การเปรียบเทียบสมรรถนะของเครื่องปฏิกรณ์แบบหลายหน้าที่สำหรับการผลิตไบโอดีเซลจากปฏิกิริยาทรานส์เอสเทอริฟิเคชันของน้ำมันปาล์ม

นายธนัชพร เหล่าสุทธิวงศ์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
สาขาวิชาวิศวกรรมเคมี ภาควิชาวิศวกรรมเคมี
คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2560

Performance comparison of multifunctional reactors for biodiesel production from transesterification of palm oil

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Engineering Program in Chemical Engineering
Department of Chemical Engineering
Faculty of Engineering
Chulalongkorn University
Academic Year 2017

Thesis Title	Performance comparison of multifunctional reactors for biodiesel production from transesterification of palm oil
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ธนัชพร เหล่าสุทธิวงศ์ : การเปรียบเทียบสมรรถนะของเครื่องปฏิกรณ์แบบหลายหน้าที่สำหรับการผลิตไบโอดีเซลจากปฏิกิริยาทรานส์เอสเทอริฟิเคชันของน้ำมันปาล์ม (Performance comparison of multifunctional reactors for biodiesel production from transesterification of palm oil) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ศ. ดร. สุทธิชัย อัสสะบำรุงรัตน์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: ผศ. ดร. กนกวรรณ ง้าวสุวรรณ, 72 หน้า.

ไบโอดีเซลจัดเป็นพลังงานทดแทนซึ่งสามารถใช้แทนน้ำมันดีเซลได้โดยผลิตได้จากการนำน้ำมันพืชมาผ่านปฏิกิริยาทรานส์เอสเทอริฟิเคชันกับแอลกอฮอล์โดยใช้ตัวเร่งปฏิกิริยาทั้งแบบเอกพันธุ์หรือวิวิธพันธุ์ อย่างไรก็ตาม น้ำมันพืชกับแอลกอฮอล์เป็นสารที่ไม่ละลายเข้าด้วยกันซึ่งส่งผลให้ปฏิกิริยาเกิดได้ช้า เครื่องปฏิกรณ์แบบหลายหน้าที่โดยเฉพาะเครื่องปฏิกรณ์แบบเกิดคาวิเตชันเป็นทางออกหนึ่งที่จะช่วยเพิ่มระดับของการผสมระหว่างน้ำมันพืชและแอลกอฮอล์ เครื่องปฏิกรณ์แบบเกิดคาวิเตชันประกอบด้วย อัลตร้าโซนิคและโฮโมจีไนเซอร์ ในงานวิจัยนี้ เครื่องปฏิกรณ์แบบอัลตร้าโซนิค โฮโมจีไนเซอร์ และการใช้เครื่องปั่นกวนแบบให้ความร้อน ได้ถูกศึกษาเพื่อเปรียบเทียบผลได้ของไบโอดีเซลและประสิทธิภาพในเทอมของผลได้ของไบโอดีเซลและพลังงานที่ใช้รวมถึงศึกษาปริมาตรของสารตั้งต้นที่แตกต่างกันโดยใช้น้ำมันปาล์มบริสุทธิ์และเมทานอลเป็นสารตั้งต้นโดยใช้อัตราส่วนโดยโมลระหว่างเมทานอลและน้ำมันพืชเท่ากับ 6 ต่อ 1 เมื่อใช้ตัวเร่งปฏิกิริยาแบบเอกพันธุ์ และอัตราส่วนโดยโมลระหว่างเมทานอลและน้ำมันพืชเท่ากับ 9 ต่อ 1 เมื่อใช้ตัวเร่งปฏิกิริยาแบบวิวิธพันธุ์ที่อุณหภูมิในการเกิดปฏิริยาเท่ากับ 60 องศาเซลเซียส และนอกจากนี้ได้ศึกษาการใช้อัลตร้าโซนิคร่วมกับคลื่นไมโครเวฟ เพื่อเพิ่มการถ่ายเทมวล และการถ่ายเทความร้อนสำหรับปฏิกิริยาทรานส์เอสเทอริฟิเคชันของน้ำมันปาล์ม

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5870163221 : MAJOR CHEMICAL ENGINEERING

KEYWORDS: BIODIESEL/ULTASONIC/HOMOGENIZER/MICROWAVE

TANUTPORN LAOSUTTIWONG: Performance comparison of multifunctional reactors for biodiesel production from transesterification of palm oil. ADVISOR: PROF. SUTTICHAI ASSABUMRUNGRAT, Ph.D., CO-ADVISOR: ASST. PROF. KANOKWAN NGAOSUWAN, D.Eng., 72 pp.

Biodiesel is classified as a qualified alternative energy that can be used for diesel engine. It is produced via transesterification of vegetable oil with alcohol in presence of homogeneous or heterogeneous catalyst. However, oil and alcohol are not totally miscible leading to slow reaction rate. Multifunctional reactors, especially cavitational reactors, are proposed to overcome this issue. The cavitational reactors include ultrasonic reactor and homogenizer reactor. In this work, ultrasonic reactor, homogenizer reactor and conventional magnetic stirred reactor were used to investigate and compare biodiesel yield and the efficiency in terms of biodiesel yield based on the energy consumption at different values of reaction mixture volume using refined palm oil and methanol as the feedstocks at a methanol to oil molar ratio of 6:1 for homogeneous catalyst (NaOH) and a methanol to oil molar ratio of 9:1 for heterogeneous catalyst (CaO) at reaction temperature of 60°C. The combination of ultrasonic and microwave irradiation was performed to increase mass and heat transfers for transesterification of palm oil.

Department:	Chemical Engineering	Student's Signature ....
Field of Study:	Chemical Engineering	Advisor's Signature ....
Academic Year:	2017	Co-Advisor's Signature ....

ACKNOWLEDGEMENTS

I would like to express the deepest appreciation to my advisor, Professor Dr. Suttichai Assabumrungrat and my co-advisor, Assistant Professor Dr. Kanokwan Ngaosuwan for their suggestions, guidance and encouragement to me. .

In addition, I would also be grateful to Associate Professor Dr. Kasidit Nootong as the chairman, Associate Professor Dr. Joongjai Panpranot and Assistant Professor Dr. Worapon Kiatkittipong as the members of the thesis committee for their comments and suggestion.

Moreover, I would like to acknowledge with gratitude to Mr. Kijchai Kanjanaprapakul, my seniors and my friend for their support, guideline and other.

Finally, I also would like to acknowledge with my parents for support and encourage me, and this dissertation would not have been possible without them

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Chapter 1

Introduction

1.1 Introduction

Biodiesel is classified as a qualified alternative energy that can be used with diesel engine. The most common way to produce biodiesel is transesterification of vegetable oil or animal fats. There are two types of catalyst used for biodiesel production; i.e., homogeneous and heterogeneous catalysts [1]. However, biodiesel production rate seems to have an obstacle by mass transfer. A mixture of vegetable oil with alcohol is not totally miscible leading to slow reaction rate. Many researchers have studied different multifunctional reactors especially caviational reactor to improve the mixing between two insoluble reactants. Moreover, the use of microwave assisted reactor can improve mass and heat transfer to drive reaction faster. It was demonstrated in earlier researches that ultrasonic irradiation can increase interphase mixing between the phases at the desired reaction conditions by producing high temperature and high pressure. This multifunctional reactor is based on cavitation phenomena. When ultrasound wave passes through liquid medium and it forms acoustic cavitation. This cavitation provides very high pressure and temperature in the liquid media [2]. Lee et al. [3] studied transesterification of palm oil using waste obtuse horn shell as catalyst under magnetic stirring. A maximum oil conversion (86.75%) was obtained at a reaction time of 6 h, 5%wt catalyst and a methanol to oil molar ratio of 12. This catalyst can be reused 3 times with more than 70% of biodiesel. The main deactivation for solid catalyst were from leaching of active species, pore blockage of catalyst and formation of new phase. Hingu et al. [4] synthesized biodiesel from waste cooking oil using potassium hydroxide in sonochemical reactors. The use of sonochemical reactors can improve mass transfer and interphases mixing between reactant and also reduce the requirement of reaction parameters. Using sonochemical reactors compared with conventional stirring at the same condition, it was found that 89.5% and 57.5% of oil conversions were obtained from the sonochemical reactor and the conventional stirring reactor, respectively. Chen et al. [5] have investigated transesterification of palm oil using ostrich eggshell-derived CaO catalyst in an ultrasonic reactor. It was found that the maximum yield was 92.7% in 60 min. Moreover, catalyst can be reused more than 8 times with slightly loss of activity due to formation of new phase (inactive phase was Ca(OH)2 and active phase was Ca-glyceroxide). Bhangu et al. [6] reported that when increasing ultrasonic frequency the bubble size was decreased and led to reduce collapse intensity which resulted in lower biodiesel yield from 35% at 44 kHz to 5% at 98 kHz at a reaction time of 15 min.

Using a homogenizer which is based on rotor/stator principle to generate shear force is another interesting approach to form emulsions of reactants. In addition, high turbulence occurs in the shear gap between rotor and stator, which provides optimum mixing of the suspension [7]. Crudo et al. [8] reported that using rotor-stator could generate hydrodynamic cavitation in transesterification of refined palm oil, bleached palm oil and waste cooking oil. The high-speed rotation of reactor generated the micron-sized droplets of the immiscible mixture to enhance mass and heat transfer. More than 99% of fatty acid methyl ester contents in the oils were obtained.

Increase of heat transfer in the reaction mixture can also boost the biodiesel production rate. Using microwave irradiation is another interesting approach to enhance reaction rate because direct energy is obtained by absorption of molecules, and then the molecules are vibrated resulting in increased heat transfer [9]. Sherbiny El et al. [10] reported that using microwave technique to produce biodiesel of Jatropha oil with a methanol to oil molar ratio of 7.5, catalyst loading of 1.5% wt and reaction temperature of 65°C. More than 97% of biodiesel yield was obtained and reaction time was reducing from 60 to 2 min when applied microwave irradiation.

Guidelines to improve the biodiesel production in term of decreasing in mass transfer limitation are by using the techniques of ultrasonic and homogenizer, and in term of increasing in heat transfer is by using microwave irradiation. Also using combination of ultrasonic and microwave was demonstrated in previous researches [11]. However, there are still lack of information for the comparison of the maximum performance of the cavitational reactors (including of ultrasonic and hydrodynamic reactor) based on the energy consumption, yield efficiency and differential reaction mixture volume using different types of catalyst (homogeneous and heterogeneous catalysts). Therefore, this research will fulfill these information as well as provide critical recommendation for the maximum performance of these technologies and guideline to use in these reactors the industrial scale. The effect of mixing characteristics on biodiesel yield will be investigated based on their energy consumption. The effect of reaction mixture volume on the biodiesel production efficiency of using direct ultrasonic, homogenizer and mechanical stirrer will also be considered. And also study the combination of ultrasonic and microwave irradiation to increase mass and heat transfers for transesterification of palm oil.

1.2 Objective

To study the performance of multifunctional reactors including direct ultrasonic probe sonoreactor, homogenizer reactor and combined ultrasonic and microwave reactor for the biodiesel production from transesterification of palm oil using homogeneous and heterogeneous catalyst.

1.3 Scope of work

1.3.1 Improve biodiesel production rate by using direct ultrasonic probe reactor and homogenizer reactor and compared to mechanical stirrer reactor, for both cases of sodium hydroxide (NaOH) and calcium oxide (CaO) catalysts on the biodiesel yield based on the energy consumption under similar transesterification condition.

1.3.2 Study the effect of reaction mixture volume (165, 500 and 1000 mL) on biodiesel yield and the biodiesel production efficiency using direct ultrasonic probe reactor, homogenizer reactor and mechanical stirrer reactor under the similar transesterification of palm oil using sodium hydroxide (NaOH) and calcium oxide (CaO) catalyst.

1.3.3 Enhance the biodiesel production rate in term of heat and mass transfers by using the combination of ultrasonic and microwave irradiation reactor for NaOH catalyzed transesterification of palm oil.

1.4 Expected outputs

The maximum performance of multifunctional reactor is recommended for biodiesel production via transesterification of palm oil.

Chapter 2

Theory

2.1 Biodiesel

Biodiesel is liquid fuel that can be used as diesel fuel. Which is produced from vegetable oil or waste cooking oil through a chemical process into biodiesel by having the catalytic acid or base. Biodiesel can be blended in diesel fuel according to require proportions. If use of pure biodiesel, called B100, which is suitable for low speed diesel engine or agricultural machinery. When using biodiesel in vehicles. The proper proportion is 5% of biodiesel to 95% of diesel which have been tested without engine modification [12].

Thailand has plants that can be used to produce biodiesel as follow [12]:

- Palm is the main plant that the government promoted as raw materials for biodiesel production. This plant grows well in tropical conditions such as south of Thailand. Palm oil can be produced vegetable oil, shortening, sweetened condensed milk, ice-cream and soap.
- Jatropha grows well in rainy season. 1 kg of Jatropha seed have a quantity of 1200 – 1400 seeds which can be extracted Jatropha oil about 0.25 – 0.35 L.
- Soybean plants are widely used while the production of the country has decreased every year. So it is not enough to biodiesel production.
- Coconut is an important plant, grown in every region of the country. However the productivity of coconut always bring for copra. If it is used as raw material in biodiesel production the government must encourage coconut growing increase.
- Algae plant is one that is gaining popularity around the world in order to produce biodiesel. Algae yield per Rai per year over palm oil 10 – 30 times (1 Rai equals to 3.95 Acres). However, the cost of extracting oil from algae is also high.

- Peanut seeds contain 50 – 60% of oil, but acreage declined steadily that led to the quantity produced is not enough to demand within the county.
- 65% of Sesame was exported to foreign countries. Sesame oil is high nutritional value that suitable for cooking than biodiesel production.
- Castor is a global market with high usage requirements apply in a range of industries such as pharmaceutical. So the quantity is not enough to be used in biodiesel production.

Various types of vegetable oil are different of fatty acid chains and content. Each fatty acid has a diverse number of carbon and double bonds. Therefore, each oil has physical and chemical effect properties that are different. The composition of fatty acids in the various type of oil is shown in Table 2.1

Table 2.1 Composition of fatty acids in vegetable oil [13].

Fatty acid	Soy bean oil	Sunflower oil	Palm oil	Peanut oil	Corn oil	Olive oil
Lauric (12:0)	0.0	0	0.1	0	0	0
Myristic(14:0)	0.0	0	0.7	0.1	0	0
Palmitic(16:0)	11.3	6.2	36.7	8.0	6.5	11.6
Palmitoleic(16:1)	0.1	0.1	0.1	0	0.6	1.0
Stearic(18:0)	3.6	3.7	6.6	1.8	1.4	3.1
Oleic(18:1)	24.9	25.2	46.1	53.3	65.6	75.0
Linoleic(18:2)	53.0	63.1	8.6	28.4	25.2	7.8
Linolenic(18:3)	6.1	0.2	0.3	0.3	0.1	0.6
Arachidic(20:0)	0.3	0.3	0.4	0.9	0.1	0.3
Gadoleic(20:1)	0.3	0.2	0.2	2.4	0.1	0
Behenic(22:0)	0	0.7	0.1	3.0	0	0.1
Erucic(22:1)	0.3	0.1	0	0	0.1	0
Lignoceric(24:0)	0.1	0.2	0.1	1.8	0.1	0.5
Nervonic(24:1)	0	0	0	0	0	0

The properties of biodiesel is a biodegradable, non-toxic and environment friendly as shown in Table 2.2

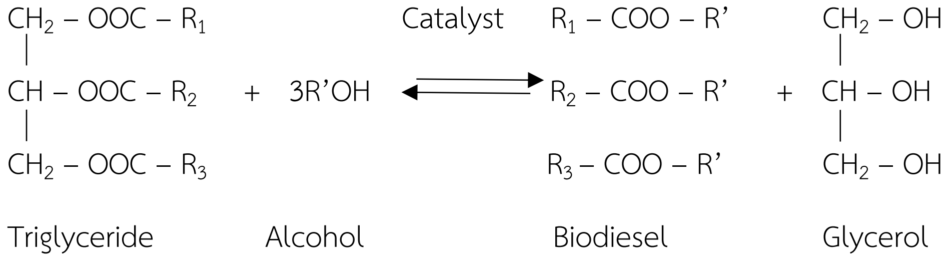
Table 2.2 Comparison of the advantages of biodiesel with petroleum diesel [12].

Experimental	Biodiesel	Petroleum diesel
Emissions of sulfur dioxide (SO_2)	Not found	Found
The destruction of the ozone layer	Reduction of 50%	Not reduced
Tiny dust	Reduction of 30%	Not reduced
The carbon monoxide (CO)	Reduction of 50%	Not reduced
Hydrocarbons	Reduction of 80%	Not reduced
The exhaust from the engine	None	Possess

2.1.1 Reactions for biodiesel production

2.1.1.1 Transesterification

Transesterification has been used in biodiesel production. This is a reaction of triglycerides with alcohol in the presence of catalyst to form fatty acid alkyl esters (biodiesel) as the main product and glycerol as the by-product. The mechanism of transesterification of triglycerides is shown in Figure 2.1



$$\begin{array}{lccccl}
CH_2-OOC-R_1 & & & & R_1-COO-R' & CH_2-OH \\
| & & & & & | \\
CH-OOC-R_2 & + & 3R'OH & \overset{Catalyst}{\rightleftarrows} & R_2-COO-R' \quad + & CH-OH \\
| & & & & & | \\
CH_2-OOC-R_3 & & & & R_3-COO-R' & CH_2-OH \\
\text{Triglyceride} & & \text{Alcohol} & & \text{Biodiesel} & \text{Glycerol}
\end{array}$$

Figure 2.1 Transesterification of triglycerides.

