiesel is an ester that produced by synthesized vegetable oils such as palm oil, rapeseed oil, coconut oil and jatropha oil etc., animal fats or waste vegetable oil. There are many methods to produce biodiesel such as microemulsion, pyrolysis, or transesterification (Hideki et al., 2001). However, the favored method that people always use is transesterification due to the low reaction temperature method. Transesterification reaction is the reaction between triglyceride and alcohol in the presence of catalyst to get the alkyl ester (biodiesel) and glycerol as the products.

The catalyst that is normally used in transesterification reaction is alkali, acid or enzyme. Acid catalyst is useful when amount of the free fatty acid in oil is high but the reaction time is long. For the enzyme catalyst, lipases are used as the catalyst that does not produce side reactions and the reaction can occur at normal temperature and pressure. But this catalyst has some disadvantages which are the expensive cost of lipases and the unstable of lipases. In the alkali catalyst, the reaction is faster and lower reaction temperature than the acid catalyst but the side reaction can take place. The side reaction will produce soap called “saponification reaction” when there are water and free fatty acid in the oil. The formed soap will take a long time to separate the product and it will also reduce the yield of alkyl ester. (Carmen et al., 2004)

The glycerol is largely removed by gravity separation or centrifugation and the methanol may in some cases be removed the flash evaporation. Neither process is 100% efficient hence a final purification stage is needed in order to meet the requirements of EN 14214 Standard.

The methyl esters cannot be classified as biodiesel until the EN 14214 Standard specifications are fulfilled. Therefore, the purification stage is essential. The untreated biodiesel contains several impurities: free glycerol, soap, metals, methanol, free fatty acids (FFA), catalyst, water and glycerides. The engine life can be reduced by high levels of impurities. There are two generally accepted methods to purify biodiesel: wet and dry washing. The more traditional wet washing method is widely used to remove excess contaminants and leftover production chemical from biodiesel. (Berrios and Skelton, 2008)

In this project, the transesterification is used to manufacture the biodiesel in the presence of alkali catalyst on the batch reactor. The ultrasonic wave and microwave are act as assistant in tranesterification reaction while the static mixer and the continuous washing equipment which were designed using CFD was used to mix the mixture. The effects of the reaction temperature and the flow rate of the reactants are explored.

OBJECTIVES

1. To evaluate efficiency of operating units in biodiesel production process.

2 To apply corresponding optimal units to the real process.

Scopes of work

1. The flow behavior of designed static mixer and wash biodiesel process was simulated by Computational Fluid Dynamics (CFD)

2. Refined palm oil, jatropha, methanol and potassium hydroxide are used as raw material

3. Equipments used in this work are:
   - Stirred tank reactor: capacity 100 liters
   - Ultrasonic: power 200 watt and frequency 28 kHz
   - Microwave: power 800 watt
   - Static mixer: length 54 centimeter. and 7 mixer elements
   - Separator: power 120 watt and speed 10,500 rpm

LITERATURE REVIEW

This section is divided into five main parts. The first part is reviewed the literatures the biodiesel, and the second part is reviewed the biodiesel production process. Then the third part shows the equipments for the continuous biodiesel production. Next part is reviewed the t-test for experiment. Finally, review the Computation Fluid Dynamic (CFD).

1. Biodiesel

Biodiesel is a diesel equivalent fuel derived biologic sources such as vegetable oils, animal fats, or recycled restaurant greases which can be used in unmodified diesel engine vehicle. Biodiesel has several district advantages with petrodiesel in addition to being fully competitive with petrodiesel in most technical aspects.

- Derivation from a renewable domestic resource ,thus reducing Dependence on and preserving petroleum
- Biodegradability
- Reduction of most exhaust emissions (with the exception of nitrogen oxidation NO_x)
- Higher flash point, leading to safer handling and storage
- Excellent lubricity, a fact that is steadily gaining importance with the advent of low – sulfur petrodisel fue

There are three basic routes to biodiesel production from oils and fats:

- Base catalyzed transesterification of the oil.
- Direct acid catalyzed transesterification of the oil.
- Conversion of the oil to its fatty acids and then to biodiesel.

Almost all biodiesel is produced using base catalyzed transesterification as it is the most economical process requiring only low temperatures and pressures. The transesterification reaction is shown in Figure 1.

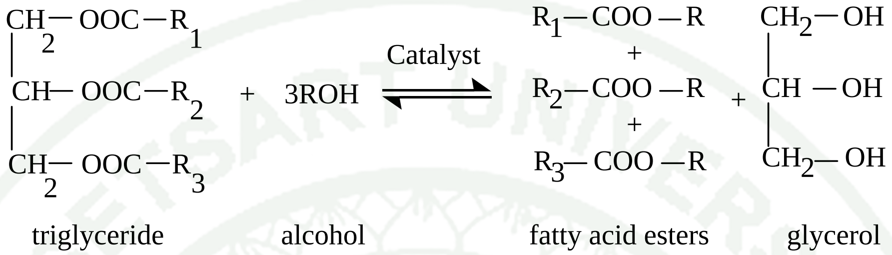


Figure 1 Transesterification reaction

Biodiesel production by transesterification contains three important substances, trigyceride, as the main component of vegetable oil or animal fat, as shown in Figure 2., alcohol and catalyst. The properties of biodiesel depends on the hydrocarbon chain of reacting triglyceride such as total number of carbon atom and number of double bounds in the hydrocarbon chain. There are variety vegetable oils which compound many fatty acids as shown in Table 1.

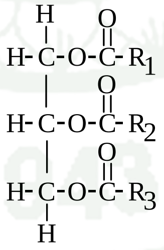


Figure 2 R1, R2 and R3 are hydrocarbon chain of fatty acyl groups of triglyceride

Table 1 Chemical properties of each vegetable oil

Vegetable Oil	Fatty acid composition, % by weight								
	16:0	18:0	20:0	22:0	24:0	18:1	22:1	18:2	18:3
Corn	11.67	1.85	0.24	0.00	0.00	25.16	0.00	60.6	0.48
Cottonseed	28.33	0.89	0.00	0.00	0.00	13.27	0.00	57.51	0.00
Crambe	2.07	0.7	2.09	0.8	1.12	18.86	58.51	9	6.85
Peanut	11.38	2.39	1.32	2.52	1.23	48.28	0.00	31.95	0.93
Rapeseed	3.49	0.85	0.00	0.00	0.00	64.4	0.00	22.3	8.23
Soybean	11.75	3.15	0.00	0.00	0.00	23.26	0.00	55.53	6.31
Sunflower	6.08	3.26	0.00	0.00	0.00	16.93	0.00	73.73	0.00
Poppyseed	12.6	4	0.00	0.00	0.00	22.3	0.00	60.2	0.5
Safflower	7.3	1.9	0.00	0.00	0.00	13.6	0.00	77.2	0.00
Seameseed	13.1	3.9	0.00	0.00	0.00	52.8	0.00	30.2	0.00
Linseed	5.1	2.5	0.00	0.00	0.00	18.9	0.00	18.1	55.1
Wheat grain*	20.6	1.1	0.00	0.00	0.00	16.6	0.00	56	2.9
Palm	42.6	4.4	0.00	0.00	0.00	40.5	0.00	10.1	0.2
Tallow	23.3	19.3	0.00	0.00	0.00	42.4	0.00	2.9	0.9
Hazeelnot Kernel	4.9	2.6	0.00	0.00	0.00	83.6	0.00	8.5	0.2
Walnut Kernel	7.2	1.9	0.00	0.00	0.00	18.5	0.00	56	16.2
Almond Kernel	6.5	1.4	0.00	0.00	0.00	70.7	0.00	20	0.00
Olive Kernel	5	1.6	0.00	0.00	0.00	74.7	0.00	17.6	0.00

*Wheat grain oil contains 11.4% of 8:0 and 0.4% of 14:0 fatty acids.

Source : Demirbas (2002)

Table 2 The biodiesel specification of EN 14214 and ASTM D 675

Properties	EN 14214		ASTM D 6751	
	Unit	Limits	Unit	Limits
Ester content	% (m/m)	96.5	-	-
Density at 15 °C	kg/m^3	860-900	-	-
Viscosity at 40 °C	mm^2/s	3.5-5.0	mm^2/sec	1.9-6.0
Flash point	°C	120 min	°C	130 min
Sulfur content	mg/kg	10 max	% mass	0.05 max
Carbon residue	% (m/m)	0.3 max	% mass	0.05 max
Cetane number		51 min		47 min
Sulfated ash	% (m/m)	0.02 max	% mass	0.02 max
Water content	mg/kg	500 max	% volume	0.05 max
Total contamination	mg/kg	24 max	-	-
Copper strip corrosion	rating	class 1		No. 3 max
Cloud Point	-	-	°C	Report
Oxidation stability	hours	6 min	-	-
Acid Value	Mg KOH/g	0.5 max	Mg KOH/g	0.8 max
Iodine value	g/100g	120 max	-	-
Linoolenic acid ME	% (m/m)	12 max	-	-
Polyunsat ME	% (m/m)	1 max	-	-
Methanol content	% (m/m)	0.2 max	-	-
Monoglyceride	% (m/m)	0.8 max	-	-
Diglyceride	% (m/m)	0.2 max	-	-
Triglyceride	% (m/m)	0.2 max	-	-
Free glycerol	% (m/m)	0.02 max	% (m/m)	0.02
Total glycerol	% (m/m)	0.25 max	% (m/m)	0.24
Alkali metals (Na+K)	mg/kg	5 max	-	-
Phosphorus content	mg/kg	10 max	% mass	0.001 max
Distillation temp.	-	-	°C	360 max

When complete the reaction, it must check standard for biodiesel. There are two international standards, EN 14212 and ASTM D 6751 as shown in Table 2

1.1 Transesterification

Transesterification consists of 3 stepwise with 2 intermediates formation of diglycerides (DG) and monoglycerides (MG). These 3 steps are shown in Figure 3

In the transesterification of biodiesel, a triglyceride reacts with an alcohol in the presence of a strong acid or base, producing a mixture of fatty acids alkyl esters and glycerol. The overall process is a sequence of three consecutive and reversible reactions, in which di and monoglycerides are formed as the intermediates.

The stoichiometric reaction requires 1 mol of a triglyceride and 3 mol of the alcohol. However, an excess of the alcohol is used to increase the yields of the alkyl esters and to allow its phase separation from the glycerol formed.

$$\text{Triglyceride (TG)} + \text{ROH} \overset{\text{Catalyst}}{\rightleftharpoons} \text{Diglyceride (DG)} + \text{RCOOR}_1$$

$$\text{Diglyceride (DG)} + \text{ROH} \overset{\text{Catalyst}}{\rightleftharpoons} \text{Monoglyceride (MG)} + \text{RCOOR}_2$$

$$\text{Monoglyceride (MG)} + \text{ROH} \overset{\text{Catalyst}}{\rightleftharpoons} \text{Glycerol (GL)} + \text{RCOOR}_3$$

Figure 3 Three steps of transesterification reaction .

The mechanism of the base - catalyzed transesterification of vegetable oils is shown in Figure 4. The first step is the reaction of the base with the alcohol, producing an alkoxide and the protonated catalyst. The nucleophilic attack of the alkoxide at the carbonyl group of the triglyceride generates a tetrahedral intermediate in second step, from which the alkyl ester and the corresponding anion of the diglyceride are formed in third step. The latter deprotonates the catalyst, thus regenerating the active species, which is now able to react with a second molecule of the alcohol, starting another catalytic cycle. Diglycerides and monoglycerides are converted by the same mechanism to a mixture of alkyl esters and glycerol.

