IRJIEST 9 (2023) 06 - 12
Available online at http://ojs.batstate-u.edu.ph
International Research Journal on Innovations in Engineering, Science and Technology

Design and development of a portable biodiesel reactor using aeration-assisted alkali catalyzed transesterification of waste cooking oil

Rejie C. Magnaye*, Neres Ann M. Repollo, Sheena Mae P. Manarin, Daniely Mae Z. Mendoza, Emmanuel D. Regondola

Department of Chemical Engineering, College of Engineering, Batangas State University – The National Engineering University, Alangilan Campus, Alangilan, Batangas City, Batangas 4200

ABSTRACT

The 21st century is an era where the world's transportation is powered by fossil fuels. Due to the said fact, the emission from using these fuels is a huge factor in contributing to the pollution of the world and climate change. This research aimed to produce a greener and more eco-friendly fuel that can be utilized as an alternative for today's fuel. It focused primarily in presenting a design which introduces aeration technology, a heating element and air-atomized spraying of catalyst for the continuous production of biodiesel from waste cooking oil. The transesterification of the biodiesel reactor comprises four stages: pre-heating, synthesis, washing and polishing. An amount of 15.88 L of waste cooking oil (WCO) was reacted with 3.96 L of CH_3OH and 66.67 g of NaOH as catalyst. The CH_3OH and NaOH were mixed in the methoxide tank by a stirrer. The temperature was controlled at 55 ^oC for 1 h of preheating. The aerator and the pump were then simultaneously turned on for the reaction to begin. The reaction lasted for 3 h and 15 min which includes the settling of glycerol. The biodiesel was washed three times using 10.4 L of H_2O per washing. Finally, the biodiesel was heated at 105 ^oC for 3 h for the purpose of polishing. The parameters that affected the production of biodiesel were CH_3OH to biodiesel ratio, catalyst concentration, and reaction time. These parameters were experimentally varied to determine the most economical for the pilot-scale production. The economic CH_3OH to biodiesel ratio was tested to be 6:1, which was the same as the optimum ratio. The lowest catalyst concentration achieved was 0.5 wt% of the straight vegetable oils (SVO). As for the reaction time, the shortest period achieved was 3 h. The biodiesel yield of 97.7 %, was considered a relatively good result at this stage of the technology. The properties of the WCO strongly suggested its efficient conversion into biodiesel. Optimal aeration, characterized by the formation of smaller bubbles that increase surface area, resulted in the highest biodiesel yield. The settling time after the reaction considerably decreased contributing to less energy utilization. On the other hand, the automation of the reactor reduced the need for manual labor which greatly improved the reactor's operation.

Keywords: biodiesel, alkali-catalyzed, aeration, waste cooking oil (WCO), transesterification

1. Introduction

One of the more viable alternatives for fossil fuels and also valuable for its environmental-friendliness is biodiesel. Biodiesel has become a portion of the equation back in the 1990's due to its benefits over petroleum diesel like major drop in greenhouse gas releases, non-sulfur emissions and non-particulate material contaminants, and low toxicity. Additionally, biodiesel returns about 90 % more energy than the energy that is utilized to produce it. Furthermore, European countries account for more than 80 % of global biodiesel consumption and biodiesel markets are undergoing noticeable growth in the United States and Asia, particularly in India and China, where the target of the government is 15 % replacement of diesel by 2020.

To design and develop a portable biodiesel reactor capable of converting waste cooking oil to biodiesel through aeration-assisted alkali-catalyzed transesterification was the focus of the study. Specifically, it aimed to determine the physicochemical properties of WCO in terms of density, viscosity, moisture content, acid value, saponification value, and fatty acid methyl ester (FAME) content; establish design parameters for the development of portable biodiesel reactor with aeration-assisted alkali-catalyzed transesterification; determine the effect of the best aeration flow rate to biodiesel yield and the waste cooking oil properties upon processing; and compare the biodiesel properties of the highest biodiesel yield to existing studies.

The study of [1,2] provided significant contribution as part of the related studies. Accordingly, the transesterification reactor was designed with the consideration of the reactor components and material specifications along with its material and energy balance [1]. The biodiesel properties conformed to the ASTM standards except for H_2O content.

2. Materials and methods

The transesterification of the biodiesel reactor comprises four stages: pre-heating, synthesis, washing and polishing. In the preheating stage, the WCO was preheated to about 60-65 ^oC in the reactor which allowed it to react immediately with the reactant and catalyst. While preheating, the CH_3OH and NaOH were stirred in the methoxide tank for at least 1 h. The catalyst (0.5 % w/w) was first added into the reactant which produced the methoxide, and then mixed with the WCO.