The factors that effect on transesterification reaction are following as [14].

- Water and free fatty acids

The substrate for via transesterification with base catalyst must have acid value less than 1 mg KOH/g. If there is acid value greater than 1 mg KOH/g, NaOH was required to neutralize free fatty acids (FFAs). Water is present in the substrate, it will cause the soap which efficiency decreased and viscosity of the product is increased including separation of glycerol is more difficult.

- Molar ratio

Methanol to oil molar ratio calculated from stoichiometric of transesterification. It should be three moles of alcohol and one mole of triglycerides to produce three moles of fatty acid alkyl esters and one mole of glycerol. However in practice, required more than three mole of methanol because glycerol can dissolve in the methanol. If using a base catalyzes transesterification it requires alcohol to oil molar ratio 6:1 to give the highest conversion.

- Catalyst

Alkali-catalyzed transesterification is much faster than acid-catalyzed. However, if a feed stock is composed of high amount of FFA. FFA can react with base catalyst and incurred saponification reaction when using a large amount of the catalyst. Acid catalyst is suitable in higher FFA come with a slower rate of reaction.

- Reaction temperature

Transesterification occurs at different temperatures depending on oil as used such as castor oil will be occurred at 20 – 35°C while soybean oil will be occurred at 45 – 60°C and waste cooking oil at 50 – 60°C. Higher temperatures will be increased rate of reaction, but not exceed boiling point of methanol (64.7°C).

- Reaction time

The conversion rate increases with reaction time due to the mixing and dispersion of methanol into oil.

2.1.1.2 Esterification

This is a reaction of free fatty acid with alcohol using an acid catalyst to form free fatty acid alkyl esters and water. The mechanism of esterification is shown in Figure 2.2

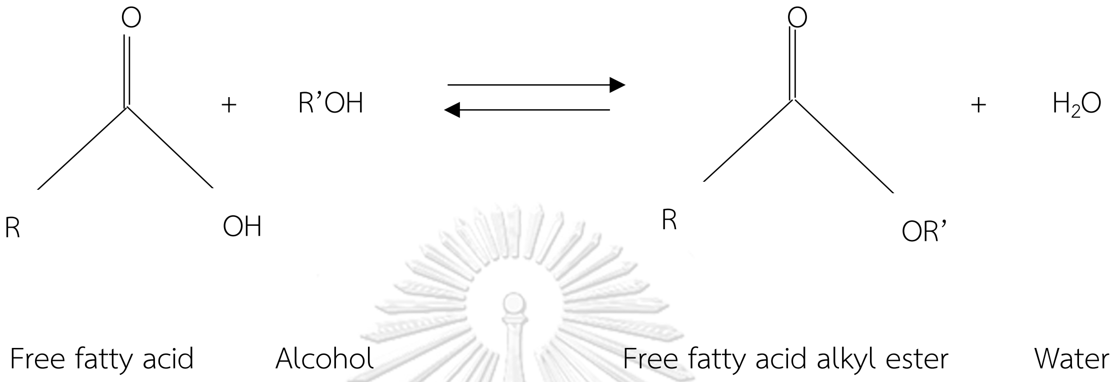


Figure 2.2 Esterification of free fatty acids with alcohols [15].

2.1.1.3 Hydrolysis

Hydrolysis reaction takes place as three stepwise. First step triglyceride is hydrolyzed to diglycerides, second step diglycerides is hydrolyzed to monoglycerides and final step monoglycerides is hydrolyzed to glycerol. In each step occurs fatty acid as shown in Figure 2.3

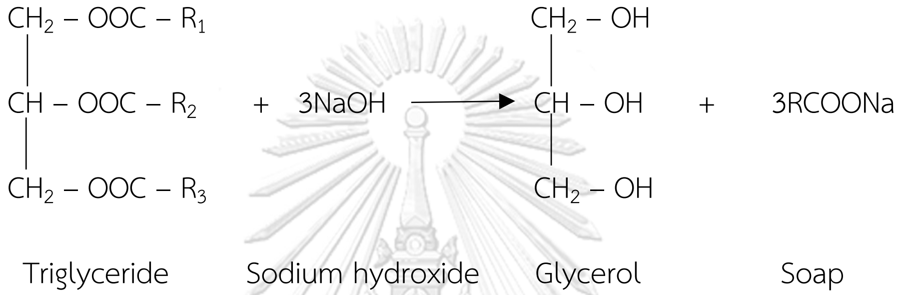
(1) $C_3H_5(OCOR)_3 + H_2O \longleftrightarrow C_3H_5(OH).(OCOR)_2 + RCOOH$
Triglyceride Water Diglyceride FFA
(2) $C_3H_5(OH).(OCOR)_2 + H_2O \longleftrightarrow C_3H_8(OH)_2.(OCOR) + RCOOH$
Diglyceride Water Monoglyceride FFA
(3) $C_3H_5(OH)_2.(OCOR) + H_2O \longleftrightarrow C_3H_5(OH)_3 + RCOOH$
Monoglyceride Water Glycerol FFA

Figure 2.3 Hydrolysis reaction of vegetable oil [16].

2.1.2 Reaction involving for biodiesel production

2.1.2.1 Saponification

Saponification is the reaction between triglycerides in fat/oil with aqueous NaOH or KOH to convert into glycerol and soap as shown in Figure 2.4. This reaction is considered an undesirable reaction because the soap caused a lot of problems in the reaction, such as soap can lower the biodiesel yield and difficult to separate from product.



$$\begin{array}{lcccl} CH_2-OOC-R_1 & & & CH_2-OH & \\ | & & & | & \\ CH-OOC-R_2 & + \; 3NaOH & \longrightarrow & CH-OH & + \; 3RCOONa \\ | & & & | & \\ CH_2-OOC-R_3 & & & CH_2-OH & \end{array}$$

Figure 2.4 Saponification process [17].

2.1.3 Properties and standard of biodiesel

Biodiesel is a fuel that produces in order to reduce the use of diesel. However, the raw materials and processes to produce biodiesel are different. The properties of biodiesel that is different. To obtain biodiesel with the same properties that can be used in diesel engines without causing subsequent problems. So it has set standard of biodiesel as shown in Table 2.3

Table 2.3 U.S. and European specifications for biodiesel B100 and biodiesel blends [18].

Property	Biodiesel (B100)				B6-B20 blends	
	U.S. (ASTM D6751-08)		Europe (EN 14214)		U.S. (ASTM D7467-08)	
	Limits	Method	Limits	Method	Limits	Method
Water and sediment (vol%, max)	0.05	D2709	0.05	EN12937	0.05	D2709
Total contamination (mg/kg, max)	-	-	24	EN12662	-	-
Kinematic viscosity @ 40°C (mm^2/s)	1.9-6.0	D445	3.5-5.0	EN3104/3105	1.9-4.1	D445
Flash point, closed cup (°C, min)	93	D93	101	EN3679	52	D93
Methanol (wt.%, max)	0.20	EN14110	0.20	EN14110	-	-
Cetane no. (min)	47	D613	51	EN5165	40	D613
Cloud point (°C)	Report	D2500	Country Specific	-	Report	D2500
Sulfated ash (wt.%, max)	0.020	D874	0.020	EN3987	-	-
Total ash (wt.%, max)	-	-	-	-	0.01	D482
Gp I metals Na + K (mg/kg, max)	5.0	EN14538	5.0	EN14108/14109	-	-
Gp II Metals Ca + Mg (mg/kg, max)	5.0	EN14538	5.0	EN14538	-	-

Table 2.3 U.S. and European specifications for biodiesel B100 and biodiesel blends [18] (cont.).

Property	Biodiesel (B100)				B6-B20 blends	
	U.S. (ASTM D6751-08)		Europe (EN 14214)		U.S. (ASTM D7467-08)	
	Limits	**Method**	**Limits**	**Method**	**Limits**	**Method**
Total Sulfur (ppm, max)	15	D5453	10	EN20846	15	D5453
Phosphorous (ppm, max)	10	D4951	4	EN14107	-	-
Acid no. (mg KOH/g, max)	0.50	D664	0.50	EN14104	0.3	D664
Carbon residue (wt.%, max)	0.05	D4530	0.30	EN10370	0.35	D524
Free glycerin (wt.%, max)	0.02	D6584	0.02	EN14105 /14106	-	-
Total glycerin (wt.%, max)	0.24	D6584	0.25	EN14105	-	-
Mono glyceride (wt.%, max)	-	-	0.80	EN14105	-	-
Diglyceride (wt.%, max)	-	-	0.20	EN14105	-	-
Triglyceride (wt.%, max)	-	-	0.20	EN14105	-	-
Distillation (T90°C, max)	36	D1160	-	-	343	D86

Table 2.3 U.S. and European specifications for biodiesel B100 and biodiesel blends [18] (cont.).

Property	Biodiesel (B100)				B6-B20 blends	
	U.S. (ASTM D6751-08)		Europe (EN 14214)		U.S. (ASTM D7467-08)	
	Limits	Method	Limits	Method	Limits	Method
Copper strip corrosion (3-h at 50° C, max)	No.3	D130	No.1	EN2160	No.3	D130
Oxidation Stability (h @ 110°C, min)	3.0	EN14112	6.0	EN14112	6	EN14112
Linolenic acid methyl ester (wt.%, max)	-	-	12.0	EN14103	-	-
Polyunsaturated acid methyl esters (wt.%, max)	-	-	1.0	prEN15799	-	-
Ester Content (wt.%, min)	-	-	96.5	EN14103	6-20 vol.%	D7371
Iodine Value (g I_2/100 g, max)	-	-	120	EN14111	-	-
Density (kg/m^3)	-	-	860-900	EN3675	-	-
Lubricity @ 60°C, WSD, microns (max)	-	-	-	-	520	D6079
Cold Soak Filterability (seconds, max)	360	D7501	-	-	-	-

2.2 Catalyst

Catalyst is the substance added to the reaction, and then make the reaction faster by involved in reaction or not. At the end of reaction, catalyst will be properties as before. Figure 2.5 shows catalytic reaction between A and B to give product P. The bonding of molecules A and B to the catalyst and react to give a product P. Then, product P separates from the catalyst and the catalyst is available for next reaction [19].

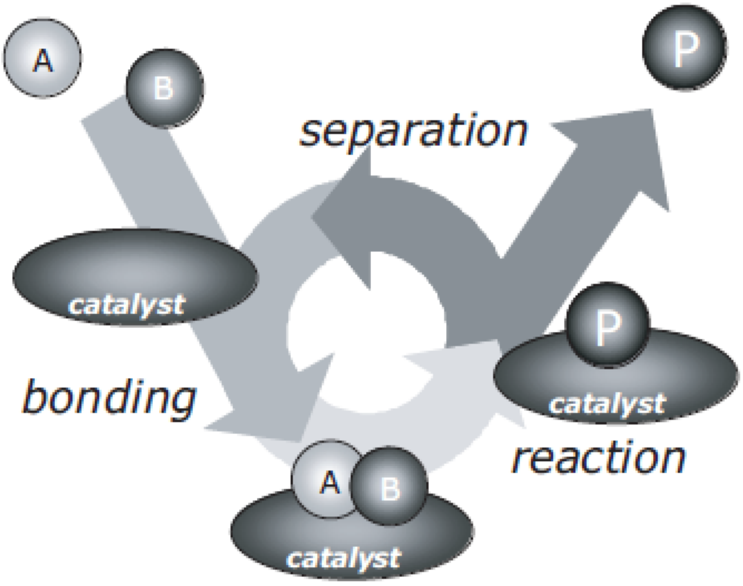


Figure 2.5 Catalytic reaction is a sequence of elementary [19].

A catalyst accelerates a chemical reaction by decreasing potential energy. Therefore, make the reaction faster, but will not make a change to energy of reaction as see in Figure 2.6

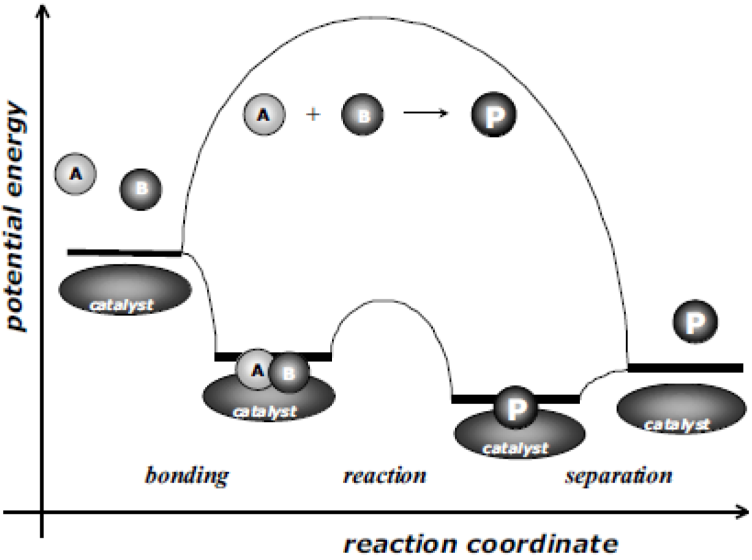


Figure 2.6 Potential energy diagram with and without catalyst [19].

2.2.1 Type of biodiesel catalyst

2.2.1.1 Homogeneous catalyst

Biodiesel is commonly produced using homogeneous base catalyst such as sodium hydroxide or potassium hydroxide. This catalyst is higher than heterogeneous catalyst, but the drawbacks from homogeneous catalyst are difficult to separate and required amount of water for washing. However, used of this catalyst has some disadvantage as a vegetable oil must be acid value less than 1 mgKOH/g because saponification reaction occurs at acid value more than 1 mgKOH/g [20].

2.2.1.2 Heterogeneous catalyst

In the present, development of solid base catalyst for biodiesel production have been popular. Calcium oxide is one more interesting due to high basic strength, cheap because it can be synthesized from natural materials and low solubility in methanol. However, this catalyst can be deactivated such as poisoned by strong adsorption of FFA on the catalyst surface [20].

2.2.2 Deactivation of heterogeneous catalyst

2.2.2.1 Poisoning

Poisoning is deactivation of catalyst by deposition of reactant, product or impurities on catalyst surface, which is a strong chemical absorption. The properties of catalyst will be changed and cannot accelerate the reaction as illustrated in Figure 2.7

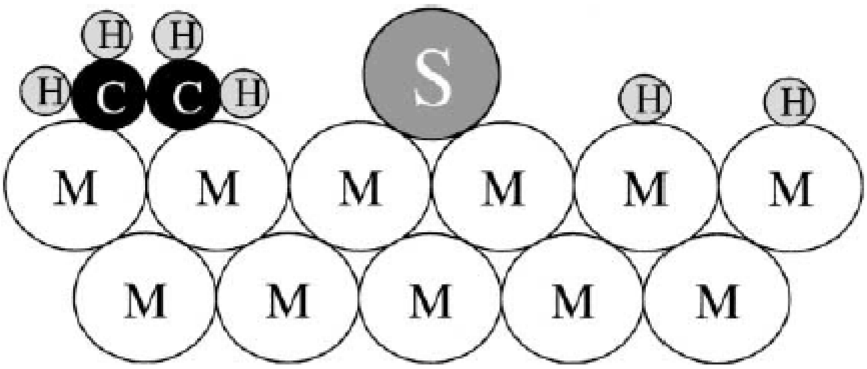


Figure 2.7 Sulfur poisoning of ethylene hydrogenation on metal surface [21].

2.2.2.2 Fouling, coking and carbon deposition

Fouling is physical deposition of species from fluid phase onto catalyst surface. Which result in carbon (or coke) formation on catalyst that causes block access of reactants to surface sites that led to activity loss of catalyst as illustrated in Figure 2.8

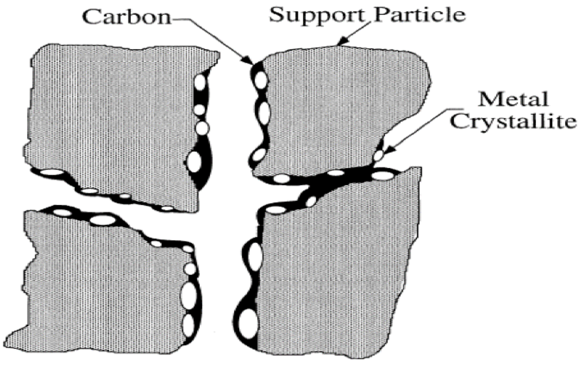


Figure 2.8 Pore plugging of supported metal catalyst [21].

2.2.2.3 Sintering

Sintering is a reduction of catalyst surface by adjust internal structure of catalyst, which mainly from heat. Sintering can be occurred in metal on support or no support by Migration of crystallite or atomic of metal into a larger crystallite or atomic as see in Figure 2.9. The factors affected deactivation is distribution of atomic particles or metal crystallite, particle size and ability to evaporate of atomic particles or metal crystallite at operation conditions.

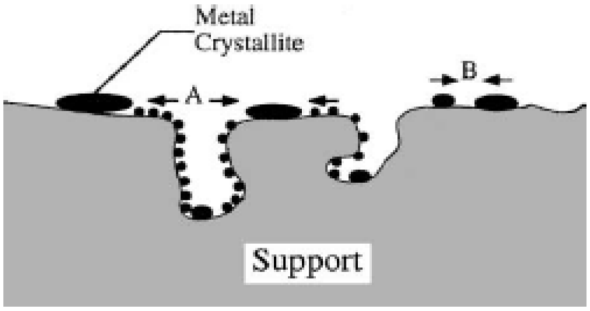


Figure 2.9 Crystallite growth due to sintering by (A) atomic migration or (B) crystallite migration [21].