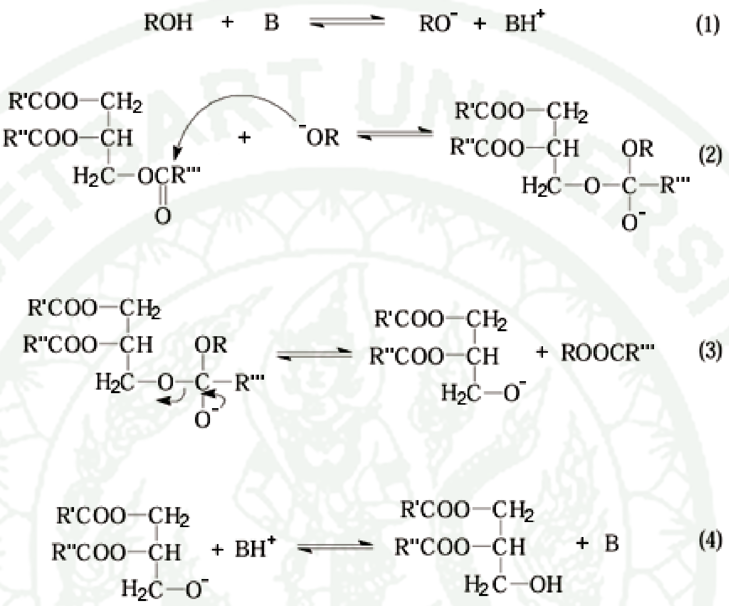


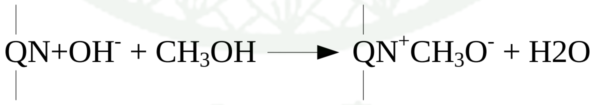
Figure 4 The mechanism of alkali catalyst transesterification

Transesterification can be either homogeneous or heterogeneous methods. However, each method has different advantages. (Marchetti *et. al.* 2007) studied the possible methods for biodiesel production. For the homogeneous method, there are several kinds of catalyst such as alkali-catalyzed and acid-catalyzed. The advantages of alkali-catalyzed method are the fast reaction, high conversion and less corrosive than acid-catalyzed. However, the disadvantage of alkali-catalyzed method is the side -reaction that is saponification. The OH^- can react with free fatty acid to form soap. The soap formation is an undesirable side-reaction, because it consumes the catalyst, decreases the yield of biodiesel and complicates the separation and purification. The problem of the soap formation is solved by using acid-catalyzed method since it does not have OH^-. However the acid-catalyzed is not a favorite method because slow reaction, corrosive catalyst and high condition (e.g. pressure and temperature). For the heterogeneous method, there are several kinds of catalysts proposed. Lipase is one of heterogeneous catalysts. The advantages are the easy separation between catalyst and glycerin. Nevertheless, the disadvantages of lipase catalyst are slow reaction and expensive cost. Other catalysts in heterogeneous process are Na/NaOH/g-Al_2O_3, ZrO_2-SiO_2, KOH/ZrO_2-SiO_2, Co_2O_3-SiO_2, Mo_2O_2-SiO_2, Na_2O-SiO_2, La_2O_3-MCENTIMETER-41 and MgO.

A mechanism is operative in the case of a heterogeneous basic Bronsted catalyst such as basic zeolite Also in this case, the formed catalytic specie is a homogeneous alkoxide.

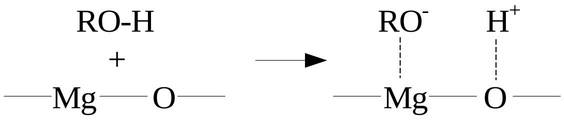
$$O.Na^+ + CH_3OH \longrightarrow O\text{-}H + CH_3ONa^+ \quad (1)$$

In the case of heterogeneous basic Bronsted catalyst such as resin with quaternary ammonium functionality (QN+OH-), the positive counterions (organic ammonium groups), being bonded directly to the support surface, electronically retain the catalytic anions on the solid surface: The reaction occurs between methanol adsorbed on the cation and ester from the liquid (Eley–Rideal mechanism).



$$QN{+}OH^- + CH_3OH \longrightarrow QN^+CH_3O^- + H2O \quad (2)$$

The formation of alkoxide groups is also a fundamental step for heterogeneous basic Lewis catalyst. For example, in the case of ethylacetate transesterification, catalyzed by MgO, the reaction occurs between the methanol molecules adsorbed on amagnesium oxide free basic sites and the ethyl acetate molecules.



$$\begin{array}{c} RO\text{-}H \\ + \\ \text{—Mg—O—} \end{array} \longrightarrow \begin{array}{cc} RO^- & H^+ \\ \vdots & \vdots \\ \text{—Mg—} & \text{O—} \end{array} \quad (3)$$

The kinetic model of biodiesel formation was proposed by many researches (Freedman, 1986; Noureddini and Zhu, 1997; Darnoko and Munir, 2000; Foon *et al.*, 2004; Karmee *et al.*, 2004; Vicente *et al.*, 2005; Vicente *et al.*, 2006). The general form of governing differential equations involving six steps is following:

$$\frac{d[TG]}{dt} = -k_1[TG][A] + k_2[DG][E]$$
$$\frac{d[DG]}{dt} = k_1[TG][A] - k_2[DG][E] - k_3[DG][A] + k_4[MG][E]$$
$$\frac{d[MG]}{dt} = k_3[DG][A] - k_4[MG][E] - k_5[MG][A] + k_6[GL][E]$$
$$\frac{d[GL]}{dt} = k_5[MG][A] - k_6[GL][E]$$
$$\frac{d[E]}{dt} = k_1[TG][A] - k_2[DG][E] + k_3[DG][A] - k_4[MG][E] + k_5[MG][A] - k_6[GL][E]$$
$$\frac{d[A]}{dt} = -\frac{d[E]}{dt}$$

where A and E are the alcohol and ester concentrations, respectively.

In addition, the activation energy and reaction rate shown in the Table 3 and 4 were estimated by the Arrhenius equation.

$$k(T) = AT^{n}e^{-E/RT} \tag{4}$$

Table 3 The energy of activation and reaction rate constant at 50 °C using NaOH

Reaction	Energy of Activation (cal/mol)	Reaction rate constants
TG → DG	13145	4.80E+07
DG → TG	9932	5.81E+05
DG → MG	19860	2.70E+11
MG → DG	14639	9.98E+09
MG → GL	6421	5.38E+07
GL → MG	9588	2.17E+04

Source: Noureddini and Zhu (1997)

Table 4 The energy of activation and reaction rate constant at 65 °C using SrO

Reaction	Energy of Activation (cal/mol)	Reaction rate constants
TG → DG	16.8	2.46E+10
DG → MG	7.97	1.79E+05
MG → GL	59.79	1.13E+34

Source: Petchtabtim (2008)

1.2 The Factors of Biodiesel's Transesterification

The factors or parameters that have the effects to the transesterification of the biodiesel manufacture in the presence of base catalyst are the following.

1.2.1 Type and Quantity of Catalyst

The different type of catalyst does not significantly effect to the reaction time but effects to the purity and yield of biodiesel. If the sodium hydroxide and potassium hydroxide is compared, it seems that in presence of sodium hydroxide the biodiesel purity is higher than in presence of potassium hydroxide due to the more selective of sodium hydroxide. In the other hand, in the presence of potassium hydroxide the biodiesel yield is higher than in the presence of sodium hydroxide because the potassium soap is softer than the sodium soap. Therefore, it is more soluble in water and is easier to wash, thus the yield of biodiesel is high. About the catalyst quantity, the reaction is faster when the concentration of catalyst is increased. (Nezihe and Aysegul, 2007a; Carmen *et.al*, 2004)

1.2.2 Type of Alcohol

A process with a steady product quality required an installation space less than the batch technology at equal capacity but at higher investment cost. (Carmen *et.al*, 2004)

1.2.3 Temperature

The purity of biodiesel is increased when reaction temperature is increased. However, the transesterification reaction temperature should at the optimum because if the temperature is too high, the alcohol will be lost and the operating cost will be high. (Nezihe and Aysegul, 2007a)

1.2.4 Reaction Time

The increasing of reaction time causes the increasing of biodiesel purity. The rate of increasing is depended on other reaction parameter such as alcohol-oil ratio, quantity of catalyst etc. (Nezihe and Aysegul, 2007a; Nezihe and Aysegul, 2007b)

1.2.5 Alcohol-Oil Ratio

At least the 3 mole of alcohol and 1 mole of oil are needed to complete the transesterification reaction and gain the 3 mole of alkyl ester and a mole of glycerol. However, the excess alcohol is required to shift the equilibrium to the product, thus the higher ratio of alcohol-oil may lead to the higher conversion of oil. (Carmen *et.al*, 2004; Carmen *et.al*, 2006a)

1.2.6 Type of Oil

The different types of oil or animal fat have the various fatty acids in different concentration so they may effect to the biodiesel properties. (Carmen *et.al*, 2006a; Carmen *et.al*, 2006b)

1.2.7 Mixing

The mixing efficiency of the reactants that effects to the yield of biodiesel is the important factor for transesterification reaction because the alcohol and oil are not completely miscible. The mixing will produce the tiny droplets which increase the reaction area. The mixing system that is always used is stirrer or ultrasonic wave. (Carmen *et.al*, 2004; Carmen *et.al*, 2006a; Carmen *et.al*, 2006b)

1.2.8 Quantity of Water and Free Fatty Acid

In alkali catalyst, the water and free fatty in the oil and the hydroxide group in the structure of catalyst will cause the side reaction called saponification. This side reaction forms the soap and consumes the catalyst. The soap can be dissolved in the glycerol phase. So the separation of alkyl ester must be cared in order to gain the most purity and yield of alkyl ester. Moreover, the soap will cause the slow separation time that will increase the cost of separation. (Nezihe and Aysegul, 2007a; Nezihe and Aysegul, 2007b; Hideki *et.al,* 2001; Carmen *et.al,* 2004)

2. Biodiesel Production Process

The base catalyzed production of biodiesel takes place as shown in Figure 5 and the detail of the biodiesel production process will explain later on.

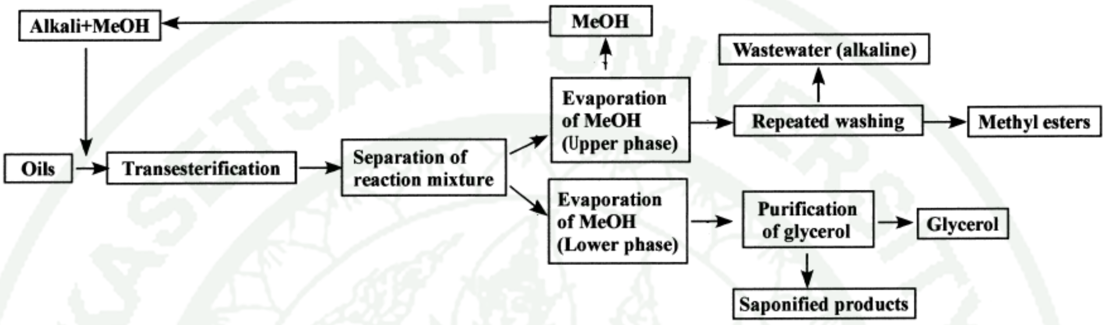


Figure 5 The biodiesel production process scheme.