*Corresponding author
Email address: rejie.magnaye@g.batstate-u.edu.ph

Once the NaOH was completely dissolved in the CH_3OH, the methoxide was ready to be added to the oil.

The synthesis stage began when the methoxide was added to the WCO at a flow rate of about 1.5 L/min. According to [3], if WCO is used as the feedstock, a molar ratio of 6:1 was adopted due to the higher percentage of free fatty acids in the oil. The dosage time was monitored to ensure the optimum reaction time for the reaction to take place. It lasted for about 35-45 min for a reaction of 3-4 h. The methoxide was sprayed into the WCO during synthesis through high pressure nozzles that atomized the CH_3OH into small droplets. As a result, the contact surface area of the CH_3OH/WCO considerably increased. The aeration system, the sparger, was turned on in this stage to assist in speeding up the reaction by producing a large interfacial area through the dispersion of gas bubbles into the liquid thus creating more reaction sites. Because of this, there was an increase in the speed of reaction between the WCO and methoxide. Aeration did not only help to create reaction sites, but it also aided in the efficient mixing of the two immiscible liquids.

Point reaction and selective aeration are the key points for faster settling time. Using point reaction method, a procedure where reaction product is formed instantaneously when the methoxide comes into contact with the WCO via aeration system, bubbles containing glycerol fall down to the bottom of the reaction once methoxide comes into contact with the WCO. Moreover, with the special design of the aeration system, the position of the sparger allowed the reaction to proceed without interfering with the settling.

After completing the synthesis stage, all the glycerol were allowed to settle with the aeration system turned off. Once all the glycerol was settled, it was drained out from the reactor. Due to the low solubility of glycerol in the esters, this separation generally occurs quickly and can be completed in 15 min. H_2O was added to the reaction mixture after the glycerol was drained completely.

In the washing stage, H_2O was pumped into the crude biodiesel in order to wash out all the unreacted methoxide. The recovery of the catalyst happens on this stage. When the aerator is switched on, the air bubbles moved upwards through the biodiesel, carrying a film of H_2O which washed the biodiesel as it passed through. At the surface, the bubbles burst, leaving a small drop of H_2O which sank down through the biodiesel carrying the contaminants. Each washing process was carried out for 30 min and was repeated for three times. Similarly, 15 min was used for the settling of H_2O and then drained out.

Lastly, the polishing stage allowed the biodiesel to be heated up at high temperature in order to vaporize any remaining impurities (mainly H_2O) so that the final product is a high-purity biodiesel that can meet the PNS ASTM standard.

3. Results and discussion

3.1. Determination of physicochemical properties of WCO

The WCO collected from a fast-food restaurant in Brgy. Sambat, Tanauan City, Batangas had undergone a trial for the determination of moisture content, kinematic viscosity, density, acid value, pH value, saponification value and FAME content. Figure 1 shows the WCO used by the researchers and tested in the laboratory of the Department of Science and Technology and SGS.

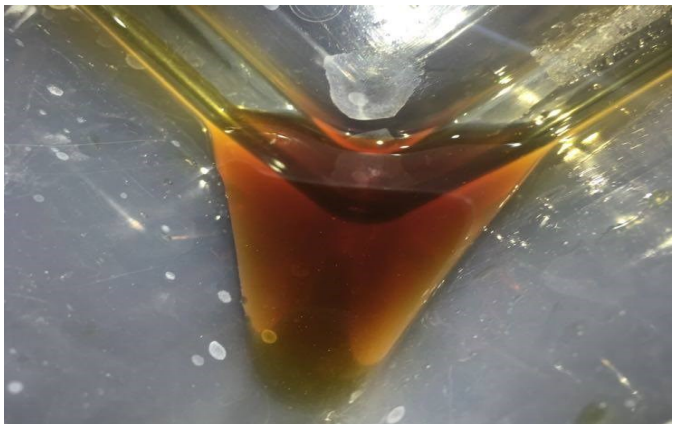


Figure 1. WCO sample.

As presented on Table 1, there was no moisture content found on the WCO. It was a good indication for the success of the reaction since it prevented the hydrolysis of the triglycerides during the reaction. NaOH in H_2O can also cause saponification, thus retarding the reaction.

Table 1. Characteristics of WCO.