2.2.2.4 Failure of catalyst

Failure of catalysts are crushing of granular, the size reduction or breakup of catalyst granules and erosion of catalyst particles. Attrition will be seen by reduction of particle size and may be out of some elements of catalyst that cause loss of mechanical properties of granular particles. And leads to blockage of the flow, which affects increases in pressure drop in system.

2.3 Ultrasonic

Ultrasonic wave is a high frequency over 20 kHz to 10^6 kHz, which is frequency range of human cannot hear. High frequency causing in chemical, biological and physical effect in various forms. Ultrasonic wave will be occurred bubbles, high pressure and hot spot [22]. In general, ultrasonic wave can be divided into two categories include: first, low power and high frequencies that used in diagnostic ultrasound. Second, high power and low frequencies or called power ultrasound that is often applied in food processing due to cavitation phenomena making better of mixing [23].

2.3.1 Cavitation

Cavitation phenomena is process that occurs in medium that make in chemical and physical change. The bubbles occur due to structure of liquid receiving ultrasound wave will be compress and stretch repeatedly as thousands of cycles. The bubbles that occurs will be larger unit break up as see in Figure 2.10. In a state of bubbles burst, found to cause temperature up to 4,727°C and pressure up to 2,000 atm in the area point of shock waves [23].

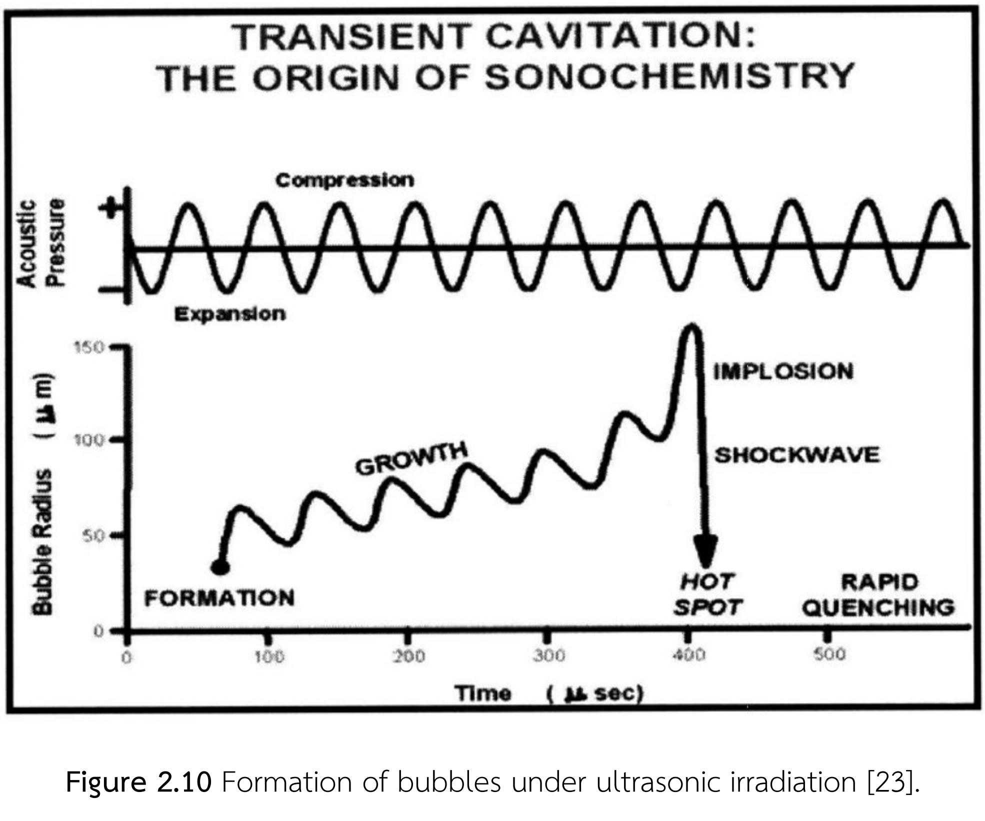


Figure 2.10 Formation of bubbles under ultrasonic irradiation [23].

2.3.2 Type of ultrasonic reactor

2.3.2.1 Direct ultrasonic (ultrasonic probe system)

Ultrasonic probe system, transducer will connect to device called horn. Shape of horn is different as shown in Figure 2.11. Generally, direct ultrasonic has the highest energy transferred to liquid medium resulting to higher intensity of cavitation. Ultrasonic probe system is shown in Figure 2.12. The disadvantages of this horn type is easily damage by corrosion or cavitation effect during process.

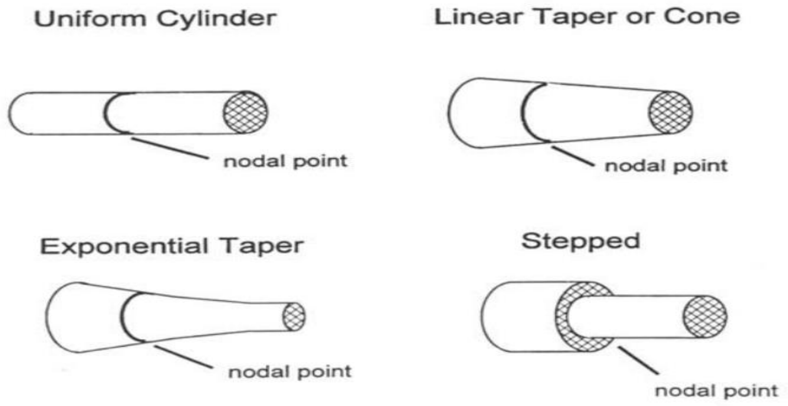


Figure 2.11 Shape of horn [23].

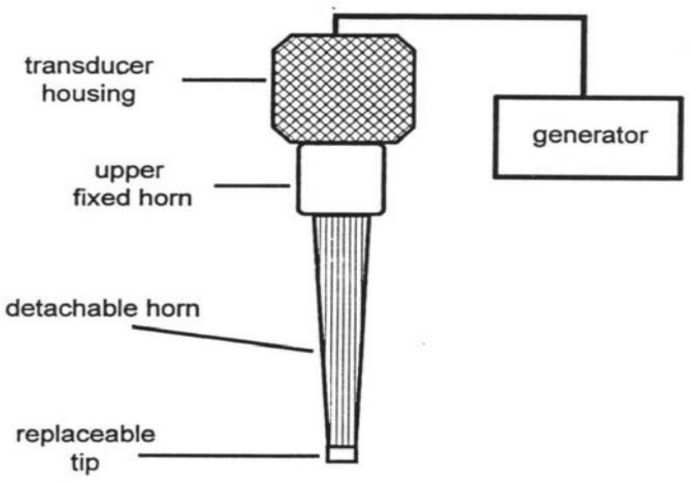


Figure 2.12 Ultrasonic probe system [23].

2.3.2.2 Indirect ultrasonic (ultrasonic baths)

Ultrasonic baths is device that mostly used in the laboratory due to inexpensive compared to probe system. Typically, transducers bonded to base and frequency with most used are approximately 40 kHz. Ultrasonic baths is shown in Figure 2.13

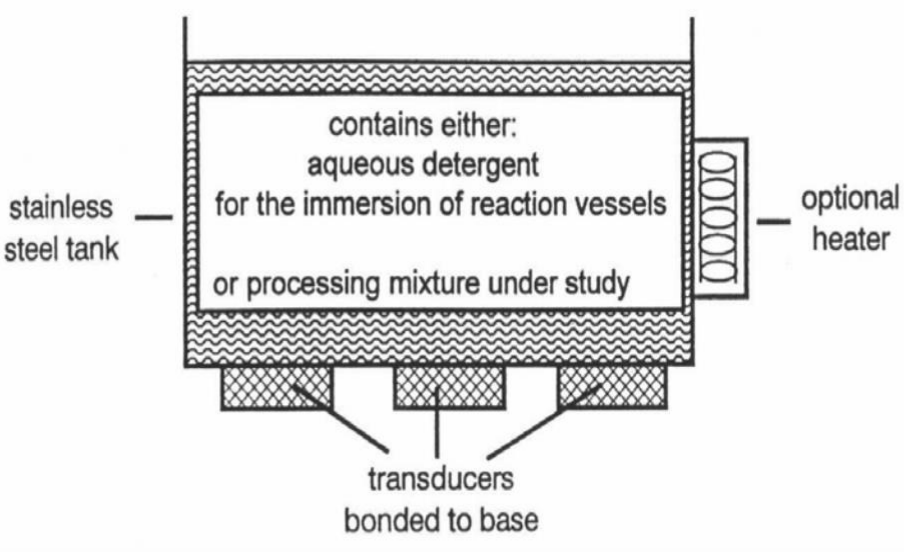


Figure 2.13 Ultrasonic baths system [23].

Ultrasonic baths system have some equipment to enhance performance such as thermostatically controlled heating, frequency sweeps, power leveling devices and timer. Ultrasonic baths generally less energy to avoid from cavitation effect at inner wall of tank. Additional, some of energy is lost due to higher liquid medium.

2.4 Homogenizer

2.4.1 Principle of homogenizer

Due to rotor/stator principle, the high rotation speed of the rotor, the medium to be processed is automatically drawn axially into the dispersion head and then forced radially through the slots in the rotor/stator arrangement as show in Figure2.14. The high accelerations acting on the material produce extremely strong shear and thrust forces. However, high turbulence occurs in the shear gap between rotor and stator, which provides optimum mixing of the suspension [24].

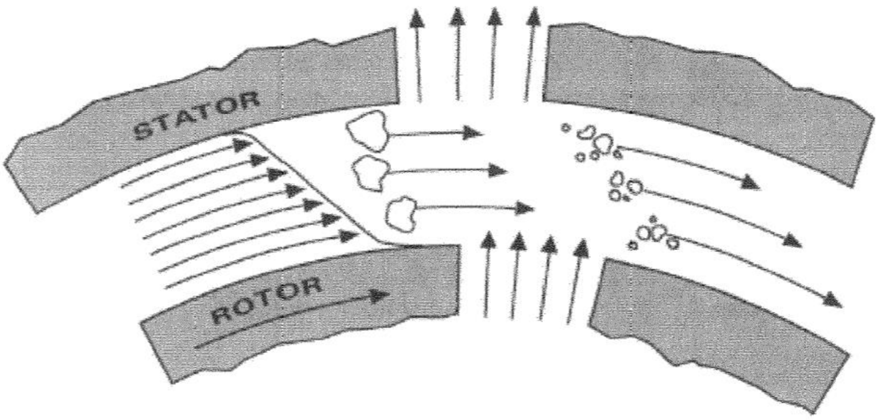


Figure 2.14 Rotor/stator principle [24].

2.4.2 Shear force

Fluid between two plates are placed in parallel when a force acts with top plate will generate shear force with fluid. This fluid was moving in manner layer due to different speed. Top layer has maximum speed equal to speed of plate and then, speed will decrease gradually until bottom layer which stick to bottom plate as shown in Figure 2.15. The shear force can improve efficiency in mixing of fluid [25].

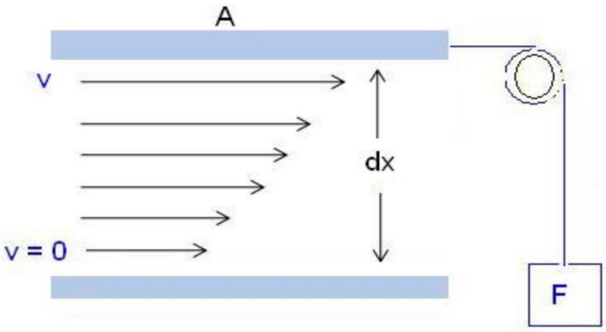


Figure 2.15 Shear force diagram [25].

2.5 Microwave

Microwave are in the frequency range 0.3 – 300 GHz. Mainly used as a power source to heat for processing in industry by magnetron transforming electric power to microwave which is not harmful to humans. Microwave is crept into the food from all sides of inside walls and then spreading toward food. When the wave crash food causing food molecules vibration and friction resulting to occurs thermal energy. Heating with microwave is better than conventional method such as quick, economical, safe and uniformity of temperature distribution [26].

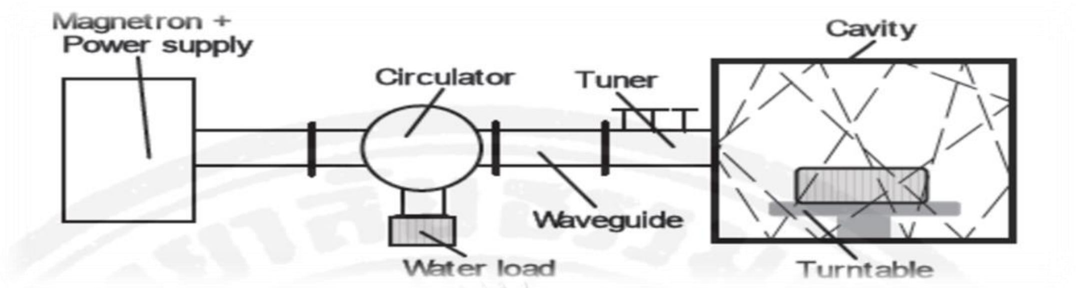


Figure 2.16 The basic components of microwave systems [27].

2.5.1 The principle of heat

Figure 2.16 shows the installed magnetron on waveguide generated microwave energy. Microwave energy pass through the waveguide into material that has been in cavity and absorbent. The absorbent will occurs vibration and friction of food molecular resulting to occur thermal energy. For absorbing of material, there will be some transmitted wave and reflected wave of material. The microwave energy is reflected depend on the dielectric properties of material. The reflected wave will damage with magnetron. Basically, microwave systems is installed a wave trap is called circulator. It is also equipped with a customizable order to reduce energy reflection by using a device called matching tuner [27].

2.5.2 Microwave equipment

2.5.2.1 Generator

A generator consist of DC power supply and magnetron. Microwave are generated by magnetron based on the DC energy changing into microwave energy.

2.5.2.2 Protective devices

Several protective devices have been introduced to the system to prevent reflection of microwave energy that may destroy magnetron. A simple way is to have thermal switch which is sensitive to high heating and cutting power off. In the complicated systems, there will be circulator that a device to transfer energy reflection into dummy load such as water or other material. Installation of water systems with circulator system is called isolator which extends the lifetime of magnetron.

2.5.2.3 Reflection monitor

Reflection monitor is a devices to measure amount of reflection wave.

2.5.2.4 Matching tuner

Matching tuner is a devices to adjust resistance form generator to suit with material which must be used with power monitor to improve efficiency of system and protected generator.

2.5.2.5 Applicator

Applicator or cavity is a part for process heating by receive microwave energy from generator. In general, the applicator is classified according to type of cavity and waveguide as shown below:

1) Cavity applicators
  - Batch cavity process
  - Continuous cavity process

2) Waveguide applicators
  - Rectangular waveguide
  - Circle waveguide
  - Tunnel waveguide
  - Bend waveguide

2.5.2.6 Other devices

Optional accessories that make system more efficient is leakage and safety control systems.

2.5.3 Caution in the use of microwave

2.5.3.1 Do not use the machine while the door is open, it will make microwave leaking out.

2.5.3.2 Do not use the machine if damage occurred or corrosion of furnace wall.

2.5.3.3 Do not operate the machine without food or water, causing sparks in the oven.

2.5.3.4 Container bottles are packed food must have to open them, otherwise it will cause broken of bottle.

2.6 Mixing methods

Mechanical agitation is the most common method that is applied to enhance mixing in the industrial scale. However, there are same limitation of using mechanical agitation such as dead zone at the bottom of reactor and also the range of ratio of liquid level to blade diameter and other mixing parameters. Therefore, the cavitation phenomena has been recently gain more attention to increase the mixing efficiency. There are four principle types of cavitation including of 1) acoustic cavitation, 2) hydrodynamic cavitation, 3) optic cavitation and 4) particle cavitation. Out of these four methods of cavitation, only acoustic and hydrodynamics cavitation generated desired intensity suitable for chemical or physical processing.

2.6.1 Acoustic cavitation

Cavitation is defined as the phenomenon of formation, growth, and eventual collapse of small bubbles within a liquid. Acoustic cavitation is well knows generated from ultrasonic irradiation, that is known to enhance biodiesel reaction through cavitation bubbles as follows: (1) the chemical effect, in which radicals such as H^*, OH^*, and HO_2^* are produced during transient collapse of bubble that accelerate of chemical reaction in liquid medium and (2) the physical effect, in which the microturbulence generated due to radial motion of bubble and creating an intimate mixing of the immiscible reactants resulting to increase interfacial area between oil and methanol phase, giving faster reaction kinetics [28].

The bubble velocity are obtained from Tsochatzidis et al. [29] who study the bubble velocity in axial and radial profile and influence of ultrasonic power.

- Axial profile of mean bubble velocity along the central axis below the horn is presented in Figure 2.17. The distance between the tip of the horn and the bottom of the tank is 68 mm, while P_{in} = 179 W. The results showed that when increase the distance from the horn, the bubble velocity is decreasing. At the above of bottom of vessel, the velocity is reducing to zero.

- Radial profile of the bubble average velocity at three different axial locations is depicted in Figure 2.18. The results showed that the velocity is decreasing with the radial distance. It means that the velocity is larger at centerline and it is reducing to zero at the side of the jet

- Influence of ultrasound power input is depicted in Figure 2.19 and variations of root-mean-square (RMS) velocity are presented in the same Figure which is slightly increased, for example from 0.1 m/s at 30 W to 0.35 m/s at 320 W. Data of Figure 2.19 correspond to a location of 30 mm under the emitter on the centerline of the jet. Data from other locations (not presented in their paper) indicate that by increasing the axial distance, bubble velocity is decreased but still varies linearly with the ultrasonic power.