Source: Hideki (2001)

2.1 Pretreating and Mixing

The vegetable oil have to be pretreated by removed the components such as free fatty acid and water that may damage the following step. And the catalyst and alcohol have to mix each other before they are added to the reactor. (Jonh, 2003; Gupta, 2008)

2.2 Reaction

The system from here should be closed in order to prevent loss of alcohol. The tranesterification reaction takes place which the pretreated oil and alcohol are reacted to form alkyl ester and glycerol. In normally, the excess alcohol is used to ensure the conversion of oil to alkyl ester. Moreover, the monitor about the quantity of free fatty acid and water in the oil should be careful because the high amount of them will cause the problem of the soap formation and the product separation. (Jonh, 2003; Gupta, 2008)

2.3 Separation

When the transesterification reaction is completed, the product, by-product, excess alcohol and catalyst will separate into two phases, upper layer and lower layer, due to the different density. Upper layer consists of biodiesel, alcohol and some soap while glycerol, alcohol, catalyst, impurities and trace of unreacted oil are in the lower layer. Thus, these two phases can be separated by gravity but sometimes the centrifugal is used to accelerate the separation. (Jonh, 2003; Gupta, 2008)

2.4 Alcohol Removal

After the glycerol and biodiesel is separated, the excess alcohol in each phase is removed by flash separation or distillation. The removed excess alcohol is recycled back to the reactor. (Jonh, 2003; Gupta, 2008)

2.5 Glycerol Neutralization

The catalyst and soap in the glycerol are neutralized by acid before send to the storage as the crude glycerol. The 80-88% purity of glycerol which is produced by removing water and alcohol is sold as crude glycerol. But in some operation, the 99% or higher purity is used to produce cosmetic or pharmaceutics. (Jonh, 2003; Gupta, 2008)

2.6 Washing

Before washing, Acid is added to neutralize the residue catalyst and to split soap. To purify the biodiesel, the excess alcohol and soap must be removed by washing with warm water because water is immiscible with biodiesel. So it can be easily separated from biodiesel. This is the end of biodiesel production process resulting in the yellow liquid with the same viscosity as diesel fuel. (Jonh, 2003; Gupta, 2008; Jon, 2005)

3. Equipments

3.1 Ultrasonic Wave

The ultrasonic wave is always used for emulsification of immiscible liquid to solve the mixing problem. The cavitational bubbles collapse at the boundary of oil and alcohol and create the tiny droplets of alcohol by the ultrasonic jet which impinges to oil phase in order to improve the reaction. Thus, the reaction is accelerated, completed faster and uses less amount of catalyst than mechanical stirring.(Carmen *et.al*, 2004; Wang *et.al*, 2007; Carmen *et.al*, 2006a; Carmen *et.al*, 2006b)

The frequency of the ultrasonic wave has the effect to the yield of biodiesel. The high frequency ultrasonic will have less the reaction time and biodiesel's yield than the low frequency. Because at the high frequency the formed soap is increased so the formation of alkyl ester is fast. But the separation of alkyl ester is hard due to the trapped of alkyl ester in the soap, thus the yield of biodiesel is decreased. (Carmen *et.al*, 2004)

3.2 Microwave

The microwave irradiation is the alternative heating system which is used in the transesterification reaction. It activates the variance of polar molecules and ions with the continuous magnetic field. The changing of electric field causes the molecules or ions to rotate rapidly and generate heat because of the friction of molecule. Therefore, the reaction is accelerated and produces high product yield in the short time. Moreover, the quantity of by-product is decreased so the separation time is reduced. (Nezihe and Aysegul, 2007a; Nezihe and Aysegul, 2007b**)**

3.3 Static Mixer

A static mixer or motionless mixer is a device for mixing two fluid materials. It consists of mixer elements contained in a cylindrical (tube) or squared housing. The purpose of the elements is to redistribute fluid in the directions transverse to the main flow such as in the radial and tangential directions. The effectiveness of this redistribution is a function of the specific design and number of elements. When compare with the conventional stirring, the static mixer is similar and sometimes better performance can be achieved at lower cost. Static mixer typically has lower energy consumptions and reduced maintenance requirements because they have no moving parts. The some potential advantages of static mixer over conventional stirring are given in Table 5. (Thakup *et.al*, 2003)

Table 5 Potential advantages of static mixer compared to conventional stirring

Static mixer	**CSTR**
Small space requirement	Large space requirement
Low equipment cost	High equipment cost
No power required except pumping	High power consumption
No moving parts except pump	Agitator drive and seals
Small flanges to seal	Small flanges plus one large flange to seal
Short residence times	Long residence times
Approaches plug flow	Exponential distribution of residence times
Good mixing at low shear rates	Locally high shear rates can damage sensitive materials
Fast product grade changes	Product grade changes may generate waste
Self-cleaning, interchangeable mixers or disposable mixers	Large vessels to be cleaned

Source: Thakup *et.al* (2003)

The commercial static mixers can be divided into five main families based on the geometry of the mixing elements which are

- open designs with helices

Figure 6 Kenics mixer.

Source: Thakup *et.al* (2003)

- open designs with blades

Figure 7 Low pressure drop mixer.

Source: Thakup *et.al* (2003)

- corrugated-plates

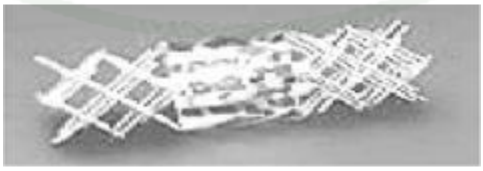


Figure 8 SMV mixer.

Source: Thakup *et.al* (2003)

- multi-layers designs

Figure 9 SMX mixer.

Source: Thakup *et.al* (2003)

- closed designs with channels or holes

Figure 10 Interfacial Surface Generator (ISG) mixer.

Source: Ross (2009)

3.4 Centrifugation and centrifuges

Centrifugation is a separation process which uses the action of centrifugal force to promote accelerated settling of particles in a solid-liquid mixture. Two distinct major phases are formed in the vessel during centrifugation :

The sediment

Usually does not have a uniform structure. Find below an example of a sediment deposit :

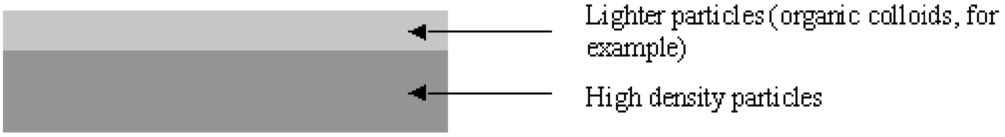


Figure 11 example of a sediment deposit

Source: Lenntech (2009)

3.4.1 The centrifugate or centrate which is the supernatant liquid.

Often clear though sometimes cloudy, due to the presence of very fine colloidal particles that are not readily settled. However it may also contain several phases if the mixture's interstitial liquid contains element with different densities, such as oils for example.

3.4.2 Centrifugal force

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

$$F_c = w^2R = 0.011\ N^2R \tag{5}$$

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

$$F_c = 0.011\ N^2R\ (r_s - r_l) \text{ x } 1/g = G\ (r_s - r_l), \tag{6}$$

with $G = w^2R / g = 0.11\ N^2R / 9.81 = 11.2 \text{ x } 10^{-4}\ N^2R$

Where r_s : density of particle

r_l : density of interstitial liquid

3.4.3 Centrifuges

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

3.4.4 Principles

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

3.4.5 Types

Most centrifuges rotate thanks to some kind of motor drive. The types of centrifuge used for sedimentation include:

- hydro cyclone
- tubular bowl
- chamber bowl
- imperforate basket
- disk stack separator
- decanter

Sedimenting centrifuges were invented for liquid solid separation and not for handling solids. It soon became apparent that these machines had wider applications, which would involve the presence of solid impurities, leading to use for separating solids from liquids.

3.4.5.1 Hydrocyclone

The simplest device to use centrifugal force to achieve separation is the hydrocyclone. It not really a centrifuge: the centrifugal separation is produced by the motion of the slurry, induced by the tangential introduction of the feed material. Its principle of operation is based on the concept of the terminal settling velocity of a solid particle in a centrifugal field. The following picture describes the conditions in an operating hydrocyclone. The feed enters tangentially into the cylindrical section of the hydrocyclone and follows a circulating path with a net inward flow of fluid from the outside to the vortex finder on the axis. The centrifugal field generated by the high circulating velocities creates an air core on the axis that usually extends on the spigot opening at the bottom of the conical section through the vortex finder to the overflow at the top. In order for this to occur the centrifugal force field must be several times larger than the gravitational one. Particles that experience this centrifugal field will tend to move outwards relative to the carrier fluid because of their relatively greater density. The larger, heavier particles will migrate rapidly to the outside walls of the cylindrical section and will then be forced to move downward to the inside of the conical wall. Small particles will, on the other hand, be dragged inward by the fluid as it moves towards the vortex finder. The solid separation occurs in the passage of the suspension along the barrel of the hydrocyclone, to form thickened slurry at the outer wall, which than leaves the hydrocyclone as a continuous stream from its discharge nozzle.

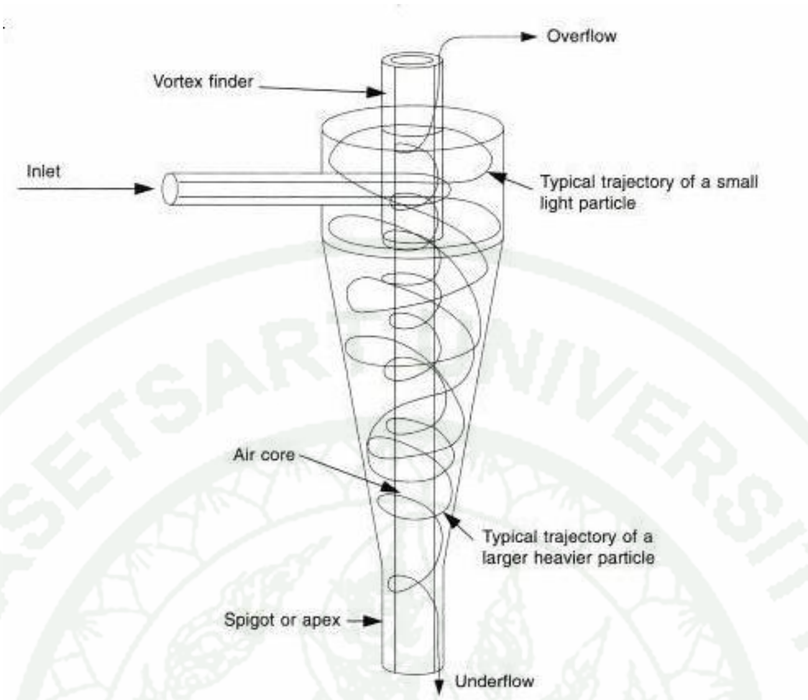


Figure 12 Hydrocyclone

Source: Lenntech (2009)

3.4.5.2 Tubular bowl centrifuge

The tubular bowl centrifuge has been used for longer than most other designs of centrifuge. It is based on a very simple geometry: it is formed by a tube, of length several times its diameter, rotating between bearings at each end. The process stream enters at the bottom of the centrifuge and high centrifugal forces act to separate out the solids. The bulk of the solids will adhere on the walls of the bowl, while the liquid phase exits at the top of the centrifuge.

As this type of system lacks a provision of solids rejection, the solids can only be removed by stopping the machine, dismantling it and scraping or flushing the solids out manually.

Tubular bowl centrifuges have dewatering capacity, but limited solids capacity. Foaming can be a problem unless the system includes special skimming or centripetal pumps.

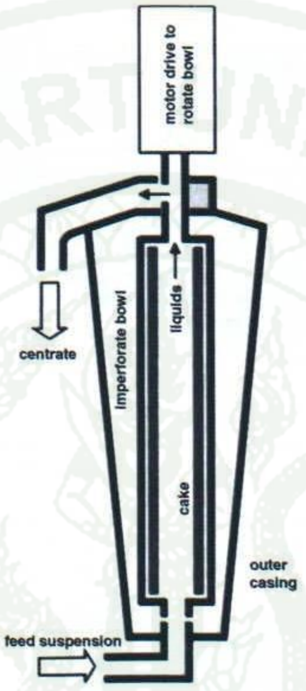


Figure 13 Tubular bowl centrifuge

Source: Lenntech (2009)

3.4.5.3 Chamber bowl centrifuge

The chamber bowl centrifuge is a number of tubular bowl arranged co-axially. It has a main bowl containing cylindrical inserts that dived the volume of the bowl into a series of annular chambers, which operate in series. Feed enters the center of the bowl and the suspension passes through each chamber in turn, at increasing distances from the axis. The solids settle onto the outer wall of each chamber and the clarified liquid emerges as an overflow from the largest diameter chamber. This device provides also a classification of the suspended solids: the coarse particles deposit in the inner chamber and the increasingly fine particle deposit on the subsequent chambers. The removal of sedimented solids requires the stopping of rotation for manual cleaning.