Properties	Test method	Results	Reference values
Moisture content	ASTM D95	0	0.12 ± 0.00%
Kinematic viscosity at 40 °C	ASTM D445	42.4 mm^2/s	51.04±0.03 mm^2/s
Density at 25 °C	ASTM D1298	916.79 kg/m^3	912.24± 4 kg/m^3
Acid value	ASTM D664	2.1 mg KOH/g	2.1 mg KOH/g
Saponification value	AOCS Cd 3-25	225.8 mg KOH/g	186.3mg KOH/g
Fatty acid methyl ester (FAME)	PNS EN 14103 modified	62.9% (V/V)	80% (V/V) max

Source: Department of Science and Technology Standard & Testing Division and SGS Testing Laboratory

According to [4], kinematic viscosity of WCO at 40 °C is 51.04 mm^2/s which showed a higher value than the obtained result which is 42.4 mm^2/s. Based on the study of [5], decreasing the kinematic viscosity of WCO is beneficial for the biodiesel to be used on engines. Density is a function of temperature and decreases as temperature increases. The obtained value of density at 25 °C, 916.79 kg/m^3, was slightly less than those recorded by [6,7] as 0.9216 and 0.9185 g/cm^3 at 23 °C, respectively. Taking into consideration the obtained acid value of 2.1 mg KOH/g, it was exactly the same as the one recorded by [6]. It corresponds to the weight percent of free fatty acid (%FFA) which is lower than 2 %, thus the methods used for biodiesel production from WCO are very similar to the conventional transesterification process with an alkali catalyst. The saponification value, as presented on Table 1, obtained was slightly higher than those ones recorded by [8,4] as 183.1 mg KOH/g and 204.77 mg KOH/g, respectively. FAME are the

components of biodiesel. Having a high percentage of it on the WCO sample is beneficial for the overall reaction for converting it to biodiesel. The obtained value of FAME content was 62.9 % serving as a good indicator for the reaction to proceed.

3.2. Design parameters of a portable biodiesel reactor

3.2.1. Manual operation design

Figure 2 shows the design that indicates the system components of the developed manual portable biodiesel reactor. Tank and pipe sizes, nozzle and aerator design, compressor, heater, mixer and pump, as well as the parameters used were already provided in the manual biodiesel reactor.

Figure 2. Actual design of the manual portable biodiesel reactor.

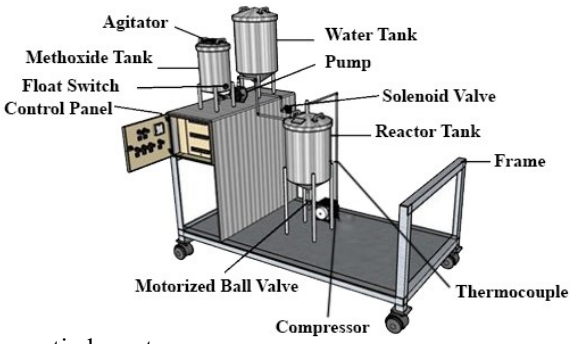
Conventional transesterification processes for producing biodiesel involve the use of agitators or stirrers for the mixing of the catalyst and oil. However, the prototype allowed the use of an aerator for the decrease in reaction time of the process thus decreasing the cost of production.

The biodiesel reactor mainly consisted of three major components: main reactor tank, methoxide tank, and aerator. The main reactor tank, a cylindrical tank with a conical bottom, has a height of 0.4 m and a diameter of 0.3 m. It has a maximum capacity of 20 L with a feed capacity of 15.88 L of waste cooking oil and 3.96 L of catalyst. A heater was installed in the reactor for heating the contents of the tank during the reaction. Three atomizing nozzles were placed at the top of the reactor for the dispersion of the catalyst in fine mist form. A sight glass was also installed at the top of the reactor for monitoring during the reaction. The second tank for the methoxide, with a height of 0.3 m and a diameter of 0.2 m, has a maximum capacity of 10 L. It has a static mixer installed at the top for the mixing of the CH_3OH and NaOH catalyst. A pump was connected with suction tubing made from clear PVC and a discharge tubing made from natural polyethylene (LLDPE). An aerator was installed as a part of the reactor. It is a circular pipe that disperses bubbles in the liquid using air coming from the compressor. Ball valves were used for the tubing and pipe and drain valves were used at the bottom of each tank.

3.2.2. Automated operation design

The design of the automated portable biodiesel reactor was illustrated in Figure 3. The modified portable biodiesel reactor consisted of instruments that were needed to automate the operation. Additional features were the control panel, the solenoid valve, the motorized ball valve, the float switch, the thermocouple for the Arduino-based temperature controller, and the elevated H_2O tank.

a)



a) Schematic layout.

b) Actual design.

Figure 3. Automated portable biodiesel reactor.

3.3. Material of construction, specifications, and working conditions

The materials used in the system were constructed based on its durability, mobility and resistance to corrosion. Because the system used highly corrosive chemicals, durable and corrosion resistant materials such as stainless steel, PVC and LLDPE tubing were used. The system also utilized heat during the process so heat resistant materials were also considered. Table 2 shows the material requirements and specifications of every system component.