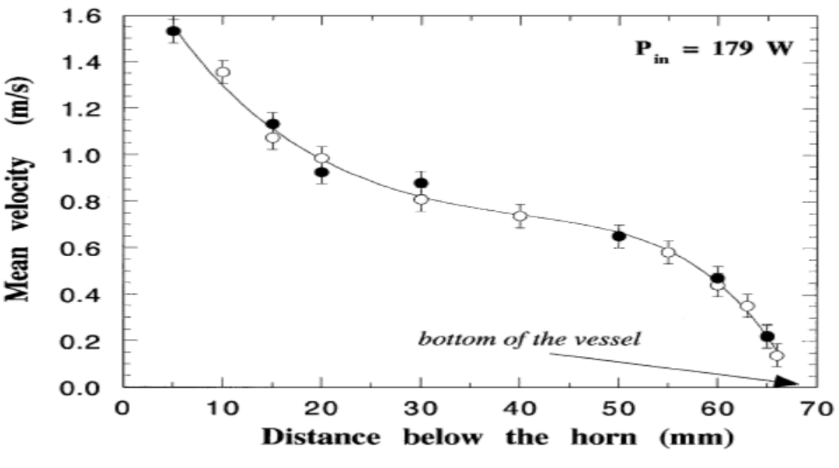


Figure 2.17 Axial profile of mean bubble velocity along the centerline under the ultrasound emitter: P_{in} = 179 W, horn-bottom distance = 68 mm [29].

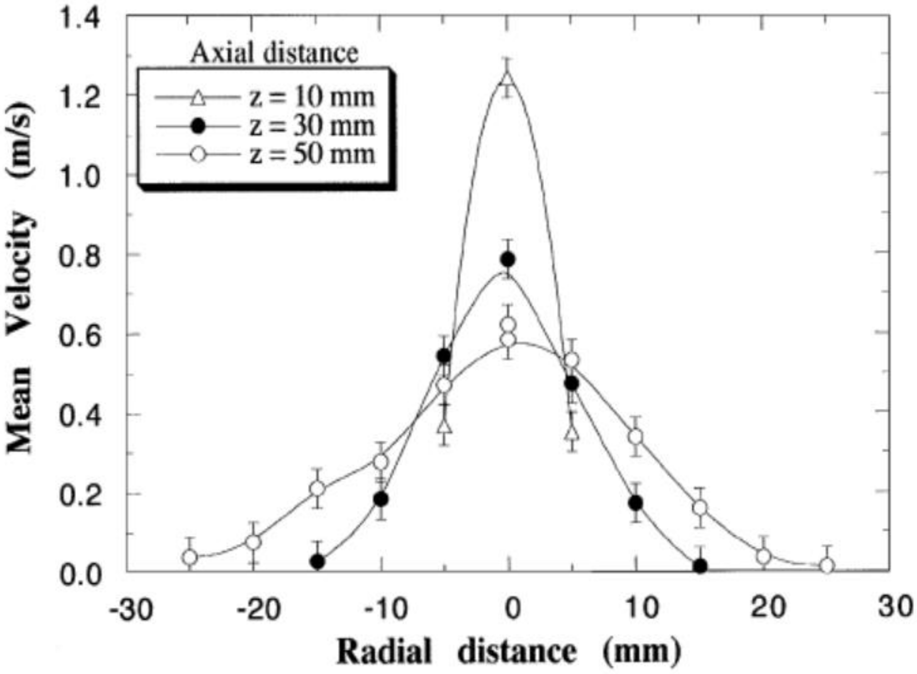


Figure 2.18 Radial profile of mean bubble velocity, at three different axial positions: P_{in} = 179 W, horn-bottom distance = 68 mm [29].

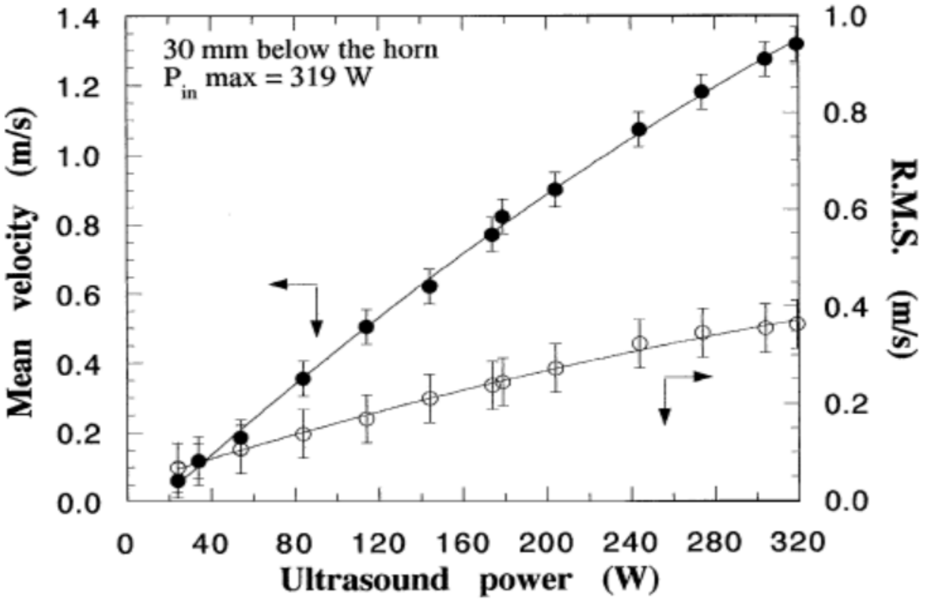


Figure 2.19 Influence of ultrasound power input on average bubble velocity and RMS velocity values: Z = 30 mm, horn-bottom distance = 68 mm [29].

2.6.2 Hydrodynamic cavitation

Hydrodynamic cavitation is well known that generated from homogenizer, As known that it can enhance degree of mixing by using geometry of system creating velocity variation as follows: (1) increasing velocity of the liquid with a corresponding decrease in the local pressure, (2) if the velocity is sufficient, the pressure around the point of vena contract to fall below the threshold pressure, the cavitation are generated and (3) the liquid jet expands, the pressure recovers, resulting in the collapse of the cavities and its intensity depend on the geometry of the constriction and the flow conditions of the liquid, i.e., [30].

High speed homogenizer are reported by Kumar et al. [31] who study modeling hydrodynamic cavitation. Figure 2.20 shown the cross section of the high speed homogenizer that consists of an impeller (rotor), rotating inside a cage-like stator which contains numerous openings or slots. At very high speed of rotation of rotor, the pressure of liquid flowing through the openings is very near to the vapor pressure of the liquid resulting to generate of vaporous cavities which subsequently collapse as the pressure recovers to the ambient pressure.

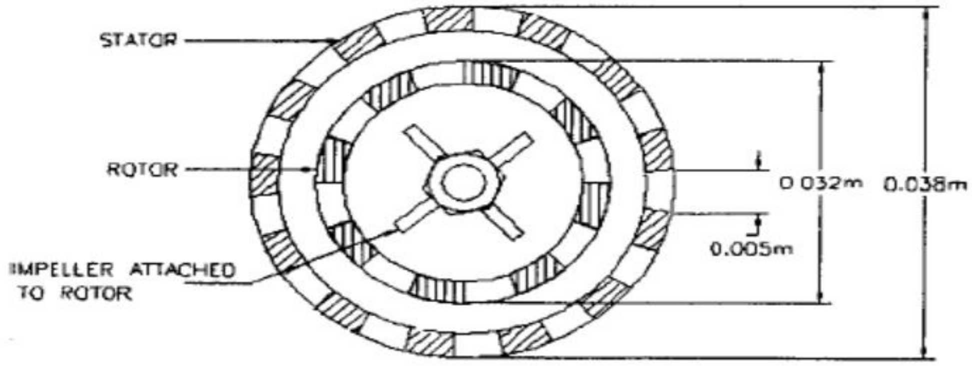


Figure 2.20 Cross section of the stator-rotor in high-speed homogenizer [31].

The cavitation phenomenon in the high-speed homogenizer (HSH) is following: [31]

- The stator of the HSH, where the velocity of the liquid is throttled through the slots in stator and moving out from the stator is calculated using the flow number $\mathrm{N_Q}$ corresponding to the rotor by using information proposed by Sano and Usui as present on equation 2.1

$$N_Q = 1.3 \left(\frac{D_r}{T}\right)^{-0.86} \left(\frac{W_r}{T}\right)^{0.82} n_r^{0.6} \tag{2.1}$$

- The velocity at the stator is calculated using the flow number as present on equation 2.2

$$V_s = \frac{N_Q N_r d_r^3}{n_{ss} H_s W_s} \tag{2.2}$$

- At the exit point of the stator, Bernoulli's principle is assumed to be valid and the pressure of the liquid leaving stator P_s can be calculated using equation 2.3, the pressure term analogous to the fully recovered downstream pressure is the atmospheric pressure P_a

$$P_s = P_a - \frac{1}{2}\rho V_s^2 \tag{2.3}$$

- The energy dissipation termP_M is obtained experimentally by calorimetric method are given in equation 2.4

$$P_M = \frac{C_p \Delta T}{\Delta t} \tag{2.4}$$

Where

D_r	is	diameter of rotor
W_r	is	height of rotor
n_r	is	the number of peripheral blades in the stator
T	is	diameter of glass beaker
N_r	is	speed of the rotor
n_{ss}	is	the number of slots in the stator
H_s	is	height of the slots in the stator
W_s	is	width of the slots in the stator

Chapter 3

Literature reviews

3.1 Conventional process and development for biodiesel production

First production of biodiesel were reported in 1981 in south-Africa and then in 1982 in Austria, New Zealand and Germany. A small pilot plant was reported in 1985 in Austria. In 1990, low speed diesel engine tests such as tractor producers as e.g. John Deere, Mercedes, Ford, Massey-Ferguson model were certified engine that is no problem. Then, Austria was made the first fuel standard in 1991. Detailed tests on product properties such as engine performance, biodegradability, emission reduction and toxicity while process economics improved as well continuously. In the beginning, the main raw materials for biodiesel production was rapeseed or canola in European, palm oil in Malaysia, soybean oil in USA and sunflower oil in France and Italy. So it can be seen that the main raw materials was locally vegetable oil in each region [32].

In the several recent years, biodiesel was produced from vegetable oil or animal fat with alcohols (mostly used methanol). The most commonly used technology for biodiesel production is transesterification of oil to produce biodiesel as main product and glycerol as by-product [33].

The transesterification can be mostly catalyzed by homogeneous or heterogeneous catalysts. The homogeneous catalyst such as NaOH, KOH (basic catalyst) was commonly used in biodiesel production due to fast of reaction rate, but the drawback of these catalyst was soap formation that found to be undesirable product because it decrease the biodiesel yield, complicate to separation and purification process [34]. The heterogeneous catalyst such as alkaline earth metal oxide – CaO was commonly used in biodiesel production. This catalyst was found to be effective catalyzed biodiesel because of low cost, easy to separate and purify process and minimal environmental impact. But the disadvantage from these catalyst was slow reaction rate compared to homogeneous catalyst [35], [36]. Table 3.1 shows the research studies of conventional process for biodiesel production [37].

Table 3.1 Research studies of conventional process for biodiesel production.

Reactor	Oil sources	Condition	Catalyst	Biodiesel yield	Reference
Mechanical stirrer	Rapeseed oil	1% of catalyst, methanol to oil molar ratio 6:1, reaction temperature 65°C, reaction time of 2 h.	KOH	95-96 %	[38]
Mechanical stirrer	Rice bran oil	0.75 % of catalyst, methanol to oil molar ratio 9:1, reaction temperature 55°C, reaction time of 1 h.	NaOH	90%	[39]
Mechanical stirrer	Sunflower seed oil	1% of catalyst, methanol to oil molar ratio 7:1, reaction temperature 60°C, reaction time of 1 h.	NaOH	90%	[40]
Fixed bed reactor	Soybean oil	6.75% Eu loading, 10% of catalyst, methanol to oil molar ratio 6:1, reaction temperature 70°C, reaction time of 8 h.	Eu_2O_3/Al_2O_3	63%	[41]

Table 3.1 Research studies of conventional process for biodiesel production (cont.).

Reactor	Oil sources	Condition	Catalyst	Biodiesel yield	Reference
Mechanical stirrer	Nahor oil	2% Li loading, 5% of catalyst, methanol to oil molar ratio 10:1, reaction temperature 65°C, reaction time of 4 h.	Li_2O/CaO	93%	[42]

From Table 3.1, the increase of methanol to oil molar ratio can shift equilibrium which provided the lower amount of catalyst or temperature were required and biodiesel yield was more than 90%. Homogeneous catalyst can accelerate faster reactions rate than that of heterogeneous catalyst due to mass transfer limitation between solid surface and reaction mixture. However, there are still took 1–2 h and 4–8 h for homogeneous and heterogeneous catalyst, respectively.

Moreover, the drawback to scale-up biodiesel production in the commercial was high biodiesel price 70-95% of total biodiesel cost was from raw material and feedstocks. Guidelines to improve economy for biodiesel production was using non-edible oil or develop new intensification method which could improve interphase mixing between two immiscible reactants to improve reaction rate [43].

3.2 Process intensification for biodiesel production

Ultrasonic irradiation has been promised as one of the appropriate technologies. It can increase interphase mixing between the phases at reaction conditions which producing high temperature and high pressure. This intensification process is based on cavitation phenomena. When the ultrasound wave passes through liquid medium and formation acoustic cavitation. This cavitation provides very high pressure and temperature in the liquid media [2].

A homogenizer which is based on rotor/stator principle to generate shear force is another interesting approach to generate emulsions of reactants. In addition, high turbulence occurs in the shear gap between rotor and stator, which provides optimum mixing of the suspension [7].

Microwave irradiation is also another interesting due to enhance reaction rate because direct energy was obtained by absorption of molecules, and then molecules was vibrated resulting to increase heat transfer [9].

Table 3.2 shows research studies performance of intensification method for biodiesel production [37].

Table 3.2 Research studies performance of intensification method for biodiesel production.

Reactor	Oil sources	Condition	Catalyst	Biodiesel yield	Reference
Ultrasonic	Refined palm oil	8% of catalyst, methanol to oil molar ratio 9:1, reaction temperature of 60°C, reaction time of 1 h, ultrasonic power of 60%, ultrasonic frequency of 20 kHz	CaO	92.7%	[5]
Ultrasonic	Canola oil	3% of catalyst, methanol to oil molar ratio 9:1, reaction temperature of 60°C, reaction time of 75 min, ultrasonic power of 40W, ultrasonic frequency of 20 kHz	CaO	95.5%	[44]

Table 3.2 Research studies performance of intensification method for biodiesel production (cont.).

Reactor	Oil sources	Condition	Catalyst	Biodiesel yield	Reference
Ultrasonic	Jatropha curcus oil	3% of catalyst, methanol to oil molar ratio 9:1, reaction temperature of 50 - 70^{o}C, reaction time of 15 min, ultrasonic power of 200W, ultrasonic frequency of 24 kHz	Na/SiO$_2$	98.53%	[45]
Ultrasonic	Triolein	1% of catalyst, methanol to oil molar ratio 6:1, reaction temperature of 25^{o}C, reaction time of 15 min, ultrasonic power of 1200 W, ultrasonic frequency of 40 kHz	KOH	95%	[46]
Homogenizer	Refined and bleached palm oil	0.41% of catalyst, methanol to oil molar ratio 5.5:1, reaction temperature of 55 ^{o}C, reaction time of 15 min	NaOH	99%	[8]
Microwave	soybean oil	3% of catalyst, methanol to oil molar ratio 7:1, reaction temperature of 65^{o}C, reaction time of 45 min	Nano CaO	91.7%	[47]
Microwave	Palm olein oil	15% of catalyst, methanol to oil molar ratio 18:1, reaction time of 4 min, microwave power of 900W	CaO	96.7%	[48]

Table 3.2 Research studies performance of intensification method for biodiesel production (cont.).

Reactor	Oil sources	Condition	Catalyst	Biodiesel yield	Reference
Combination of microwave and ultrasound	Waste vegetable oil	1% of catalyst, methanol to oil molar ratio 6:1, reaction time of 2 min, reaction temperature of 68.8°C, MW/US power of 100/100 W	NaOH	97%	[11]
Hydrodynamic Cavitation	Waste cooking oil	1% of catalyst, methanol to oil molar ratio 6:1, reaction time of 15 min, reaction temperature of 60°C	KOH	98.1%	[49]

From Table 3.2, ultrasonic irradiation can increase mass transfer resulting to fast reaction rate. The optimum condition were methanol to oil molar ratio of 9, catalyst loading 3-8%wt, reaction temperature of 60°C and reaction time were in range 15–75 min in both caste of homogeneous and heterogeneous catalyst. Homogenizer can also increase mass transfer. Over 95% of biodiesel yield was obtained using 0.41%wt of catalyst, methanol to oil molar ratio of 5.5, reaction temperature of 55°C and reaction time of 15 min. Microwave irradiation can enhance heat transfer but could not increase mass transfer resulting to require more catalyst and methanol to shift equilibrium. Therefore, the combination of microwave and ultrasonic can increase heat and mass transfer resulting to fast reaction rate. The reaction was took 2 min to reach equilibrium (97% of biodiesel yield) at 1%wt of catalyst and methanol to oil molar ratio of 6.