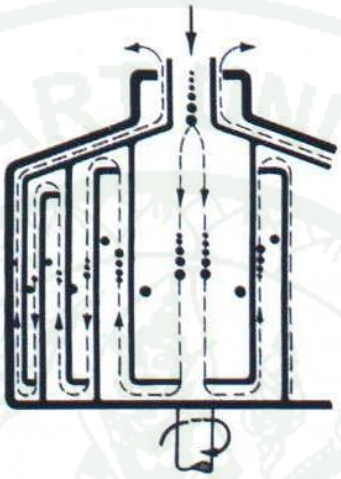


Figure 14 Chamber bowl centrifuge

Source: Lenntech (2009)

3.4.5.4 Imperforate basket centrifuge

The imperforate basket centrifuge is used when if the solid content of the suspension is higher. It consists of a simple drum-shaped basket or bowl, usually rotating around a vertical axis. The solids accumulate and compress as effect of the centrifugal force, but they are not dewatered. The residual liquid is drained out when the rotation of the bowl is stopped. The layer of solids is removed manually by scraping or shoveling. Unloading can be achieved semi automatically first by use of a skimmer pipe to remove the residual liquid and then by lowering a knife blade into the solid and so cutting it out from the bowl. This allows avoiding the switching off of the machine.

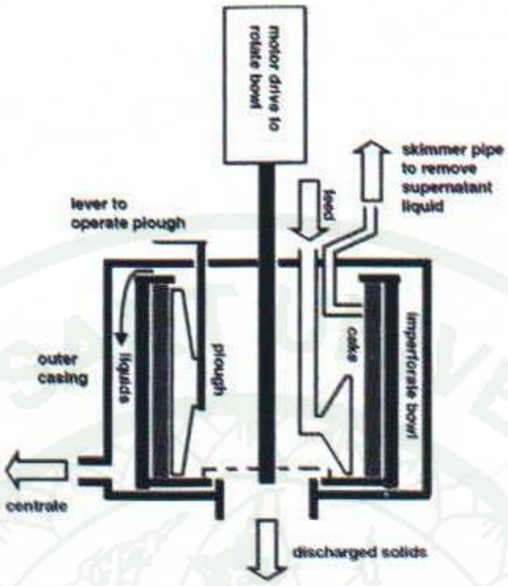


Figure 15 Imperforate basket centrifuge

Source: Lenntech (2009)

3.4.5.5 Disk stack separator

The simplest design is a closed bowl, containing the disk stack, with any solids present collecting at the outer part of the bowl, from which they have to be removed manually after stopping rotation. The solids are discharged from the bowl by a number of methods, including the basic use of nozzles, which are open continuously, allowing a thick slurry to discharge. In the more complicated design valved nozzles open automatically when the solid depth in the bowl reaches a certain value, and then close again when most of the solids have been discharged. In the most complicated design the bowl is opened: its shell splits circumferentially for a short period, with the opening also controlled by solids depth in the bowl.

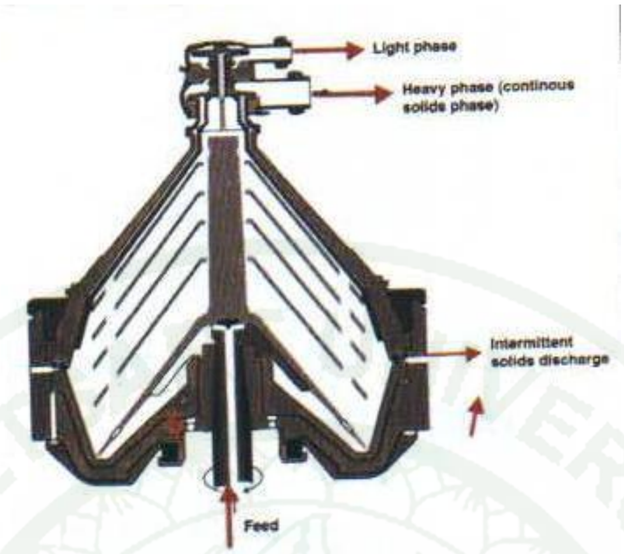


Figure 16 Disk stack separator

Source: Lenntech (2009)

3.4.5.6 Decanter

The decanter centrifuge is the only sedimentation centrifuge designed from the start to handle significant solid concentration in the feed suspension. At the same time it can achieve quite good degrees of clarification of the liquid concentrate. Although a complicated piece of machinery it embodies a simple principle. They consist basically of a horizontal cylindrical bowl (1) rotating at a high speed, with a helical extraction screw (2) placed coaxially. The screw perfectly fits the internal contour of the bowl, only allowing clearance between the bowl and the scroll. The differential speed between screw and scroll provides the conveying motion to collect and remove the solids, which accumulate at the bowl wall.

1. Cylindroconical bowl
2. Helical extraction screw (scroll)
3. Feed
4. Distributor

5. Ring space
6. Settled product
7. Liquid level
8. Drying zone
9. Clarified liquid
10. Adjustable thresholds

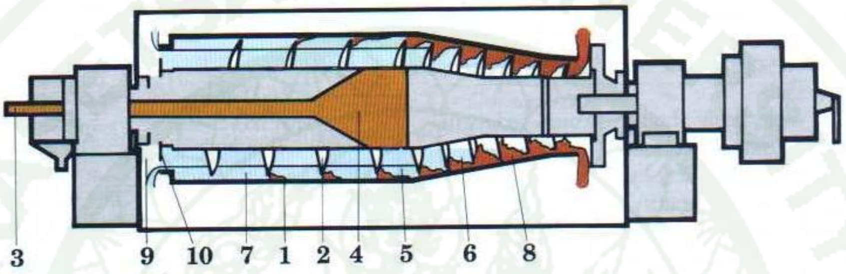


Figure 17 Decanter

Source: Lenntech (2009)

The product to be treated (3) is introduced axially into the unit by appropriate distributor (4). It is propelled into the ring space (5) formed by the internal surface of the bowl and the body of the scroll. The separation process basically takes place inside the cylindrical section of the bowl. The relative velocity of the scroll pushes the settled product (6) along into the bowl. The conveyance of the solids into the length of the cone enables the sediment to pass out of the clarified liquid phase. As the feed is continuous a liquid level (7) is established in the unit following a cylindrical surface that constitute the internal surface of the liquid ring. Once the solids have passed out of the liquid ring the remaining section of the cone all the way up to the ejector provide the final draining: this section is known as the drying zone (8). The clarified liquid (9) is collected at the other end of the bowl by flowing through the adjustable threshold (10), which restrict the liquid ring of the unit. A cover that enables the clarified liquid as well as the sediments to be collected protects the rotor.

The decanter operates mainly by sedimentation a process causing the separation of suspended solids by virtue of their higher density than the liquid in which they are suspended. If the density difference is high than gravity may provide sufficient driving force for the separation to occur in a reasonable time. If the density difference is small, or the particle size is very small, than gravity separation would take too long and the separation force must be increased by the imposition of centrifugal forces many times that of gravity alone.

3.4.6 Advantages

The prime beneficial characteristic of the decanter is its ability to remove separated solids from the separation zone on a fully continuous basis.

By comparison with:

- Gravity sedimentation: the decanter can achieve separations that would be very difficult in a clarifier or lamella separator, and it produces drier solids.
- Hydrocyclones: the decanter has a much higher liquid capacity, can handle much higher slurry concentrations, and producer much drier solids.
- Tubular bowl centrifuges: the decanter offers higher capacities, the ability to handle concentrate slurries, and continuous operation.
- Imperforate basket centrifuges: the decanter operates continuously, can handle much higher solids concentrations, and produces much drier solids.
- Disk stack centrifuge: the decanter is truly continuous in operation, can handle much higher solids concentrations in the feed slurry and produces drier solids.

The advantages of the decanter are its wide range of potential use, coupled with its continuous operation, its ability to accept a wide range of feed concentrations, and its availability in a wide range of feed capacities.

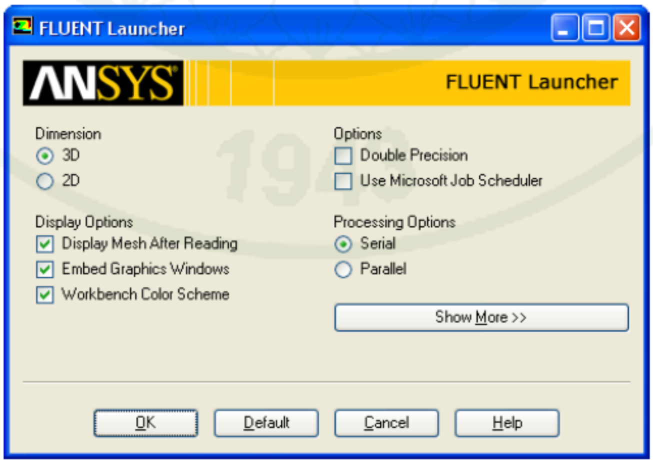
3.4.7 Applications

The decanter centrifuge can be used for most types of liquid/solid separation. It can be used for the classification of solids in liquid suspension or for the clarification of liquids. It can also be used in the recovery of a valuable solid from its suspension in the liquid and it can wash the recovered solid from its mother liquor. The decanter can also dewater slurries to a high level of dryness and it can finally be operated so as to act as a thickener, producing clear liquid and more concentrated slurry.

4. Computation Fluid Dynamic (CFD)

4.1 Starting ANSYS FLUENT Using FLUENT Launcher

Whether you start ANSYS FLUENT either from the Linux/UNIX or Windows command line with no arguments, from the Windows Programs menu, or from the Windows desktop, FLUENT Launcher will appear, where you can specify the dimensionality of the problem (2D or 3D), as well as other options (e.g., whether you want a single-precision or double-precision calculation):



Dimension
● 3D
○ 2D

Options
☐ Double Precision
☐ Use Microsoft Job Scheduler

Display Options
☑ Display Mesh After Reading
☑ Embed Graphics Windows
☑ Workbench Color Scheme

Processing Options
● Serial
○ Parallel

Figure 18 FLUENT Launcher

Under Dimension, select 3D for the three-dimensional solver, or select 2D for the twodimensional solver.

The Display Options allow you to make decisions related to the graphics windows:

- You can choose to have ANSYS FLUENT automatically display the mesh immediately after reading a mesh or case file by using the Display Mesh After Reading option (enabled by default). All of the boundary zones will be displayed, except for the interior zones of 3D geometries. Note that your decision regarding this option can be overridden after you have launched ANSYS FLUENT: simply change the status of the Display Mesh After Reading option in the Select File dialog box that opens when you are reading in the mesh or case file.

- You can choose to have the graphics windows embedded within the ANSYS FLUENT application window by using the Embed Graphics Windows option (enabled by default), rather than having floating graphics windows.

- You can choose to use the default Workbench Color Scheme in the graphics windows(i.e., a blue background), rather than the classic black background.

4.2 Steps for Using a Multiphase Model

The procedure for setting up and solving a general multiphase problem is outlined below, and described in detail in the subsections that follow. Remember that only the steps that are pertinent to general multiphase calculations are shown here. For information about inputs related to other models that you are using in conjunction with the multiphase model, see the appropriate sections for those models.

1. Enable the multiphase model you want to use (VOF, mixture, or Eulerian) and specify the number of phases. For the VOF and Eulerian models, specify the volume fraction scheme as well.

Models → Multiphase → Edit...

2. Copy the material representing each phase from the materials database.

Materials

3. Define the phases, and specify any interaction between them (e.g., surface tension if you are using the VOF model, slip velocity functions if you are using the mixture model, or drag functions if you are using the Eulerian model).

Phases

4. (Eulerian model only) If the flow is turbulent, define the multiphase turbulence model.

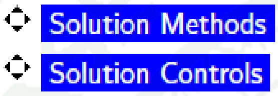
Models → Viscous → Edit...

5. If body forces are present, enable gravity and specify the gravitational acceleration.

Cell Zone Conditions → Operating Conditions...

6. Specify the boundary conditions, including the secondary-phase volume fractions at flow boundaries and (if you are modeling wall adhesion in a VOF simulation) the contact angles at walls.