In addition, Chuah et al. [49] reported in term of biodiesel yield efficiency using hydrodynamic cavitation reactor. It was found that 12.50×10^{-4} g/J was obtained from transesterification of waste cooking oil using potassium hydroxide as a catalyst using methanol to oil molar ratio of 6, catalyst loading 1%wt, reaction temperature of 60°C and reaction time of 15 min. Biodiesel yield was produced up to 98.1% in only 15 min.

This research is aim to study key factors of process intensification method for improvement of biodiesel production base on energy consumption under condition of methanol to palm oil molar ratio of 6, catalyst loading of 1%wt NaOH, reaction temperature of 60°C and reaction time of 3 h for homogeneous catalyst, and methanol to palm oil molar ratio of 9, catalyst loading of 10%wt CaO, reaction temperature of 60°C and reaction time of 4 h.

Chapter 4
Experimental

4.1 Materials

4.1.1 Reactants

4.1.1.1 Refine Palm oil was purchased from a local market in Thailand.

4.1.1.2 Methanol (≥99.8% purity) was purchased from Avantor Performance Materials, lnc.

4.1.2 Catalysts

4.1.2.1 Sodium hydroxide (99.0% purity) was purchased from Merck Company Limited.

4.1.2.2 Calcium oxide (96% purity) fine powder was purchased from Sigma-Aldrich.

4.1.3 Analytical agents

4.1.3.1 Heptane (99.5% purity) was purchased from Ajax Finechem Pty Ltd.

4.1.3.2 Methyl heptadecanoate (≥99.0% purity) was purchased from Sigma-Aldrich.

4.2 Equipment

4.2.1 Equipment used in biodiesel synthesis

4.2.1.1 Equipment used in synthesis of biodiesel in batch reactor is shown in Figure 4.1 which consists of (1) heater (2) oil bath (3) 3-neck-bottom flask (4) magnetic stirrer (5) reaction mixture (6) condenser (7) thermometer.

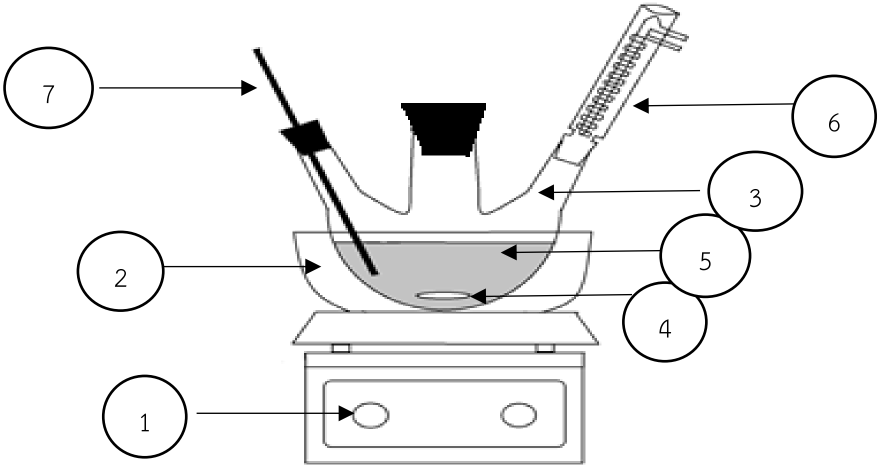


Figure 4.1 Batch reactor.

4.2.1.2 Direct ultrasonic horn (batch reactor)

The equipment as shown in Figure 4.2 which consists of (1) generator (2) transducer housing (3) detachable horn and (4) fan will apply to batch reactor. Two frequency of ultrasonic horn was applied to the reactor including of 20 and 40 kHz.

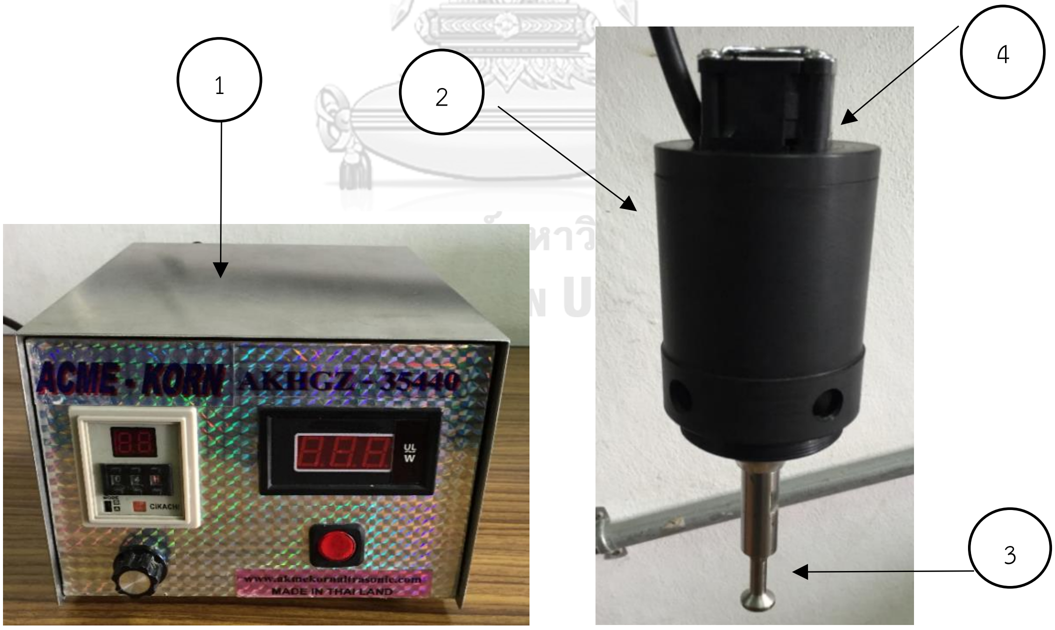


Figure 4.2 Direct ultrasonic systems.

4.2.1.3 Homogenizer (batch reactor)

The equipment as shown in Figure 4.3 consist of (1) power drive unit (2) knurled screw (3) rotor and (4) stator will apply to batch reactor.

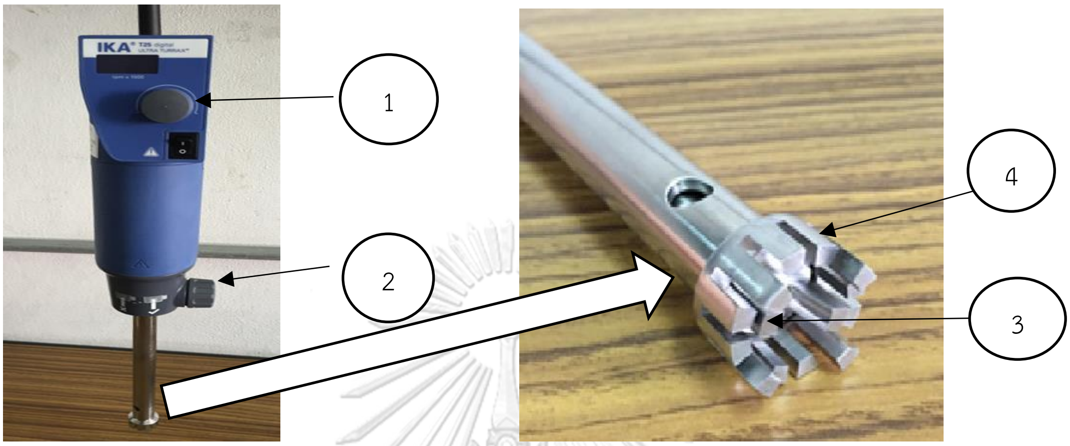


Figure 4.3 Homogenizer systems.

4.2.1.4 Combination of ultrasonic and microwave (semi-batch reactor)

The equipment as shown in Figure 4.4 consist of (1) ultrasonic transducer (2) thermocouple (3) household microwave oven and (4) quartz tube.

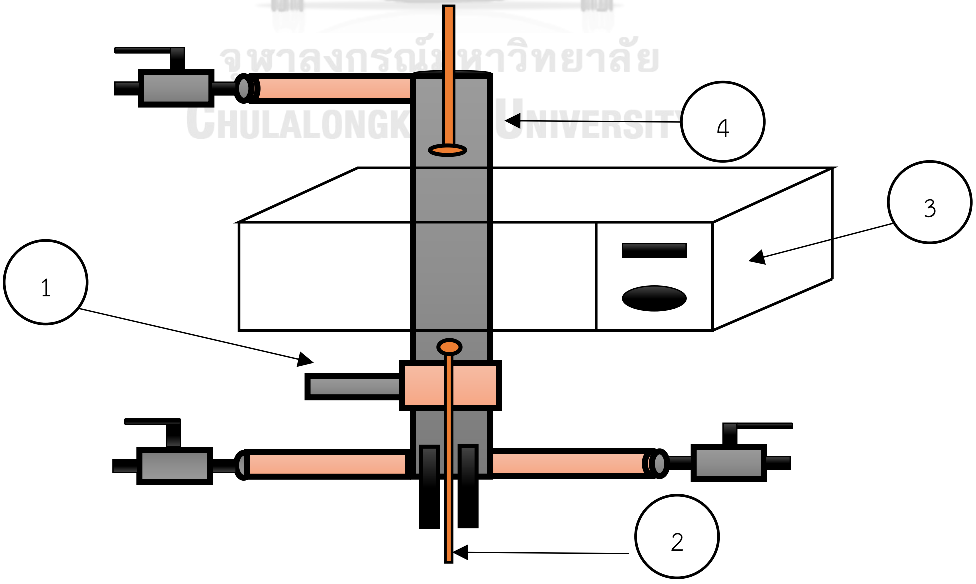


Figure 4.4 Combination of ultrasonic and microwave systems.

4.3 Experimental setup

4.3.1 Catalyst synthesis method are follows:

Calcium oxide was calcined in muffle furnace with heating rate 10^{o}C/min from room temperature to 900^{o}C and holding for 5 h, and then kept it in desiccator before use.

4.3.2 Biodiesel synthesis from transesterification of palm oil

In case of batch reactor, the fixed amount of catalysts (1%wt of NaOH or 10%wt of CaO catalyst) was mixed with methanol before transferring into preheated oil. A 3-neck-bottom flask was immersed into an oil bath to control temperature of reaction mixture at 60^{o}C. The 3-neck-bottom flask was plugged with a condenser and thermometer. A magnetic stirrer with stirring speed of 800 rpm, 20 kHz of direct ultrasonic probe and homogenizer with the same power of 80 W were used to study mixing characteristics under the similar condition. The reaction times are 3 h and 4 h for homogeneous and heterogeneous catalysts, respectively. In case of continuous reactor, also used the same condition with batch reactor except power input. 1000 and 2000 mL of 3-neck-bottom flask was used to kept oil and methanol, respectively. The reactant was pumped by peristaltic pump into reactor. The reaction product from both case resulting reaction mixture was transferred and centrifuged to separate fatty acid methyl ester out from reaction mixture before being analyzed by gas chromatograph (GC).

4.3.3 Enhance biodiesel production rate by using direct ultrasonic probe reactor with frequency of 20 kHz (US 20 kHz), ultrasonic probe reactor with frequency of 40 kHz (US 40 kHz), and homogenizer reactor (HZ) as compared to mechanical stirrer reactor (MS), for both cases of NaOH and CaO catalyzed transesterification of palm oil on the biodiesel yield based on the energy consumption as following experiment on section 4.3.2.

4.3.3 Study the effect of reaction mixture volume (165, 500 and 1000 mL) on biodiesel yield and the biodiesel production efficiency using direct ultrasonic probe reactor, homogenizer reactor and mechanical stirrer reactor under the similar transesterification of palm oil as following experiment on section 4.3.2.

4.3.4 Enhance the biodiesel production rate by using the combination of ultrasonic and microwave for NaOH catalyzed transesterification of palm oil in term of heat and mass transfers as following experiment on section 4.3.2.

4.4 Biodiesel yield calculation

The biodiesel yield were analyzed by Shimadzu GC-2010 Plus with DB-WAX capillary column and detected by flame ionization detector (FID). Methyl heptadecanoate was used as internal standard and the biodiesel yield was determined according to EN14103 standard as shown in Eq. (4.1)

$$\text{Biodiesel yield}\ (\%) = \frac{(\sum \mathrm{A}) - \mathrm{A_{IS}}}{\mathrm{A_{IS}}} \times \frac{\mathrm{C_{IS}}\ \mathrm{x}\ \mathrm{V_{IS}}}{\mathrm{m_s}} \times 100\% \qquad (4.1)$$

Where $\sum \mathrm{A}$ Total area of peak

$\mathrm{A_{IS}}$ Area of methyl heptadecanoate

$\mathrm{C_{IS}}$ Concentration of methyl heptadecanoate (mg/mL)

$\mathrm{V_{IS}}$ Volume of methyl heptadecanoate (mL)

$\mathrm{m_s}$ Mass of biodiesel sample (mg)

4.5 Yield efficiency calculation

The yield efficiency is defined in Eq. (4.2) [49]

$$\text{Yield efficiency} = \frac{\text{Amount of product produced (g)}}{\text{Power supplied}\ \left(\frac{\mathrm{J}}{\mathrm{s}}\right) \text{x reaction time(s)}} \qquad (4.2)$$

Chapter 5

Results and Discussion

5.1 Characterization of CaO.

Figure 5.1 shows XRD patterns of uncalcined and calcined CaO catalysts. The results showed that calcium hydroxide ($Ca(OH)_2$) peaks were present only for uncalcined CaO at 2 θ = 28.8^0, 34.1^0, 47.1^0 and 50.8^0. While for calcined CaO at 900°C, CaO peaks were observed at 2θ = 32.3^0, 37.4^0 and 53.9^0. It was noted that calcination temperature of 900°C can completely eliminate calcium hydroxide which indicated the active phase to catalyzed transesterification of palm oil [50].

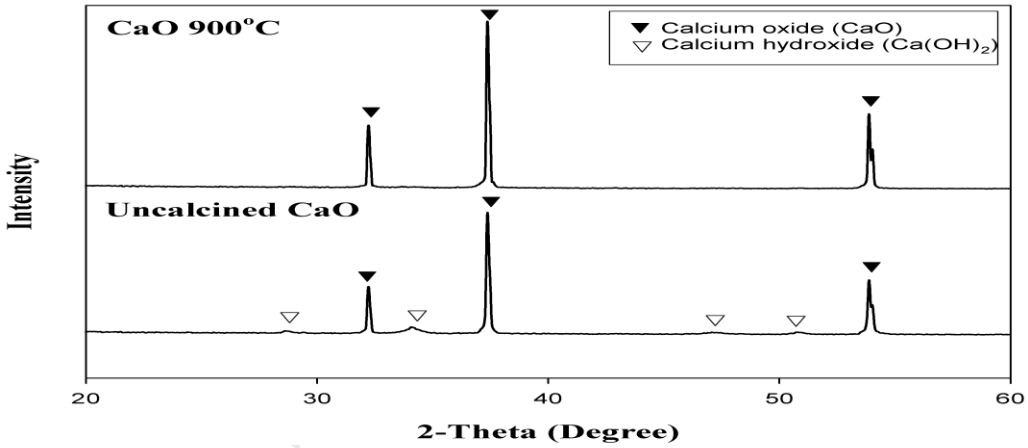


Figure 5.1 The XRD patterns of calcium oxide catalyst (calcined at 900°C and uncalcined).

There are two sections to enhance the biodiesel performance in the multifunctional reactor including of 1) using cavitational reactor to enhance mass transfer and 2) using the combination of microwave and ultrasonic to enhance mass and heat transfer of transesterification of palm. Moreover, the biodiesel yield and yield efficiency obtained from multifunctional reactor have been compared to the conventional biodiesel reactor (mechanical stirrer reactor, MS).

5.2 Effect of biodiesel yield on various cavitational reactors compared to conventional reactor.

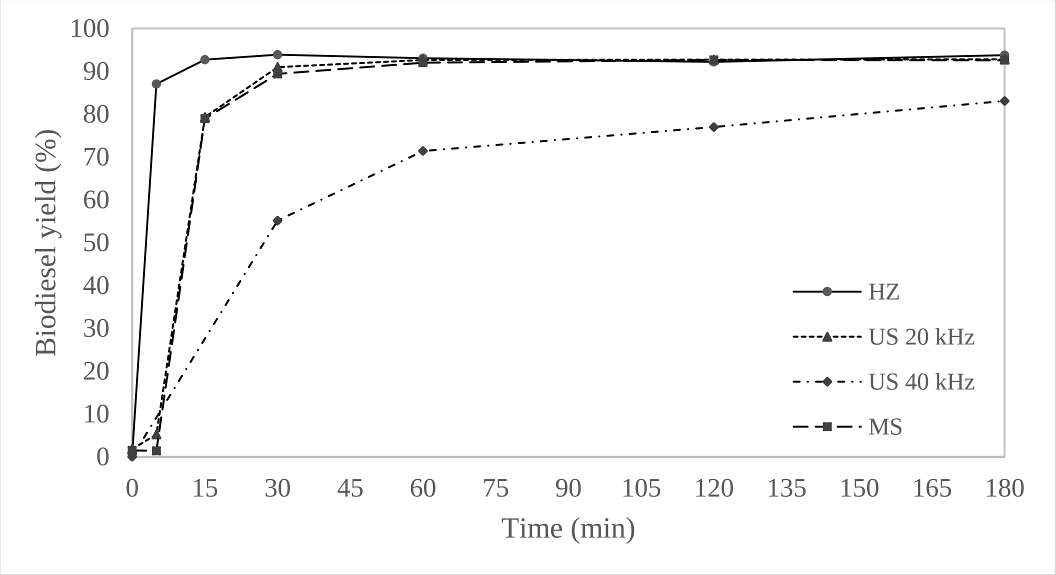


Figure 5.2 Effect of biodiesel yield on various cavitational reactors compared to conventional reactor (MS) at small reaction mixture volume of 165 mL (Homogeneous catalyst).

The effect of biodiesel yield on various cavitational reactors compared to conventional reactor (magnetic stirrer, MS) for reaction mixture volume of 165 mL with using NaOH as catalyst on transesterification of palm oil was investigated as illustrated in Figure 5.2. The reaction was carried out at a reaction temperature of 60 °C, methanol to oil molar ratio 6:1, NaOH loading 1%wt, and reaction time of 3 h. The cavitational reactors were including of HZ, US 20, US 40 kHz and MS. The result showed that HZ can produce biodiesel yield of 87.06% and 92.71% in 5 and 15 min, respectively while US 20 kHz produced biodiesel yield of 90.98% in 30 min. US 40 kHz provided the lower biodiesel yield compared to US 20 kHz. This is more likely due to the bubble size was decreased when increases ultrasonic frequency resulting to reduce intensity and shear forces from the cavitation collapse [51]. MS obtained the similar biodiesel yield (at the same reaction time) to US 20 kHz while gave the slower reaction rate compared to HZ. However, biodiesel yield obtained from all reactors also reach equilibrium at 60 min except US 40 kHz. Based on this reaction mixture volume (165mL), HZ, US 20 kHz and MS can provide the sufficient mixing phenomena to overcome mass transfer limitation between two immiscible liquids (palm oil and methanol) and no obstacle for homogeneous catalyst (NaOH).

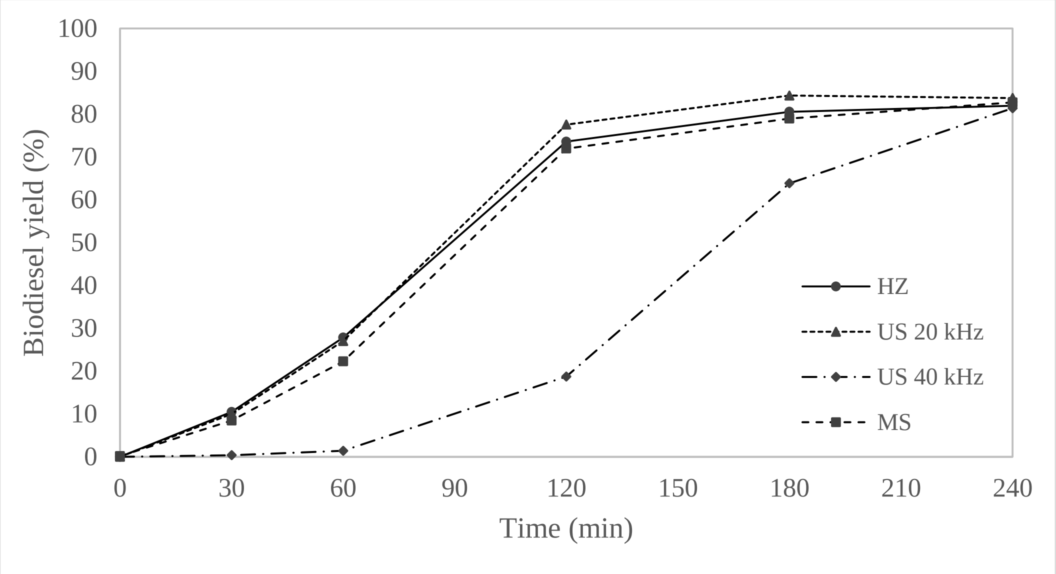


Figure 5.3 Effect of biodiesel yield on various cavitational reactors compared to conventional reactor (MS) at small reaction mixture volume of 165 mL (Heterogeneous catalyst).

Figure 5.3 demonstrates the effect of biodiesel yield on various cavitational reactors compared to conventional reactor (MS) for heterogeneous catalyst (CaO). Experiments were carried out at a reaction temperature of 60°C, methanol to oil molar ratio 9:1, catalyst loading 10%wt, and reaction time of 4 h. This selected condition was due to the fact that is major problems of heterogeneous catalyst is the formation of three phases leading to slow reaction rate [52]. Increase amount of catalyst can be accelerated reaction rate as well as increase amount of methanol, which in turn to shift the reaction equilibrium to the right-hand side and more contact with catalyst [5]. Liu et al. [53] have been investigated transesterification of soybean oil to biodiesel using CaO. It was found that biodiesel yield increased from 55% to 90% when amount of CaO was increased from 2%wt to 12%wt. Increase amount of catalyst could be promote more active site, however, excess amount of catalyst also reduce mass transfer in the reaction mixture. Moreover, methanol is high vapor pressure which help cavitation phenomena in the reaction mixture. Figure 5.3 indicates that the induction period was still observed for all reactor. Biodiesel yield obtained from all reactors from

0 to 180 min was similar trend. US 20 kHz give the highest biodiesel yield as well as HZ while biodiesel yield obtained from US 40 kHz was lowest. As described earlier that is the bubble size was decreased when increases ultrasonic frequency which in turns reduces intensity and shear forces generated by the cavitation collapse with the control input power of 80 W could not enough to increase degree of mixing in the reaction mixture to turbulence flow for US 40 kHz. To obtain maximum performance of using US 40 kHz, the control of input power should be increased. However, at 240 min of reaction time, biodiesel yield was carried out in all reactors reach the similar equilibrium. Vujicic et al. [54] reported that when using calcium oxide the induction period was expected due to catalytic active sites formation. First hypothesis, induction period from internal mass transfer between active site and reaction mixture. Another hypothesis, calcium oxide was an active phase of transesterification at the first reaction period. Then, glycerol was produced during reaction and react with calcium oxide to form a new active phases (calcium diglyceroxide) [55]. Moreover, biodiesel produced during reaction could be solvent to increase miscibility between oil and methanol [56]. Then, the solubility of reaction mixture was increased resulting to faster the reaction rate on biodiesel production.

When consideration the effect of using homogeneous and heterogeneous to catalyze transesterification of palm oil. The obtained biodiesel yield using NaOH is higher than that of CaO catalyst due to its similar phase as reactant, but the drawbacks from homogeneous catalyst is difficult to separate and required high amount of water for purification of biodiesel [57]. Using HZ was appropriated to carry out NaOH catalyzed transesterification of palm oil while using HZ to carry out CaO catalyzed transesterification was lower biodiesel yield compared to US 20 kHz. This might be attributed to the velocity of reaction mixture in HZ was decreased when using solid catalyst resulting to decrease the generation of cavitation [30]. For liquid-solid reaction, the caviation phenomena can produce one positive and one negative effect including of providing sufficient cavitation intensity (increase mass transfer between two immiscible liquid and active site of catalyst) as well as destruction of structural and make mechanical defect of solid catalyst [58]. US 20 kHz, US 40 kHz and MS gave the similar biodiesel yield for homogeneous catalyst. On the other hand, for heterogeneous catalyst, reaction carried out in US seems to be faster reaction rate than that of MS due to US can improved mass transfer to produce the smaller droplets in reactant phases, resulting to drastic increase in an interfacial area [59].

5.3 Effect of reaction mixture volume on biodiesel yield.

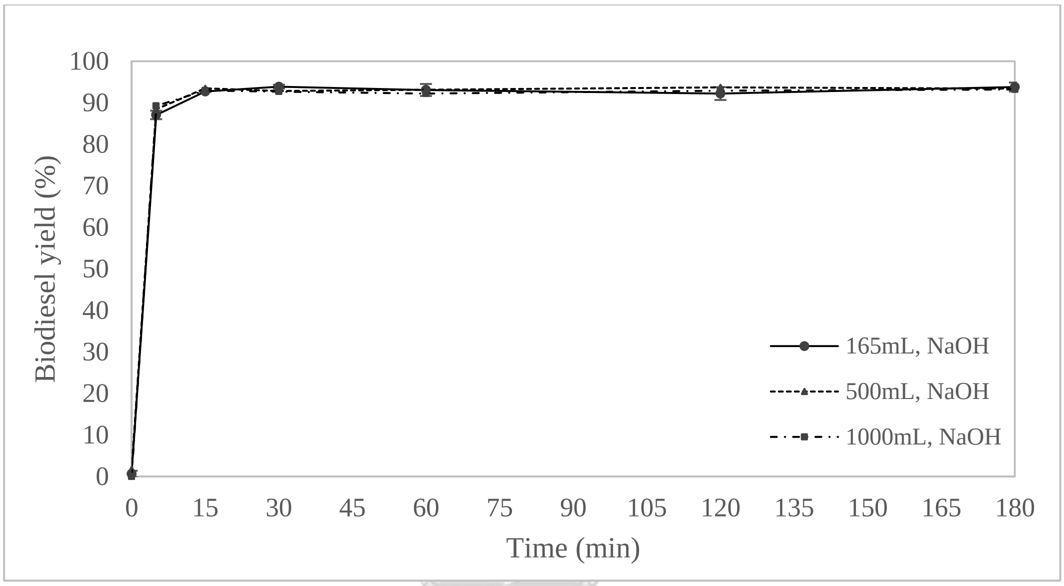


Figure 5.4 Effect of reaction mixture volume on biodiesel yield using homogenizer reactor (Homogeneous catalyst).

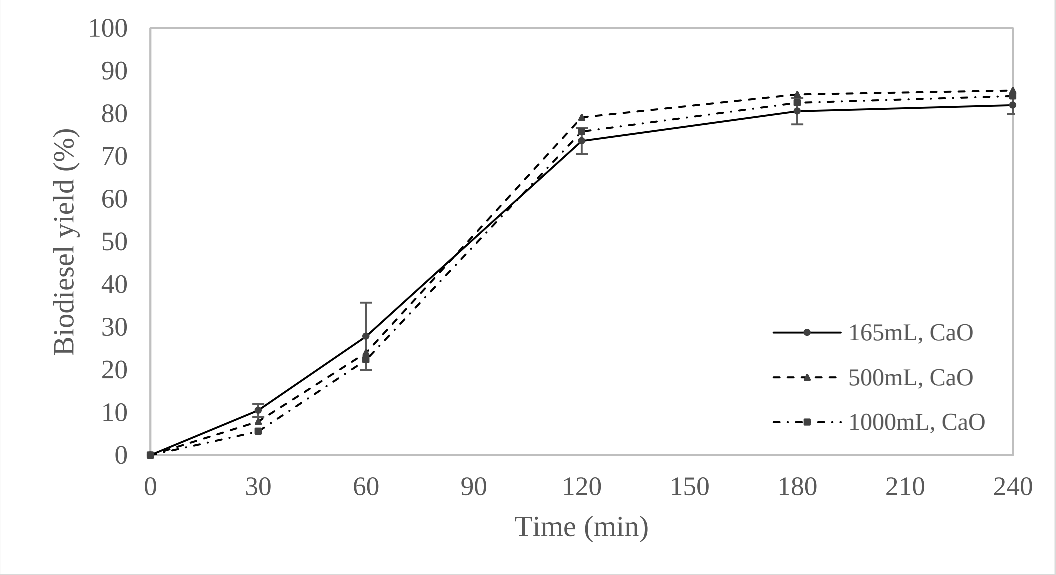


Figure 5.5 Effect of reaction mixture volume on biodiesel yield using homogenizer reactor (Heterogeneous catalyst).

The aim of this experimental was to illustrate the highest performance of each mixing type of reactor for biodiesel production using both homogenous and heterogeneous catalyst. Most of previous literature only concern the effect of power on the biodiesel yield and aim to reduce the reactor volume to obtain higher biodiesel yield. However, there are no literature proposed on the use of maximum reaction mixture volume to obtain the highest biodiesel yield. Therefore, the effect of reaction mixture volume on biodiesel yield was investigated by using the same condition and same input electrical energy. The reaction mixture volume was increased from 165 to 500 and 1000 mL which refer to about 3 and 6 times, respectively. Figure 5.4 and 5.5 demonstrate the effect of reaction mixture volume on using HZ for homogeneous and heterogeneous catalyst. The results showed that biodiesel yield along the reaction time from HZ was similar for all reaction mixture volume from 165 to 1000 mL. This attributed to the fact that, as the rotor/stator principle generates high shear force which occurs in the shear gap between rotor and stator leading to emulsifier of the suspension. This is more likely due to velocity of the liquid with a corresponding decrease in the local pressure, if the local pressure fall below the vapor pressure of methanol, then the cavitation are generated [49]. This results was comparable to the results obtained by Mohod et al. [60] who studied the high speed homogenizer to produce biodiesel of 92.3% using sunflower oil with 3% of KOH, molar ratio of methanol to oil of 12 and reaction temperature of 50°C along the reaction time of 2 h. Our research used lower catalyst and methanol to oil molar ration than the results from Mohod et al. Joshi et al. [61] who reported that using high speed homogenizer to produce biodiesel in the presence of heterogeneous catalyst (CaO). The maximum biodiesel yield was 84% at catalyst loading of 3%wt, molar ratio of methanol to oil of 10, reaction temperature of 50°C and reactor size of 300 mL with using speed of rotation of 12,000 rpm. Our research used higher catalyst but lower methanol than that of Joshi et al. work. Even though, based on the same input power, increase of reaction mixture volume from 165 to 1000 mL results to obtain similar biodiesel yield in HZ.

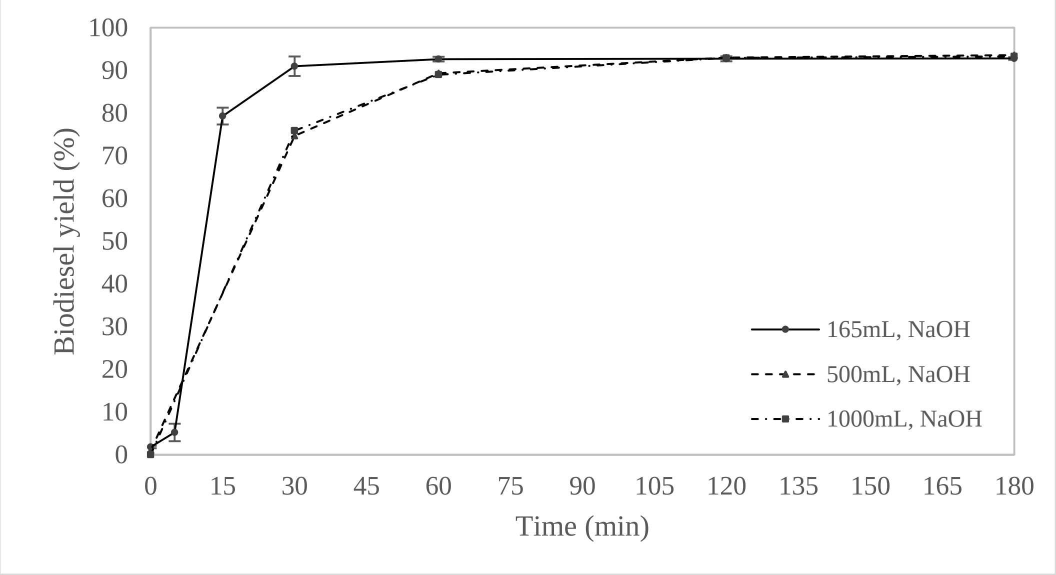


Figure 5.6 Effect of reaction mixture volume on biodiesel yield using 20 kHz of ultrasonic reactor (Homogeneous catalyst).

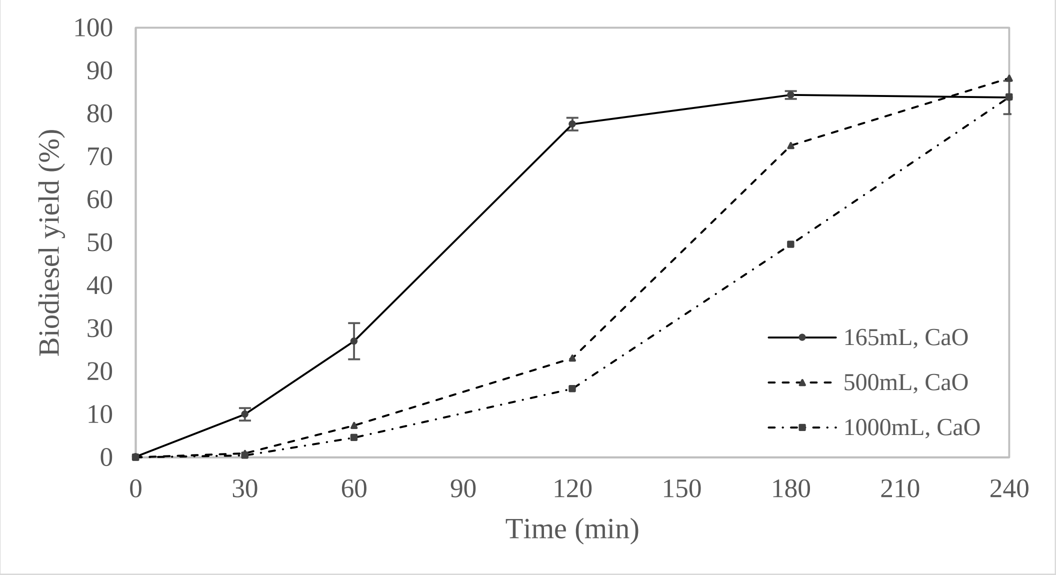


Figure 5.7 Effect of reaction mixture volume on biodiesel yield using 20 kHz of ultrasonic reactor (Heterogeneous catalyst).