7. Set any model-specific solution parameters.



8. Initialize the solution and set the initial volume fractions for the secondary phases.

9. Calculate a solution and examine the results. Postprocessing and reporting of results are available for each phase that is selected. (Fluent, 2010)

MATERIALS AND METHODS

Materials

1. Raw Material
    - Keson Refined palm oil
    - Potassium hydroxide
    - Methanol
2. Hardware
    - Ultrasonic with the frequency of 28 kHz and capacity 3 L
    - Heating system with the power of 3000 watt
    - Stirrer and motor for stirring at 600 rpm
    - Valves
    - Thermocouples type K
    - Personal computer CPU (Intel(R) Pentium(R) D CPU 3.40 GHz (2CPUs))
    - Four pumps for feed and hot water
    - Flow meters
    - Temperature controllers
    - Microwave with the power of 800 watt
    - Disk stack separator with the power of 120 watt
    - Gas Chromatography
3. Soft wares
    - Software Fluent 12.1
    - Software SolidWorks
    - Microsoft Window XP

Methods

1. Design Procedures

1.1 The static mixer

The kenics static mixer was selected to study in biodiesel production because of the low pressure drop and easy in designing. The mixer elements were calculated and adjusted width, length and thickness of the mixer element to be 2.54, 7.62 and 0.15 centimeters for using in this work.

1.2 The continuous washing equipment

The equipment for purifying biodiesel is using water washing in this work because of the low cost to build equipment and easy designing. The equipments were calculated and adjusted sizing into two parts; spray water part has 10.16 centimeters diameter and 78 centimeters length, and setting tank part has 15.24 centimeters diameter and 1329 centimeters length

1.3 The developing the model

After the size of the static mixer and equipment for washed biodiesel were designed, the models of them were developed in the SolidWorks program. The triangle grids were then generated in the model using WorkBench program in order to use for finite element calculation. Furthermore, the boundary conditions, the mass flow inlet, mass flow outlet and wall, and equilibrium drop diameter were input in the program.

1.4 Analyzing model

The model with grid and boundary conditions was analyzed in order to predict the flow pattern and distribution of the fluid using Fluent program. In this program, the behavior of flow, laminar flow, mass flow rate and properties of each component were specified. The calculation for designing was shown in Appendix C. After the program compiled, the results were shown in the color contours.

2. Experiments

The experiments were divided into two parts which are the experiment of the transesterification reaction and purification biodiesel process. The potassium hydroxide was used as catalyst. The ratio of 6:1 methanol to oil ratio and 1% wt potassium hydroxide were used in the experiments. The palm oil was preheated at 70°C to its reaction temperature before the mixing of methanol and potassium hydroxide.

2.1 The conventional stirred tank

The impeller with heater was used to mix between the palm oil and the methoxide. The reaction temperature was maintained at 60°C.

2.2 The static mixer

The static mixer was used to mix between the palm oil and the methoxide, the flow rate at 3 L/min was fed into the static mixer and repeating pump in the static mixer for 1 hour.

2.3 The microwave

The experiment of microwave was similar to the experiment of static mixer.

2.4 The ultrasonic

The experiment of ultrasonic was similar to the experiment of static mixer.

2.5 The conventional stirred tank with static mixer

The conventional stirred tank was used to mix between the palm oil and the methoxide about 5 minute before pumping into the static mixer with the flow rate of 3 L/min.

2.6 The static mixer with microwave and ultrasonic

The static mixer, microwave and ultrasonic were connected in series. The palm oil was preheated at 70°C then the palm oil and methoxide were fed continuously to the static mixer with 6:1 mthoxide to oil ratio. The flow rate at 3 L/min was fed through the static mixer, the microwave and ultrasonic.

2.7 Separate and purification

After reaction finished, biodiesel was sent into the centrifugal separator with the flow rate 1.5 L/min. The biodiesel was separated from glycerol and sent to the equipment for washing biodiesel. And the flow rates of biodiesel and water which has 1:1 biodiesel to water ratio were varied along 1.5, 3.0 L/min and 4.5 L/min to find the optimal flow rate which gave the maximum purity of biodiesel of the equipment for wash biodiesel.

2.8 Keeping the sample

The experiments were carried out for one hour by sampling at the bottom valve of the reactor when the reaction time was 5, 10, 15, 30 and 60 minutes.

2.10 Analyzing the purity of biodiesel

1. The biodiesel was weighed around 50 milligram in the vial for analyzing %FAME.
2. Adding the one-milliliter heptadecanoate and shaking by vortex mixer for one minute.
3. Analyzing this solution by gas chromatography.
4. Calculating the biodiesel's %FAME from the peak area of gas chromatography as shown in Appendix C.

RESULTS AND DISCUSSION

This result section is divided into two main parts. The first part covers a design of the static mixer and equipment for washing biodiesel by computational fluid dynamics, and the second part extends the results of experiment from all equipments.

1. Design equipment

1.1 Static mixer

The computational fluid dynamics (CFD) was used to evaluate the mixing efficiency of the kenics static mixer**.** The distribution of methanol into the palm oil could be observed from the volume fraction of palm oil where the blue and red spots were the feed methanol and palm oil, respectively. Therefore, at the beginning the volume fraction of palm oil and methanol were equaled to 1 and 0. After the reactants were flowed through the static mixer, the methanol was more distributed into the palm oil. Figure 19, showed the volume fraction of palm oil and methanol were mixing inside the static mixer.

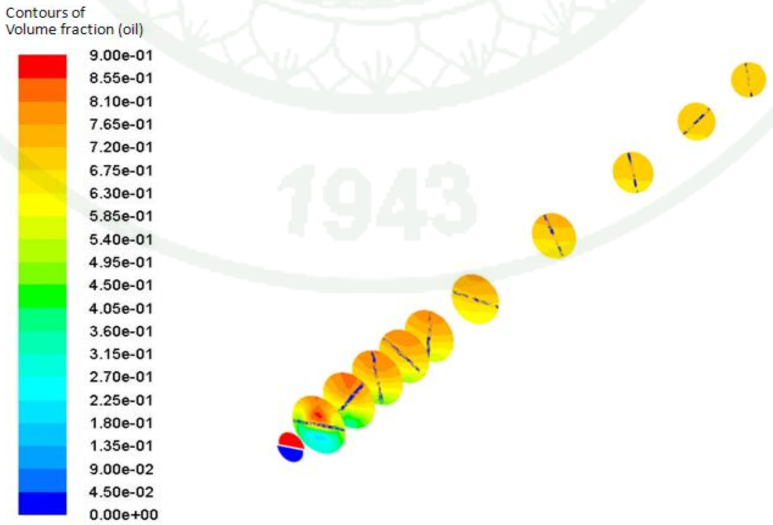


Figure 19 The simulation of static mixer from using CFD

This static mixer was designed to have 7 mixer elements. The mixer elements were calculated and adjusted width, length and thickness of the mixer element to be 2.28, 7.62 and 0.15 centimeter for using in this work as shown in Figure 20. Then three parts of a static mixer were connected in series. Reactants were fed into top of the first static mixer. Then, the mixture flowed into the second static mixer and the third static mixer, respectively. And the mixture flowed out at the bottom of the third static mixer as shown in Figure 21.

Figure 20 Dimension of static mixer (millimeter)

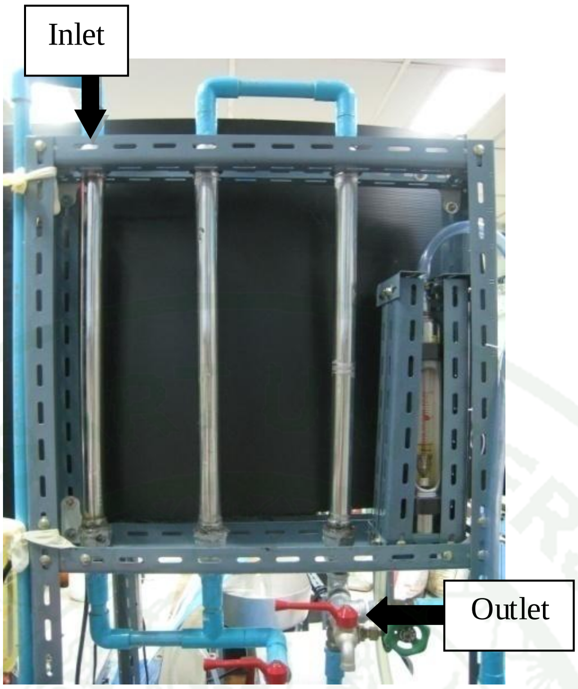


Figure 21 Three parts of a static mixer connected in series

1.2 Continuous equipment for washing biodiesel

The computational fluid dynamics (CFD) was to evaluate the mixing efficiency of the equipment for washing biodiesel. The distribution of water into the biodiesel can be observed from the volume fraction of biodiesel where the blue and red spots were the feed water and biodiesel, respectively. Therefore, at the beginning the volume fraction of biodiesel and water were equaled to 1 and 0. After water and biodiesel passed through the tube for washing part, the water was sprayed into the biodiesel. Then biodiesel was mixed with water and flowed into the setting tank. After that the biodiesel was separated from water and overflowed into the next washing part again as shown in Figure 22.

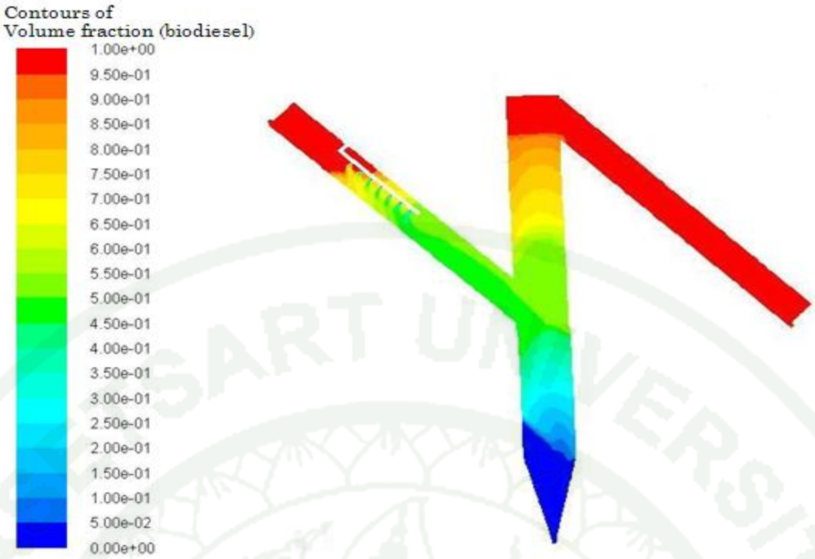


Figure 22 The simulation of equipment for washing biodiesel from using

Therefore, the tube for spraying water inside washing part has 30.6 centimeter length and 1.27 centimeter diameter as shown in Figure 23. The outside washing part has 78 centimeter lengths and 10.16 centimeter diameters. The setting tank has 132.9 centimeter, length and 15.24 centimeter diameter were used in this work as shown in Figure 24.