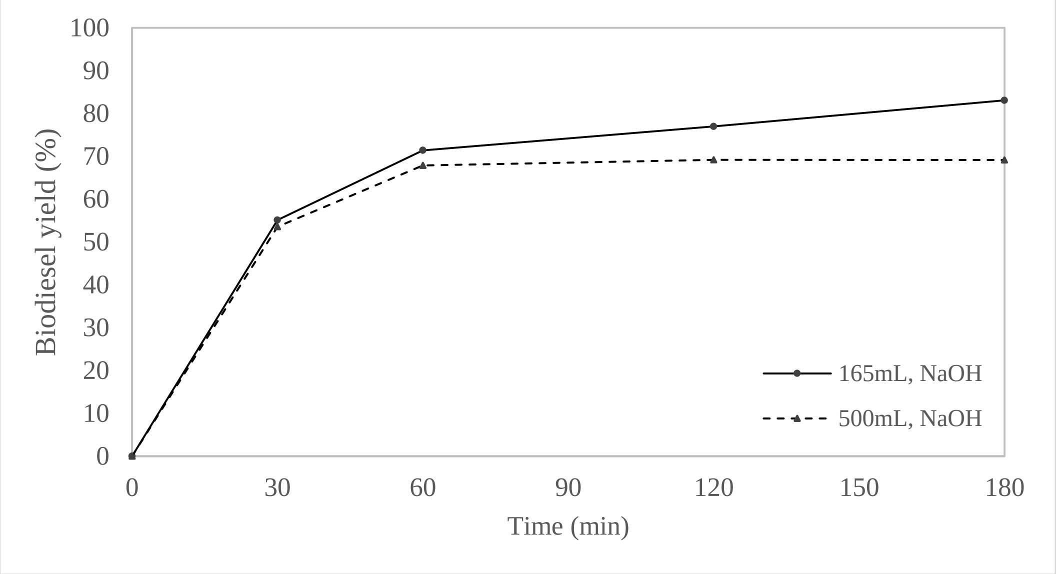


Figure 5.8 Effect of reaction mixture volume on biodiesel yield using 40 kHz of ultrasonic reactor (Homogeneous catalyst).

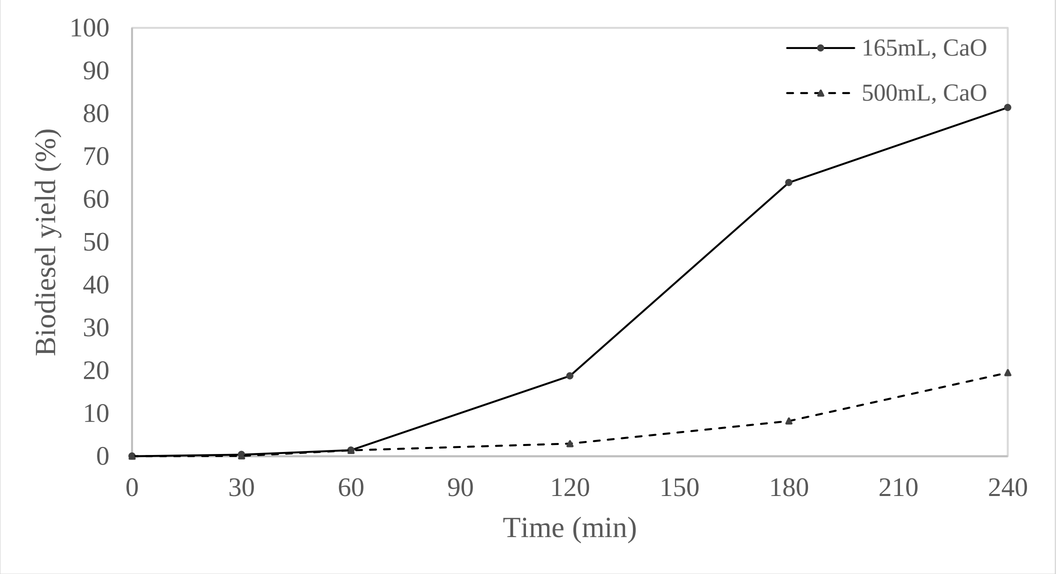


Figure 5.9 Effect of reaction mixture volume on biodiesel yield using 40 kHz of ultrasonic reactor (Heterogeneous catalyst).

Figure 5.6 demonstrates the effect of reaction mixture volume on US 20 kHz using NaOH catalyst. The results showed that when increase reaction mixture volume to 3 and 6 times resulting to decrease reaction rate in the beginning of reaction time. However, the biodiesel yield was reach to reaction equilibrium at the same time of 60 min for 500 and 1,000 mL except the small reaction mixture volume of 165 mL (reaction was reach equilibrium at 30 min). On the other hand, using heterogeneous catalyst which can be clearly seen in Figure 5.7. The increase in reaction mixture volume to 3 and 6 times reduce biodiesel yield to 3 and 6 times. Using US 40 kHz for NaOH catalyst was depicted in Figure 5.8, the results showed that increasing reaction mixture volume, the biodiesel yield was decreased due to bubble size was decreased when increasing ultrasonic frequency which in turns reduces intensity and shear forces generated by the cavitation collapse. Reaction mixture volume effect can be seen significant difference in biodiesel yield for CaO catalyst (Figure 5.9). Biodiesel yield was decreased of 8 times with increasing of 3 times of reaction mixture volume. Furthermore, higher frequency required higher power to obtain maximum biodiesel yield and correlation between ultrasonic power input and amplitude of the wave can be seen in our previous research [62]. Kumar et al. [45] reported that when increasing ultrasonic power, more number of cavitation bubbles are generated leading to increased biodiesel yield. Mahamuni et al. [63] also investigated the effect of frequency of ultrasonic on biodiesel yield. The increase in the frequency of ultrasonic can decrease the size of bubble leads to decrease implosion time of cavitation bubble resulting to slow reaction rate.

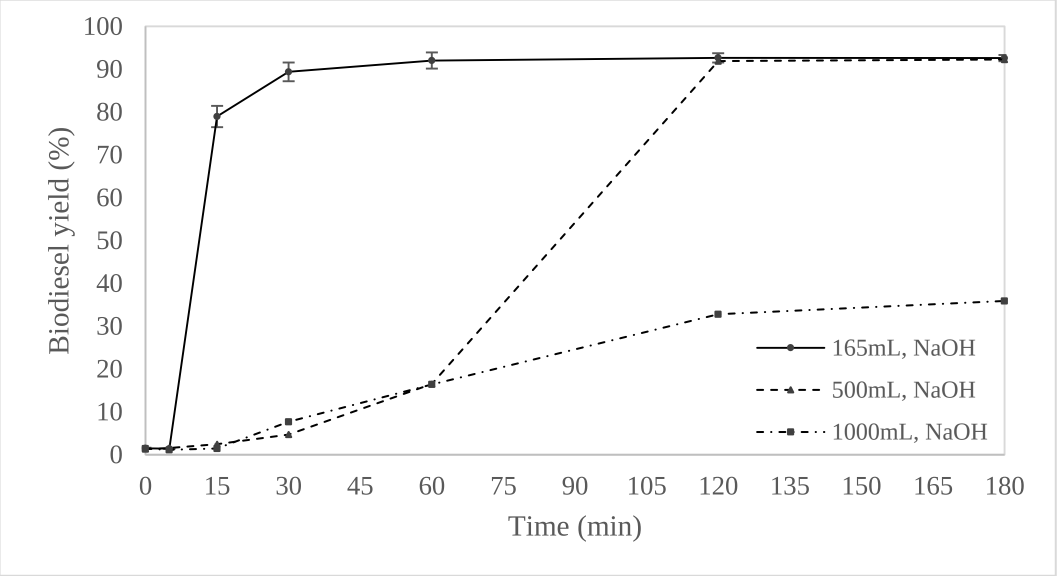


Figure 5.10 Effect of reaction mixture volume on biodiesel yield using magnetic stirrer reactor (Homogeneous catalyst).

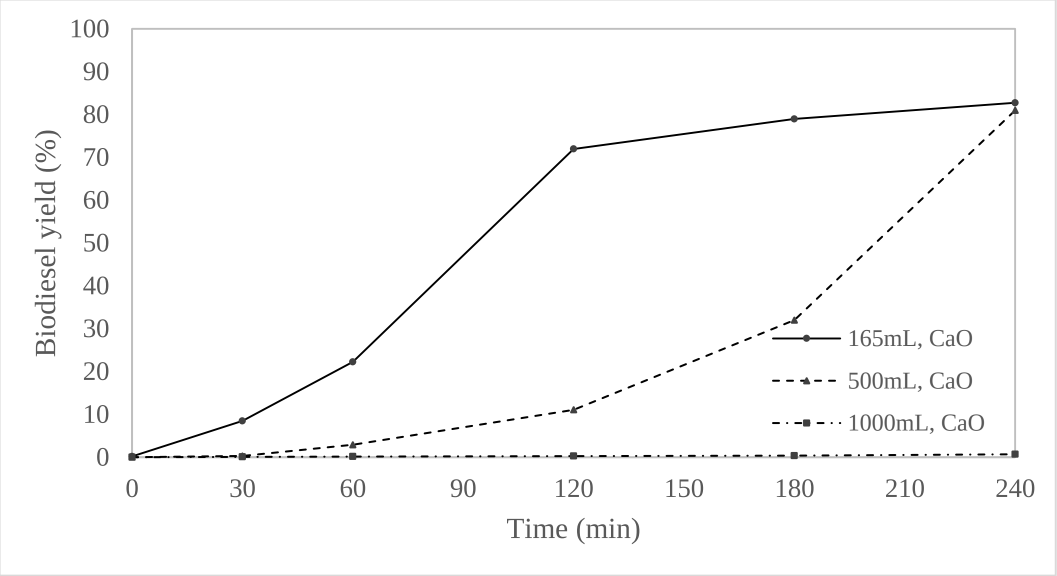
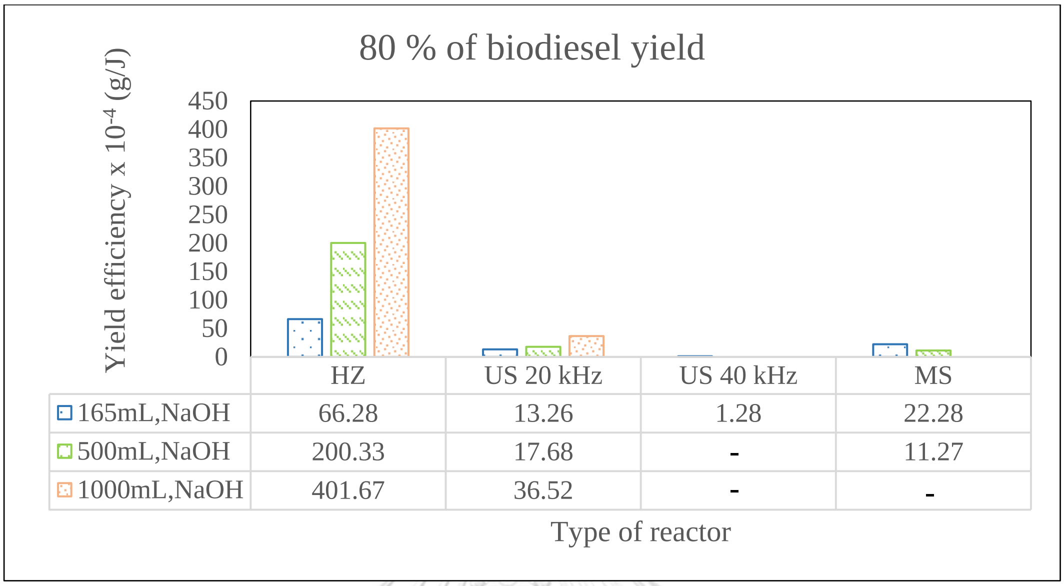


Figure 5.11 Effect of reaction mixture volume on biodiesel yield using magnetic stirrer reactor (Heterogeneous catalyst).

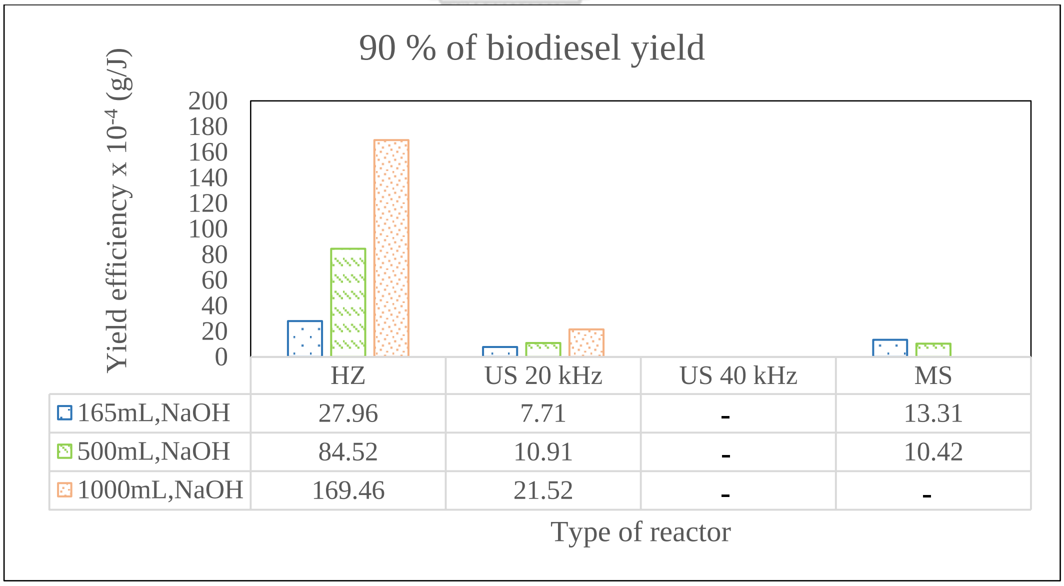
Figure 5.10 and 5.11 illustrate the effect of reaction mixture volume using MS. The results showed that biodiesel yield was significantly decreased with increase reaction mixture volume, especially for heterogeneous catalyst. Using of conventional mixing as blade or turbine or magnetic bar requires the appropriate the ratio of liquid level to blade diameter, but increase the reaction mixture volume leading to increase to liquid level. Therefore, the ratio of liquid level and blade diameter was changed resulting to lower biodiesel yield.

5.4 Effect of various cavitational reactors on yield efficiency based on 80% of biodiesel yield.



	HZ	US 20 kHz	US 40 kHz	MS
☐ 165mL,NaOH	66.28	13.26	1.28	22.28
☐ 500mL,NaOH	200.33	17.68	-	11.27
☐ 1000mL,NaOH	401.67	36.52	-	-

Figure 5.12 Effect of various cavitational reactors on yield efficiency based on 80% of biodiesel yield in homogeneous catalyst.



	HZ	US 20 kHz	US 40 kHz	MS
☐ 165mL,NaOH	27.96	7.71	-	13.31
▨ 500mL,NaOH	84.52	10.91	-	10.42
☐ 1000mL,NaOH	169.46	21.52	-	-

Figure 5.13 Effect of various cavitational reactors on yield efficiency based on 90% of biodiesel yield in homogeneous catalyst.

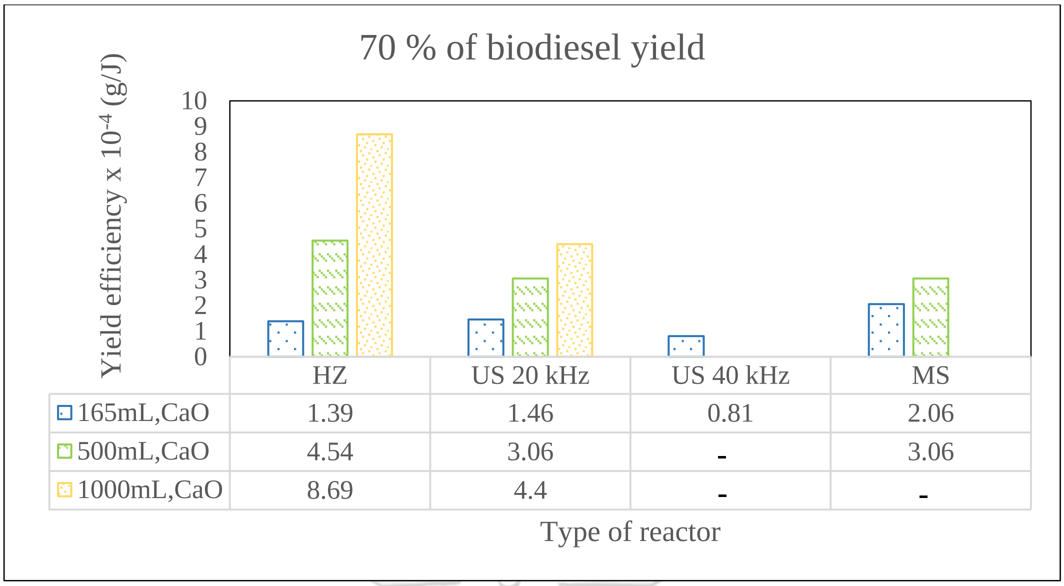


Figure 5.14 Effect of various cavitational reactors on yield efficiency based on 70% of biodiesel yield in heterogeneous catalyst.