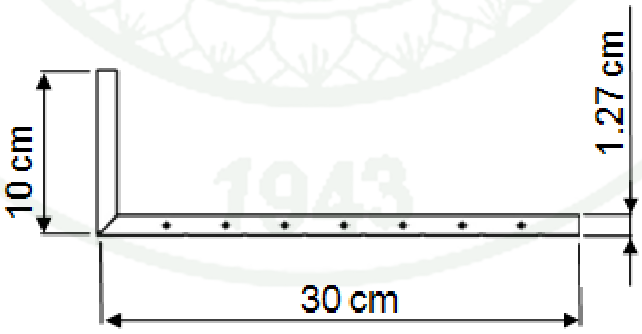


Figure 23 Dimension of tube for spray water

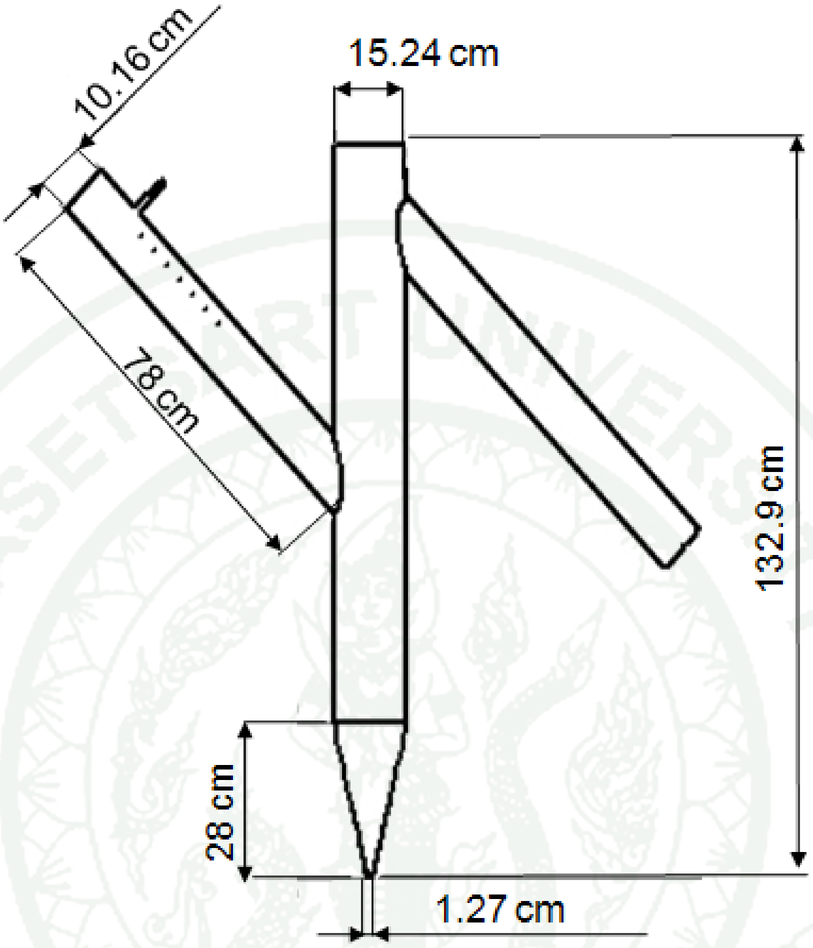


Figure 24 Dimension of Equipment for washing Biodiesel

Figure 25, showed that the real size of tube for spraying water inside washing part, and also three parts of a equipment for washing biodiesel connected in series were shown in Figure 26.

Figure 25 Tube for spray water inside of washing part

Figure 26 Equipment for washing biodiesel

2. The Results of Experiment

2.1 Conventional stirred tank

The conventional stirred tank with heater was used to mix the reactants. %FAME increased according to time and was equal to 94% after 60 minutes as shown in Figure 27.

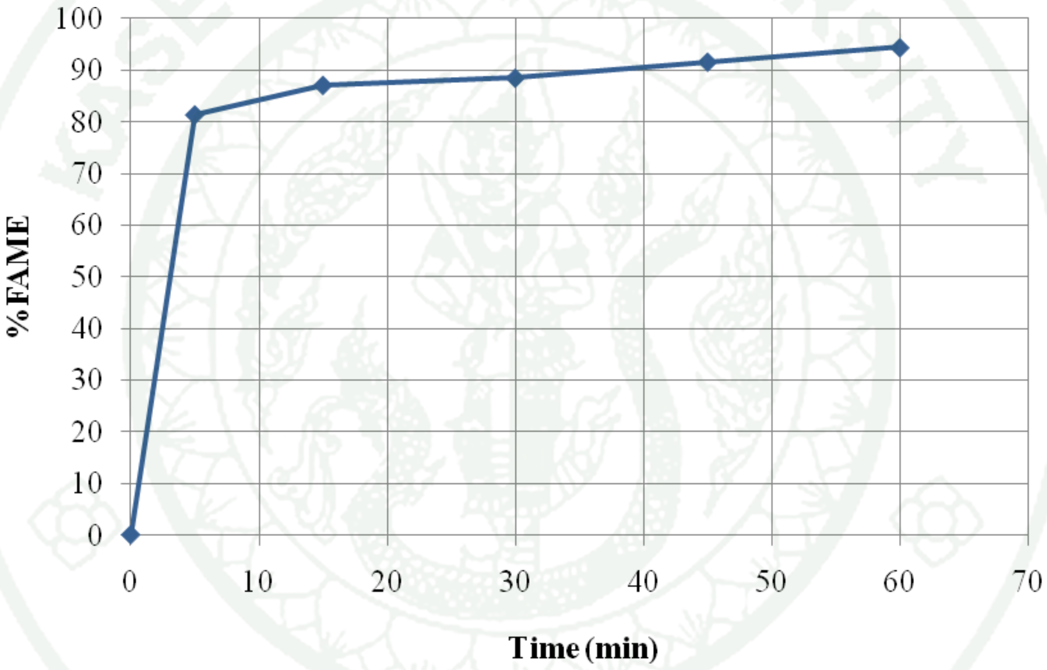


Figure 27 The results of batch process from conventional stirred tank

2.2 Static mixer

The static mixer was used to mix the reactants. In this experiment, the flow rate of 3 L/min was used. %FAME increased according to time and was equal to 92% after 60 minutes reaction time as shown in Figure 28.

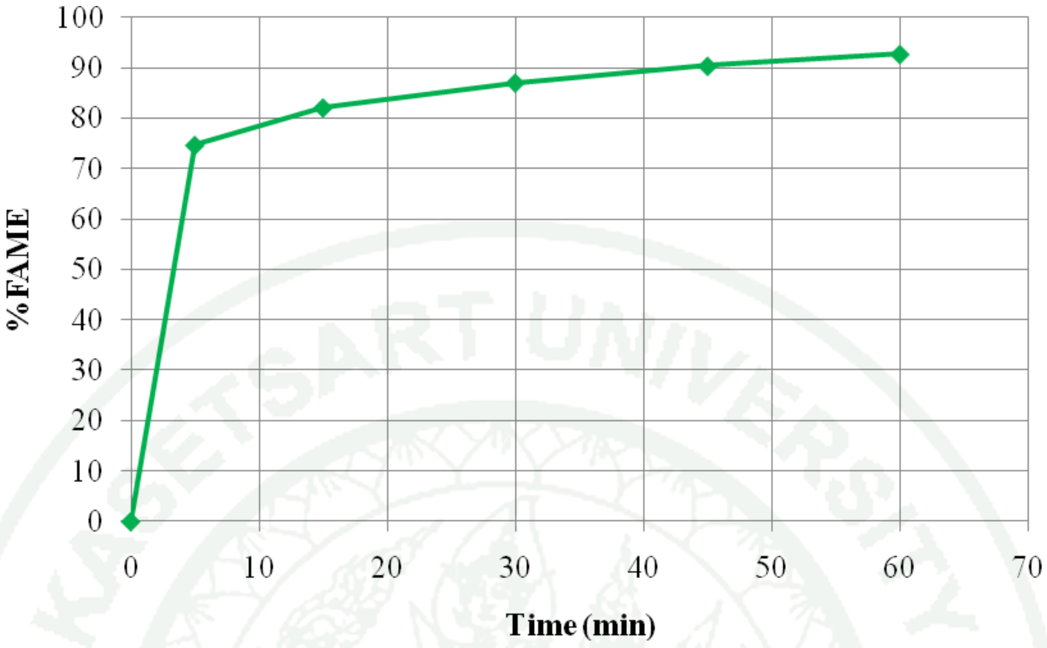


Figure 28 The results of batch process from static mixer

2.3 Microwave

The microwave was used to mix the reactants. In this experiment, the flow rate of 3 L/min was used. %FAME increased according to time and was equal to 91% after 60 minutes as shown in Figure 29.

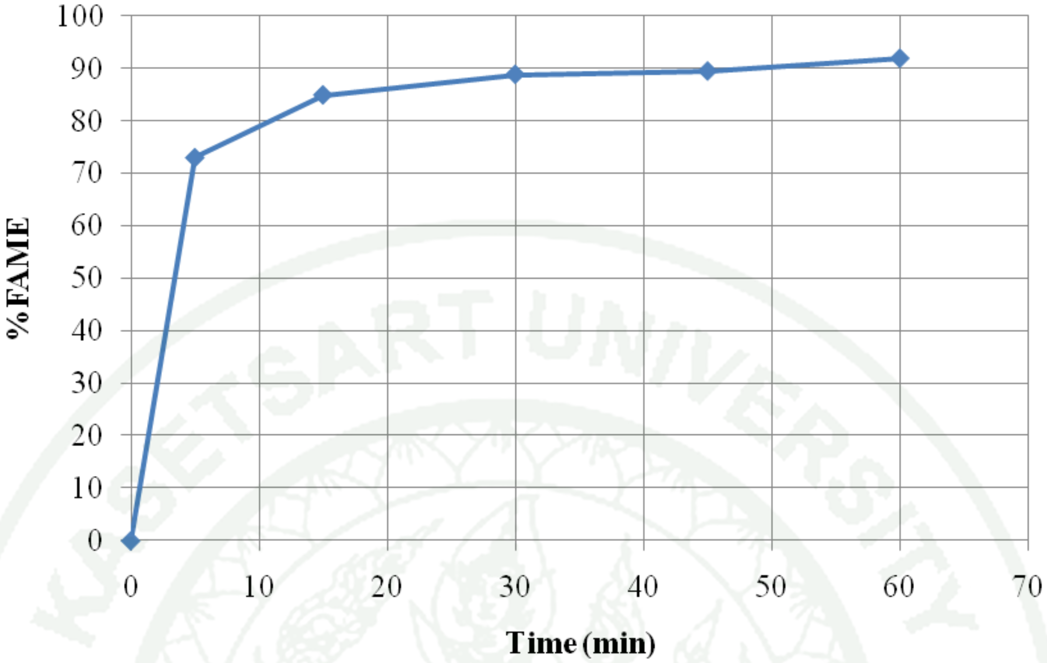


Figure 29 The results of batch process from microwave

2.4 Ultrasonic

The ultrasonic was used to mix the reactants. In this experiment, the flow rate of 3 L/min was used. %FAME increased according to time and was equal to 91% after 60 minutes as shown in Figure 30.

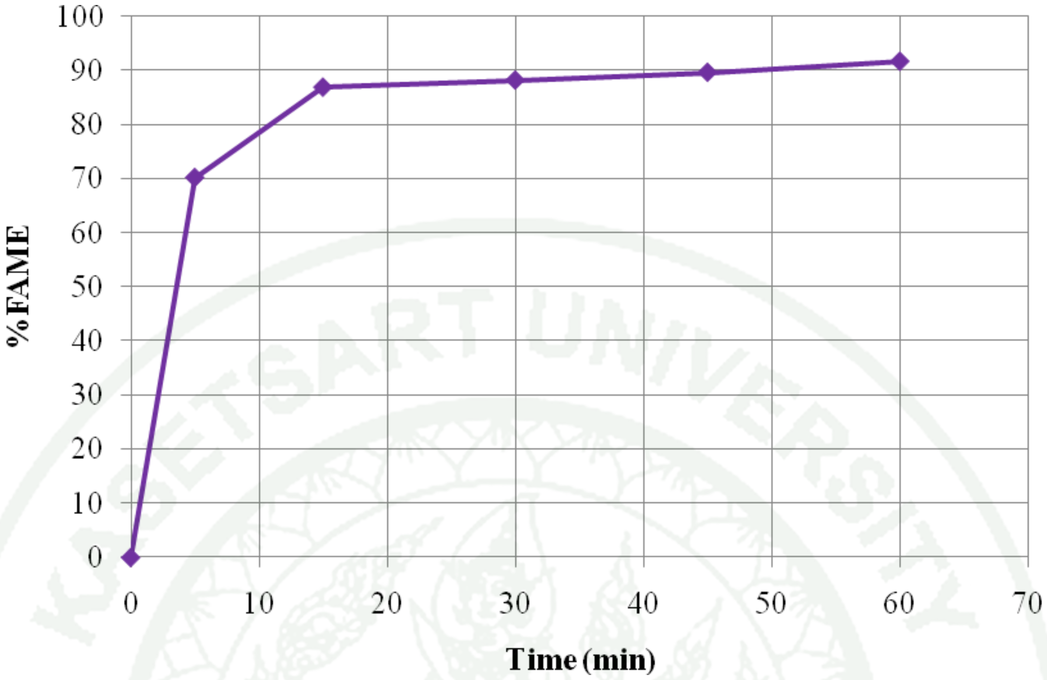


Figure 30 The results of batch process from ultrasonic

2.5 The conventional stirring tank with static mixer

The conventional stirred tank and static mixer were used together. The reactants were stirred in the conventional stirred tank by the impeller about 5 minute before flowed through the static mixer. The results showed that %FAME increased about 81% when biodiesel flowed out from the conventional stirring tank then %FAME increased to 85.9% when biodiesel flowed through the static mixer as shown in Figure 31.

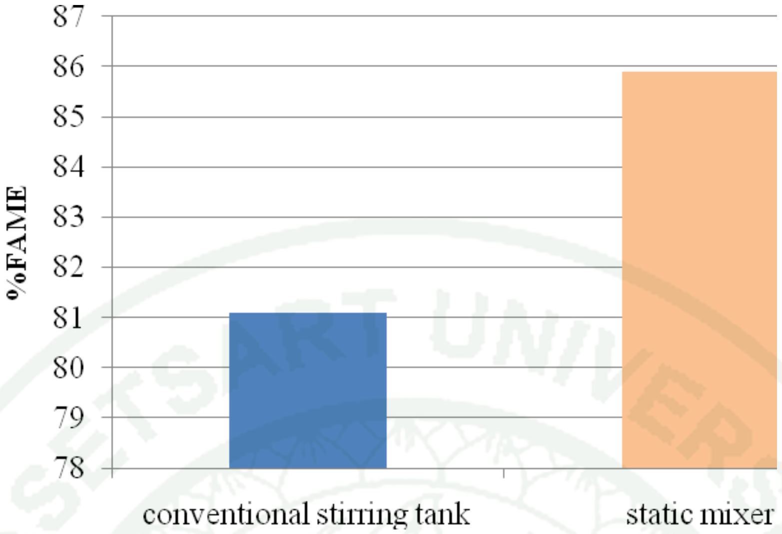


Figure 31 The results of conventional stirring tank with static mixer

2.6 The static mixer with microwave and ultrasonic

The static mixer, microwave and ultrasonic was connected respectively. The palm oil was preheated at 70°C then the palm oil and methoxide were fed continuously to the static mixer with 6:1 mthoxide to oil ratio. The result showed that %FAME increased about 48% when the mixture flowed out from static mixer then %FAME increased to 69% when the mixture flowed through the microwave and ultrasonic as shown in Figure 32.

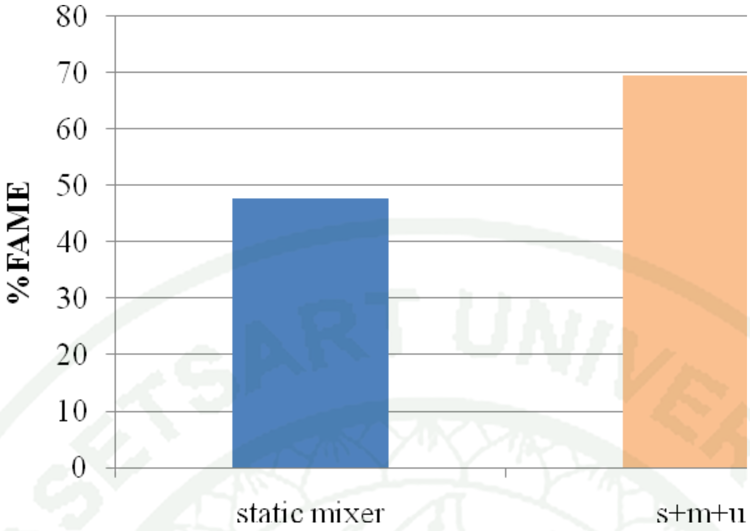


Figure 32 The results of static mixer microwave and ultrasonic

2.7 Comparison between the equipments

When the results of the conventional stirring tank, static mixer, microwave and ultrasonic were compared at the same time, after 60 minutes. The conventional stirring tank had the highest %FAME because the heater was used in the conventional stirring tank. Next, the static mixer, microwave and ultrasonic, respectively as show in Figure 33. However, when the results of the conventional stirring tank, static mixer, microwave and ultrasonic were compared in terms of %FAME/Energy at the same time, after 60 minutes. The static mixer had the highest %FAME/Energy next ultrasonic, microwave and the conventional stirring tank, respectively as show in Figure 34.

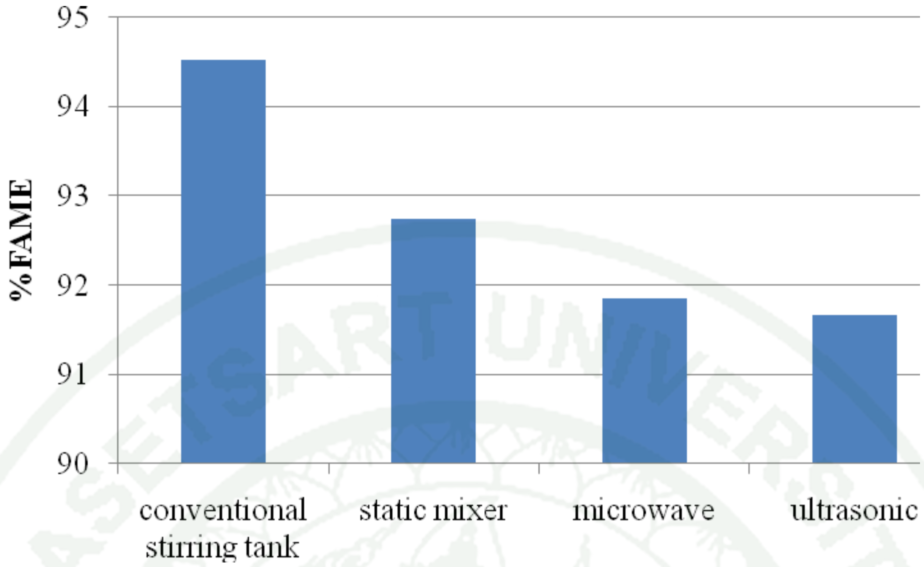


Figure 33 Comparison of %FAME for each equipment at 60 min

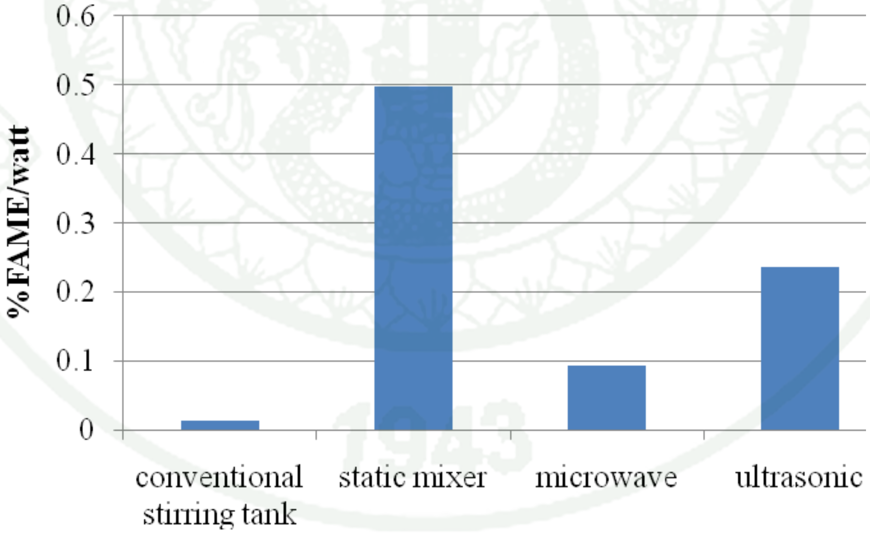


Figure 34 Comparison of %FAME/Energy for each equipment at 60 min

2.8 The separator

The biodiesel was separated from glycerol by using the centrifugal separator. When the biodiesel was observe with the naked eye, the result shown that the biodiesel did not have glycerol. Therefor, the centrifugal separator could separate between biodiesel and glycerol as shown in Figure 35.

Figure 35 Products after flow through separator

2.9 The result of equipment for washing biodiesel

The flow rate was the factor that effected on the purity of biodiesel. The flow rate of experiments was varied at 1.5, 3.0 and 4.5 L/min respectively. The Table 6 shown that the results of soap content at flow rate 1.5, 3.0 and 4.5 L/min were constant 0.3 wt % at amount of washing 3, 4 and 5 times, respectively. Therefore, the flow rate and times of washing biodiesel did not have influence with % soap.

Table 6 The results of soap content in biodiesel (mg/kg)

Number	**1.5 L/min**	**3.0 L/min**	**4.5L/min**
Before	240.9	240.9	240.9
3	4.87	4.83	4.78
4	4.79	4.75	4.72
5	4.74	4.72	4.68

Table 7 showed the result of alkali content in the biodiesel. The flow rates of experiments were varied at 1.5, 3.0 and 4.5 L/min at the amount of washing 3, 4 and 5 times, respectively. The results showed that every washing time at 1.5, 3.0 and 4.5 L/min of the flow rate did not have alkali in biodiesel. Therefore, the flow rate and the washing times did not effected with purity of biodiesel.

Table 7 The results of alkali content in biodiesel (mg KOH/kg)

Number	**1.5 L/min**	**3.0 L/min**	**4.5L/min**
before	200	200	200
3	0	0	0
4	0	0	0
5	0	0	0

Table 8 showed the result of total glycerol contain in the biodiesel. The flowrate of experiments were varied at 1.5, 3.0 and 4.5 L/min at the amount of washing 3, 4 and 5 times, respectively. The results showed that every washing time at 1.5, 3.0 and 4.5 L/min of the flow rate were remained the standard is not over 0.25 wt %. Therefore, the flow rate and the washing times did not effected with purity of biodiesel.

Table 8 The result of total glycerol contain in biodiesel (wt%)

Number	**1.5 L/min**	**3.0 L/min**	**4.5L/min**
before	3.34	3.34	3.34
3	0	0	0
4	0	0.083	0
5	0.083	0	0

All results showed that the flow rate did not affect the purity of biodiesel and the times for washing were 3 times.

CONCLUSION AND RECOMMENDATION

Conclusion

The production of biodiesel was carried out by using four different equipments namely: the conventional stirred tank, the microwave, the ultrasonic and the static mixer. The static mixer used in this work was designed using CFD. The designed static mixer had length of 53.34 centimeters, diameter of 2.28 centimeters and 7 mixer elements were used in this work. For the experiments of the biodiesel production by the conventional stirred tank the temperature was controlled at 60 °C, the microwave, the ultrasonic and the static mixer were used the flow rate at 3 L/min. When the results from those four equipments were compared, it showed that maximum %FAME was obtained from the conventional stirred tank because of fed heating energy. However when the results from that equipment were compared in terms of %FAME/Energy, it showed that maximum %FAME/energy was obtained from the static mixer the next was ultrasonic, microwave and the conventional stirred tank, respectively. There it can be concluded that the static mixer, the microwave and the ultrasonic can be used instead of the conventional stirred tank. But the microwave and the ultrasonic had capacity smaller and used energy more than the static mixer. Therefore, the static mixer was optimal for continuous biodiesel production.

New design of washing process of biodiesel can use only water. The equipment for washing biodiesel was designed using CFD. The designed equipment for washing biodiesel had the outside washing part has 78 centimeter lengths and 10.16 centimeter diameters, and the setting tank has 132.9 centimeter, length and 15.24 centimeter diameter were used in this work. For the experiments of washing the biodiesel process, the flow rates of biodiesel and water which has 1:1 biodiesel to water ratio were varied to be 1.5, 3.0 and 4.5 L/min, and the times of wash were varied to be 3, 4 and 5 times. The results showed that the triples washing apparatus in series yielded an acceptable result with the optimal flow rate 4.5 L/min of water.

Recommendation

1. Should increase the retention time in the microwave and ultrasonic to increase %FAME.

2. Washing equipment should study of diameter and length for spray water into biodiesel.

3. Washing equipment should study of angle and length for mixing tube.

4. Washing equipment should study of diameter for settling tank to separate between biodiesel and glycerol.

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APPENDICES

Appendix A

Equipment part

A.1 Microwave

Microwave with the power of 800 watt which has Teflon tube to allow the components to flow through to accelerate the transesterification reaction is shown in Figure A1.