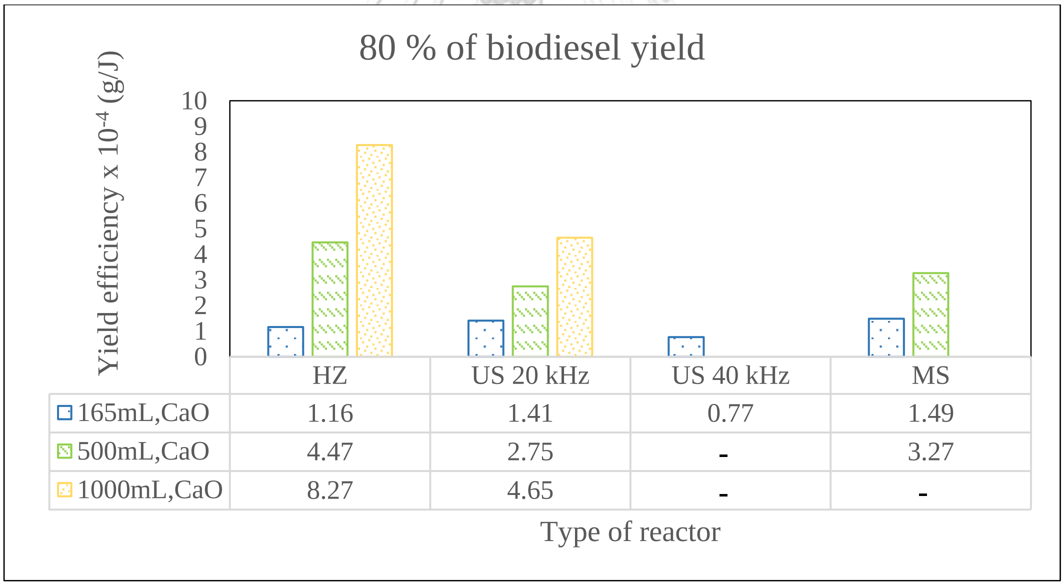


Figure 5.15 Effect of various cavitational reactors on yield efficiency based on based on 80% of biodiesel yield in heterogeneous catalyst.

From the above section, biodiesel yield was the major parameter to evaluate the effect of mixing type on the biodiesel production using both homogeneous and heterogeneous catalyst. However, it could not be considered only one parameter as biodiesel yield. Therefore, yield efficient was used to evaluate the reactor performance based on energy usage, amount of biodiesel production and also reaction time. The yield efficiency was used for consideration whether cavitational reactor are suitable for biodiesel production based on the similar power input. The measurement of total energy consumption was included power input from each cavitational reactor and heater. Figure 5.12 and 5.13 illustrate the effect of mixing type in the various cavitational reactors on yield efficiency in homogeneous catalyst. The selection of biodiesel yield for calculating yield efficiency is refer to maximum of biodiesel yield using NaOH and CaO catalyst [46, 59, 61, 64]. Then, the yield efficiency was calculated based on the selected biodiesel yield of 80% and 90%. As can be seen, HZ was the most suitable multifunctional reactor for biodiesel production providing the maximum yield efficiency of 401.67 x 10^{-4} g/J and 169.46 x 10^{-4} g/J for the reaction mixture of 1000 mL based on 80% and 90% of biodiesel yield, respectively. This results indicated HZ can eliminate mass transfer using the lower energy consumption as seen in Figure 5.4. Biodiesel yield of 87% was achieved in 5 min in all reaction mixture volumes and provided the higher yield efficiency than that obtained from Chuah et al. [49]. They studied the intensification of biodiesel synthesis from waste cooking oil in a hydrodynamic cavitation reactor using potassium hydroxide as a catalyst. They reported that biodiesel yield efficiency was 12.50 x 10^{-4} g/J under similar condition except power (maximum power output of 4 kW). Biodiesel yield efficiency from this work also provided higher yield efficiency than that obtained from Ghayal et al. [65]. They reported that biodiesel yield efficiency was 12.8 x 10^{-4} g/J with methanol to oil molar ratio of 6, catalyst loading of 1%wt, reaction temperature of 60°C, reaction time of 10 min and power of 7.5 kW. As can be seen, the yield efficiency equation (4.2) was divided by energy required for reaction. In this work the energy required for reaction was only 80 W, is lower than the previous work and also reaction mixture volume resulting to higher yield efficiency than the others.

The yield efficiency obtained from US 20 kHz based on 90% of biodiesel yield was lower than 80% of biodiesel yield. This is attribute to the fact that this condition require more reaction time to achieve 90% of biodiesel yield leading to more input power was used. Yield efficiency based on 80% of biodiesel yield were 13.26, 17.68

and 36.52 x 10^{-4} g/J and yield efficiency based on 90% of biodiesel yield were 7.71, 10.91 and 21.52 x 10^{-4} g/J for reaction mixture of 165, 500 and 1000 mL, respectively. It can be conclude that US can be increase the reaction mixture volume, however, its lower results than HZ. Gole et al. [66] reported that hydrodynamic cavitation have more efficient as compared to ultrasonic reactor. The yield efficiency was 0.16 x 10^{-4} g/J for ultrasonic reactor and 13.5 x 10^{-4} g/J for hydrodynamic cavitation which corresponding to Gogate work [58]. They reported that yield efficiency of ultrasonic reactor were 0.86 x 10^{-4} g/J and 33.7 x 10^{-4} g/J for hydrodynamic cavitation reactor. For US 40 kHz, the yield efficiency was 1.28 x 10^{-4} g/J for 165 mL. When increased reaction mixture up to 500 mL yield efficiency would not use to calculate due to maximum yield was 69.19% as lower the requirement.

The yield efficiency of MS based on 80 and 90% of biodiesel yield. The results showed that yield efficiency was decreased when increased reaction mixture. This is due to the use of conventional mixing blade or turbine or magnetic bar was appropriate with the ratio of liquid level to blade diameter to get optimum performance. For example, to place the agitator higher or lower in the tank, or much deeper tank may be needed to achieve the desired process result [67].

For CaO catalyst as depicted in Figure 5.14 and 5.15. The yield efficiency was calculated based on 70% and 80% of biodiesel yield. It indicated that the induction period was observed for all mixer types resulting to lower yield efficiency than that of NaOH catalyst. However, biodiesel yield obtained from CaO catalyzed transesterification of palm oil using HZ was higher than the other reactors. This results also confirm that the highest performance for biodiesel in term of energy input, short reaction time, and large reaction mixture volume production can be produce in HZ.

This section will be show the combination of microwave and ultrasonic to enhance mass and heat transfer for transesterification of palm oil.

5.5 Enhance biodiesel yield using the combination of microwave and ultrasonic reactor

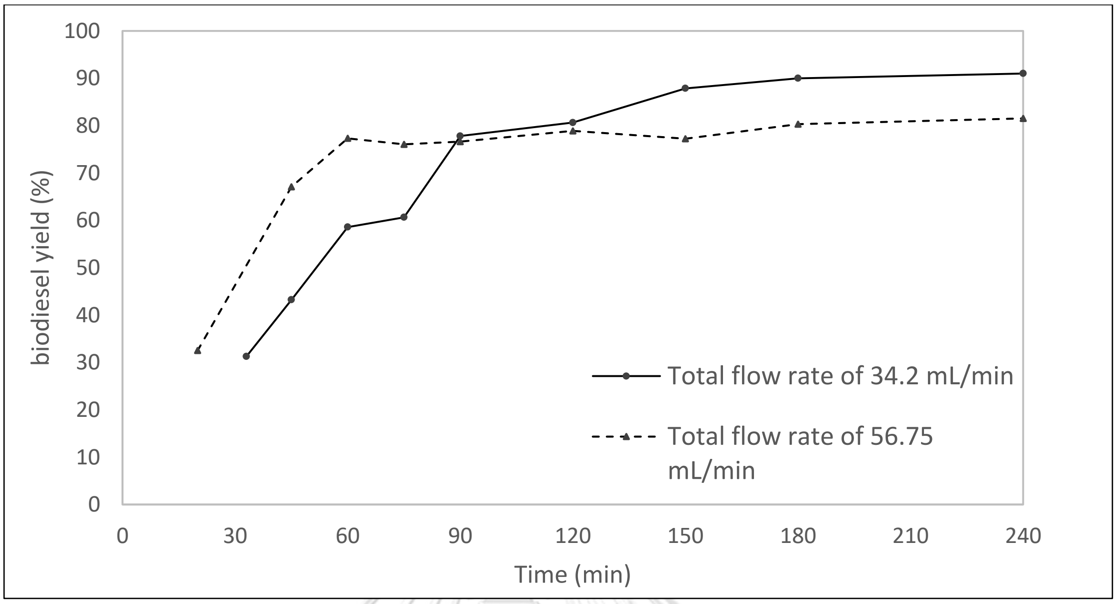


Figure 5.16 Biodiesel yield with difference total flow rate using the combination of microwave and ultrasonic reactor.

Biodiesel yield with difference total flow rate using the combination of microwave and ultrasonic reactor was depicted in Figure 5.16. The reaction was carried out at a reaction temperature of 60°C, methanol to oil molar ratio of 6:1, NaOH loading 1%wt and two total flow rate values 34.2 and 56.75 mL/min with corresponding to residence time of 17.82 and 10.74 min, respective. The results showed that the reaction time needed to reach the steady state of biodiesel yield. The start-up time of 150 min was required for total flow rate of 34.2 mL/min and start-up time of 60 min was required for total flow rate of 56.75 mL/min. The highest biodiesel yield was obtained from total flow rate of 34.2 mL/min. This is due to lower flow rate was help to enhance the contact time of emulsion of reactant resulting to increase biodiesel yield [68]. The yield efficiency at 4 h was depicted in Table 5.1. It can be seen the yield efficiency of total flow rate 34.2 mL/min was 35.09 x 10^{-4} g/J and 47.06 x 10^{-4} g/J for total flow rate of 56.75 mL/min. This results also confirmed that using combination of microwave and ultrasonic can produce biodiesel in a shorter reaction time (less than 20 min). This multifunctional reactor is more potential to apply for transesterification of palm oil in the industrial scale.

Table 5.1 Yield efficiency on microwave and ultrasonic reactor at steady state condition.

Total flow rate (mL/min)	Residence time (min)	Biodiesel yield (%)	Yield efficiency (g/J)
34.2	17.82	91	35.09×10^{-4}
56.75	10.74	81.52	47.06×10^{-4}

Chapter 6

Conclusions and recommendation

6.1 Conclusions

Multifunctional reaction especially, cavitational reactor provides benefit to biodiesel production when increasing the reaction mixture volume for homogenous catalyzed transesterification of palm oil. The initial rate of heterogeneous catalyzed transesterification was decreased with increasing reaction mixture volume in all reactors. Yield efficiency is a recommended indicator for biodiesel production. This parameter can be a guideline to select the optimum condition to produce biodiesel with highest reactor performance. Homogenizer reactor showed the highest biodiesel yield and yield efficiency based on the energy consumption and it can produce more biodiesel (by increasing reaction mixture volume) compared to the other reactors. The combination of microwave and ultrasonic is a potential candidate for application of the transesterification of palm oil in the industrial scale. Operation at low feed flow rate gave the maximum biodiesel yield, but lower yield efficiency when compared to the one at high feed flow rate.

6.2 Recommendation

1. The effects of ultrasonic power and reaction temperature in the continuous process are important parameters influencing the reaction. Therefore, they should be further studied in more details to achieve higher performance.

2. Study effect of operating parameters of combination of microwave and ultrasonic reactor using heterogeneous catalyst for transesterification of palm oil and its reusability.

3. For heterogeneous catalyst, the induction period was also obtained in the cavitational reactor. Therefore, increasing amount of catalyst should be performed to clarify the internal mass transfer causing of induction period.

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APPENDIX

Appendix A

Yield efficiency calculation

The biodiesel yield efficiency is defined in Equation below

$$\text{Biodiesel yield efficiency} = \frac{\text{Amount of product produced (g)}}{\text{Power supplied } \left(\frac{\text{j}}{\text{s}}\right) \text{x reaction time (s)}}$$

For example, calculated yield efficiency of homogenizer at 165 mL and 90% of biodiesel yield.

At first step, we might change g of oil to mol of oil. In this section 1117.48 g of oil was used.

So, mol of oil = $\frac{117.48\ \text{g}}{847\ \text{g/mol}}$ = 0.1387013 mol

Second step, one mol of oil converted to three mol of methyl ester.

So, mol of methyl ester = 0.138701 x 3 = 0.416104 mol

Third step, mol of methyl ester converted to g of methyl ester.

So, g of methyl ester = 0.416104 mol x 286.7 g/mol = 119.297 g of methyl ester

Fourth step, the selection of 90% biodiesel yield was converted to g of actual methyl ester.

So, g of actual methyl ester is equal to $\frac{119.297\text{g x } 90\ \%}{100\%} = 107.3673\ \text{g}$

Five step, selection of reaction time of 90% biodiesel yield from actual graph in Figure A.1.

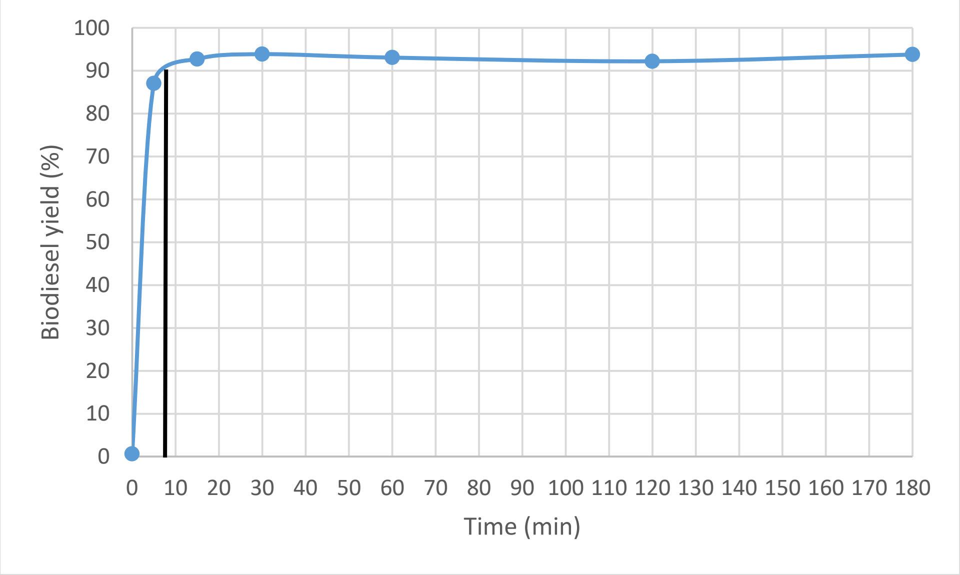

Figure A.1 biodiesel yield on homogenizer at 165 mL.

From the graph selection of reaction time for 90% biodiesel yield was about 8 min.

Thus,

$$\text{Biodiesel yield efficiency} = \frac{107.3673\ (\mathrm{g})}{80\ \left(\frac{\mathrm{j}}{\mathrm{s}}\right) \mathrm{x}\ 480\ (\mathrm{s})} = 0.002796\ \mathrm{g/J}$$

Appendix B

How to reset microwave power measurement

1. Press 1 + 2 for two time
2. Press 3 until it showed EN 1
3. Press 1 until it showed save and press 3
4. Press 3 + 4 for 10 second and it will reset total power to zero and press1

Appendix C

Calculation of velocity at the stator (Homogenizer reactor)

When the velocity of the liquid is throttled through the slots in stator and moving out from the stator is calculated using the flow number N_Q corresponding to the rotor by using information proposed by Sano and Usui as present on equation C.1

$$N_Q = 1.3\left(\frac{D_r}{T}\right)^{-0.86} \left(\frac{W_r}{T}\right)^{0.82} n_r^{0.6} \qquad (C.1)$$

Where

D_r is diameter of rotor

W_r is height of rotor

n_r is the number of peripheral blades in the stator

T is diameter of glass beaker

So,

The flow number of reaction mixture volume of 165 mL was 13.72

The flow number of reaction mixture volume of 500 mL was 13.92

The flow number of reaction mixture volume of 1000 mL was 14.01

And the velocity at the stator is calculated using the flow number as present on equation C.2

$$V_s = \frac{N_Q N_r d_r^3}{n_{ss} H_s W_s} \qquad (C.2)$$

Where

N_r is speed of the rotor

n_{ss} is the number of slots in the stator

H_s is height of the slots in the stator

W_s is width of the slots in the stator

So,

Velocity at the stator of reaction mixture volume of 165 mL was 0.66 m/s

Velocity at the stator of reaction mixture volume of 500 mL was 0.662 m/s

Velocity at the stator of reaction mixture volume of 1000 mL was 0.659 m/s

It can be clearly see, the velocity at the stator was not significantly different when increased reaction mixture volume. Meaning the cavitation was still high even if reaction mixture volume was increased.

Appendix D

Calculation of agitation process proportions (magnetic stirrer reactor)

The designer of an agitation process vessel have been commonly used proportions as followed:

$$\frac{D_a}{D_t} = \frac{1}{3} \qquad \frac{H}{D_t} = 1 \qquad \frac{J}{D_t} = \frac{1}{12} \qquad \frac{E}{D_t} = \frac{1}{3} \qquad \frac{W}{D_a} = \frac{1}{5} \qquad \frac{L}{D_a} = \frac{1}{4}$$

Where

D_a is Total length of impeller

D_t is Width of tank

H is Height of liquid in tank

J is Width of baffle

E is distance between impeller and lower tank

W is Diameter of impeller

L is Length of impeller

The listed standard above are widely accepted and are the basis of many published correlations of agitator performance. Increasing reaction mixture volume in this work is still used the same impeller that leads some proportions so far away from the listed standard resulting to mass transfer limitation.

For example, calculated $\frac{D_a}{D_t}$ at reaction mixture volume of 165 and 1000 mL.

At 165 mL, $\frac{D_a}{D_t}$ was 0.33

At 1000 mL, $\frac{D_a}{D_t}$ was 0.19

From this calculation, it can be seen $\frac{D_a}{D_t}$ was decreased to 1.75 time when increased reaction mixture volume to 1000 mL.

VITA

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