Appendix Figure A1 Microwave

A.2 Ultrasonic

The ultrasonic apparatus which has 3.645 liter in volume, 28 hertz in frequency and can adjust the power as shown in Figure 2.6 is used to produce the ultrasonic wave.

Appendix Figure A2 Ultrasonic.

A.3 Heater and Heating Tank

Heater installed inside the heating tank. For used to heat oil up before that oil was fed into the reactor.

Appendix Figure A3 Heater (left) and Heating tank (right)

A.4 Methanol tank

The tank was used to mix between methanol and potassium hydroxide before the mixture was fed into reactor as show in Figure A.5.

Appendix Figure A4 Methanol tank

A.5 Reactor

The one hundred-liter reactor has the impeller to mix the reactants that will flow through the bottom of the reactor before are pumped to other equipments.

Appendix Figure A5 Conventional stirred tank reactor

A.6 Centrifuge Separator

The Disk stack separator (10,050 rpm) was used to separate between biodiesel and glycerol as show in Figure A.7.

Appendix Figure A6 Disk stack separator

A.7 Flow Meter

The flow meter was used to measure the flow rate of the components in the process. In this work, the flow meter can measure the flow rate in the range of 0 to 250 liter per hour as shown in Figure A8. It consists of a tapered tube with a float inside that is pushed up by flow and pulled down by gravity. At a higher flow rate more area is needed to accommodate the flow, so the float rises.

Appendix Figure A7 Flow meter.

Appendix B

Design Part

B.1 Designing of Kenics Static Mixer

In this designing, the size of static mixer was scaled up from Andre *et al.*'s study which has the width, length and thickness of the mixer element to be 2 centimeter, 3 centimeter and 0.08 centimeter, respectively. The desired diameter of tube was 2.54 centimeter, thus the width of mixer element was 2.28 centimeter.

$$Ratio\ between\ new\ width\ and\ old\ width = \frac{2.28}{2}$$

$$= 1.14$$

$$\therefore Length\ of\ mixer\ element = 1.14 \times 3$$

$$= 3.42 \quad cm.$$

The thickness of mixer element was 0.04 times of its width

$$\therefore Thickness\ of\ mixer\ element = 0.04 \times 2.28$$

$$= 0.09 \quad cm.$$

B.2 Calculation of Volume Faction

The biodiesel's reaction was carried out in the presence of 6:1 alcohol to oil ratio. Therefor, in the mixture has 7 mole ratios.

The molecular weight of palm oil (MW_p) = 702 g/mol
The molecular weight of methanol (MW_m) = 32 g/mol
The density of palm oil at 60°C (ρ_p) = 870.2 kg/m^3
The density of methanol (ρ_m) = 791.8 kg/m^3

$\therefore$ Volume faction of palm oil

$$= \frac{1kmol \times MW_p}{(1kmol_p \times MW_p) + (6kmol_m \times MW_m)}$$

$$= \frac{1kmol \times 702\,kg/kmol}{(1kmol_p \times 702\,kg/kmol) \times (6kmol_m \times 32\,kg/kmol)}$$

$$= \frac{702kg \div \rho_p}{(702kg \div \rho_p) + (192kg \div \rho_m)}$$

$$= \frac{702kg \div 870.2kg/m^3}{(702kg \div 870.2kg/m^3) + (192kg \div 791.8kg/m^3)}$$

$$= 0.76888461$$

$\therefore$ Volume faction of methanol

$$= 1 - 0.76888461$$

$$= 0.231111593$$

B.3 Calculation of Mass Flow Rate

The maximum flow rate of pump is 37 L/min

$$Mass\ flow\ rate\ of\ palm\ oil\ (\dot{m}_p) = X_p \times \rho_p \times Q$$

$$Ratio\ of\ palm\ oil\ in\ fluid\ (X_p) = 0.7688461$$

$$Density\ of\ palm\ oil\ (\rho_p) = 870.2 \quad kg/m^3$$

$$\dot{m}_p = 0.76888461 \times 870.2\frac{kg}{m^3} \times 37\frac{L}{\min} \times \frac{1m^3}{1000\,L} \times \frac{1\min}{60\,s}$$

$$= 0.41kg/s$$

$$\textit{Mass flow rate of methanol}\ (\dot{m}_m) = X_m \times \rho_m \times Q$$

$$\textit{Ratio of methanol in fluid}\ (X_m) = 0.231111539$$

$$\textit{Density of methanol}\ (\rho_m) = 791.8 \quad kg/m^3$$

$$\dot{m}_m = 0.231111593 \times 791.8\frac{kg}{m^3} \times 37\frac{L}{\min} \times \frac{1m^3}{1000\,L} \times \frac{1\min}{60\,s}$$

$$= 0.11 kg/s$$

Appendix C

Experiment Part

C.1 Determination of Quantity of Methanol

The molecular weight of palm oil = 702 g/mol

The molecular weight of methanol = 32 g/mol

The density of palm oil at 60°C = 870.2 kg/m^3

In this work, we use methanol: palm oil to be 6:1

$$\therefore \text{weight of methanol} = (6\ mol)\left(32\ \frac{g}{mol}\right)$$

$$= 192\ g$$

Use 40 liters of palm oil

$$\therefore \text{weight of palm oil} = \rho V$$

$$= \left(870.2\frac{kg}{m^3}\right)(40L)\left(\frac{1m^3}{1000L}\right)\left(\frac{1000g}{1kg}\right)$$

$$= 34{,}808\,g$$

$$\therefore \text{weight of methanol} = \frac{(34{,}808g)(192g)}{702g}$$

$$= 9520\,g$$

In this work, we use KOH 1% by wt of palm oil

$$\therefore \text{weight og KOH} = \frac{(34{,}808g)(1g)}{100g}$$

$$= 348.08\,g$$

C.2 Analysis of Methyl Ester

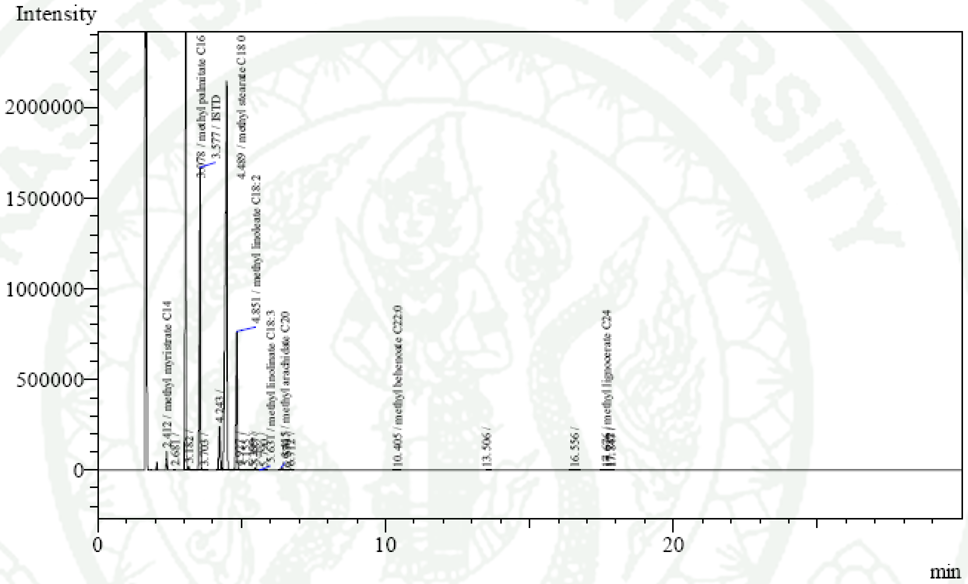
Gas chromatography gave the graph as shown in Figure C1. Percentage of methyl ester can be calculated from the peak area as shown in the following equation.

$$\%FAME = \frac{total\ area - area\ of\ C_{17}}{area\ of\ C_{17}} \times \frac{volume\ of\ C_{17} \times conc.of\ C_{17}}{weight\ of\ sample} \times 100$$

For example, in the conventional stirring at 50°C reaction temperature and 30 minutes reaction time,

-The concentration of standard (C_{17}) = 10.012 mg/ml

-The weight of sample = 52.7 mg



Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	2.412	58627	53290	0.237	%		4	methyl myristrate C
2	2.681	7725	4071	0.031				
3	3.078	8084259	3384417	32.727	%		5	methyl palmitate C1
4	3.182	52163	19628	0.211		SV		
5	3.577	4188328	1668697	16.955	%	V	15	ISTD
6	3.703	8927	2984	0.036		V		
7	4.243	821865	239040	3.327		V		
8	4.489	9020365	2144251	36.516	%	SV	7	methyl stearate C18
9	4.851	2287972	763879	9.262	%	SV	9	methyl linoleate C1
10	4.927	1947	1070	0.008		T		
11	5.155	13890	4246	0.056		T		
12	5.399	3989	1125	0.016				
13	5.487	40018	11714	0.162		V		
14	5.631	2986	864	0.012	%	V	10	methyl linolinate C1
15	5.790	1691	239	0.007		V		
16	6.415	57921	15264	0.234	%		11	methyl arachidate C
17	6.570	2834	601	0.011		V		
18	6.712	24687	5766	0.100		V		
19	10.405	8453	1497	0.034	%		12	methyl behenoate C
20	13.506	1198	257	0.005		SV		
21	16.556	3058	309	0.012		V		
22	17.676	6099	740	0.025	%		14	methyl lignocerate C
23	17.782	1617	530	0.007		V		
24	17.847	1778	397	0.007		V		
Total		24702397	8324876					

Appendix Figure C1 The graph from gas chromatography.

From Figure C1,

The area of standard (C_{17}) = 4,188,328

The total area = 24,702,397

Thus;

$$\%FAME = \frac{24,702,397 - 4,188,328}{4,188,328} \times \frac{1 \times 10.012}{52.7} \times 100$$

$$= 93.05\,\%$$

C.3 Residence Time Calculation

For microwave;

Tube’s length in the microwave = 447 cm.

Tube’s diameter = 0.8 cm.

So;

$$Tube's\ area = \frac{\pi D}{4}$$

$$= \frac{\pi (0.8)^2}{4} = 0.503 cm^2$$

$$Tube's\ volume = A \times L$$

$$= (0.503)(447) = 224.7 \quad cm^3$$

$$= 0.223 \quad L$$

At flow rate 80 L/h;

$$Residence\ time = \frac{V}{q}$$

$$= \frac{0.223 L}{80 L/h}$$

$$= 2.7875 \times 10^{-3} h = 10\ sec$$

For ultrasonic;

Length (L) = 45 cm.

Width (W) = 9 cm.

Height (H) = 9 cm.

So;

$$Volume = H \times L \times W$$

$$= 3,645 \quad cm^3 = 3.645 \quad L$$

At flow rate 80 L/h;

$$Residence\ time = \frac{V}{q}$$

$$= \frac{3.645L}{80L/h}$$

$$= 0.04556\,h = 164 \quad sec$$

For static mixer;

Length = 54 cm.

Housing tube’s diameter = 2.54 cm.

Element’s length (L) = 7.62 cm.

Element’s width (W) = 2.28 cm.

Element’s thickness (T) = 0.15 cm.

Number of elements (N) = 7 element

So;

$$Housing\ tube's\ volume = \frac{\pi D^2}{4} \times L$$

$$= \frac{\pi (2.54)^2}{4} \times 54 = 273.62 cm^3$$

$$Element's\ volume = W \times L \times T \times N$$

$$= 2.28 \times 7.62 \times 0.15 \times 7$$

$$= 18.24 \quad cm^3$$

$$Volume\ static = V_{housing} - V_{elements}$$

$$= 273.62 - 18.24$$

$$= 255.38 \quad cm^3 = 0.255 \quad L$$

At flow rate 37 L/min;

$$Residence\ time = \frac{V}{q}$$

$$= \frac{0.255L}{37L/\min}$$

$$= 6.89 \times 10^{-3} min = 0.4 \quad sec$$

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