Additional materials were used to achieve the upgraded design for the automatic portable biodiesel reactor. The materials used in the system were constructed based on its durability, mobility and resistance to corrosion. The system also utilizes heat during the process so heat resistant materials were also considered.

Table 2. Design specifications.

Parts	Dimension	Materials of construction
Main reactor tank	Height: 0.4 m Diameter: 0.3 m Capacity: 28.274 L Safety factor: 20 %	Stainless steel
Methoxide tank	Height: 0.3 m Diameter: 0.2 m Capacity: 9.425 L	Stainless steel
Aerator	Outer diameter: 0.28 m Middle diameter: 0.26 m Inner diameter: 0.24 m	Aluminum
Pump	4-100 %stroke length 220 V / 50Hz	PVDF
Heater	Length: 11 in bended 2000 W / 220 V	Stainless steel
Stirrer	Length: 0.28 m	Stainless steel
Nozzle	Inner diameter: ¼ in	Stainless steel
Compressor	220 V / 50 Hz 58 W Output: 75 L/ min	Stainless steel
H_2O tank	Height: 0.7 m Diameter: 0.3 m Capacity: 49.480 L	Stainless steel
Suction tubing	Inner diameter: ½ in	Clear polyvinyl chloride
Discharge tubing	Inner diameter: ½ in	LLDPE
Sight glass	Length: 0.25 m	Acrylic

3.4. Determination of the effect of the best aeration to biodiesel yield

Preliminary testing was conducted at Batangas State University PB Main Campus II in April 2019. Five trials were performed to assure the consistency of the prototype. The third to fifth trials were conducted at Automatic Motors. The reactor produced a high yield of biodiesel which averaged 92.37 % obtained from the trials in Table 3.

Table 3. Aeration rate during the trials.

Trial	Aeration rate	Biodiesel %yield
1	Standard	90.05
2	Standard	92.38
3	Standard	91.56
4	Adjusted	93.51
5	Adjusted	94.33

It is observed that the yield of the biodiesel from the first three trials was less than the fourth and fifth trial. During the first three trials, the aerator produced slightly larger bubbles than those produced during the last two trials. It can be said that the smaller the bubbles produced, the higher the biodiesel yield. The smaller bubbles produced more surface area covered for the fine sprayed methoxide thus improving the rate of reaction.

Figure 4 shows the bubbles produced during the trials. The left photo was the aerator setup used during the first three trials while the right photo was the aerator setup used during the last two trials. It is evident that the right photo shows the production of uniform smaller bubbles than the left photo.

Figure 4. Aeration setup used during the trials.

The standard flowrate of air coming from the compressor was 75 L/min. The flow rate was adjusted in order to determine the optimum aeration rate for the reaction. For the fourth and fifth trials, the flow of air was lessened to about half of the original flow rate which is 37.5 L/min. It produced higher yields compared to the first three trials (Table 3). Hence, decreasing the flow rate increased the efficiency of the aeration and further produced higher yields of biodiesel.

3.5. Determination of the characteristics of biodiesel

The preliminary analysis of biodiesel produced was based on its physical appearance as compared to the existing biodiesel B100. Out of the five samples, the fourth and fifth samples were the only ones close to the appearance of the existing biodiesel B100. The series of testing conducted by the proponents of the laboratory were flammability test, biodiesel conversion test or 3/27 test, and determination of density. All the samples passed the flammability test indicating the presence of biodiesel.

The 3/27 test is a qualitative test that can be done in a laboratory. This is done to reflect how effective the process is to convert the triglyceride to biodiesel successfully. This is usually the first testing for biodiesel, as it can determine the success the biodiesel has to be commercialized as pure and high-grade biodiesel. Therefore, passing the 3/27 test would give an estimation on the effectiveness of the conversion. If the test passes, the biodiesel sample would be sent for further testing through ASTM and EN standards for commercializing potential. The test was done in a vial of at least 30-mL large. Twenty-seven mL of CH_3OH was placed in the vial, followed by 3 ml of the biodiesel product dropwise. It was ensured that the two substances were at room temperature first before adding them to the test tube with separate pipettes. The test tube was then closed and gently shaken, before it was left to

rest for at least 5-10 min. The test tube is then tilted at about a 45° for around 10-15 min, any liquid that settles to the bottom by the end of 15 min is a fail. It passes when the biodiesel completely dissolves in the CH_3OH.

Figure 5 shows the actual photos of the 3/27 test conducted by the researchers. By visual evidence, the initial trial runs failed the test while the actual runs passed the test. In the first three trials, yellow globules can still be seen at the bottom of the test tubes and compared to the last two, the solution is cloudier. The result from the actual runs presents a clear and colorless solution denoting full conversion of the oil to biodiesel. Figure 6 shows the product results from the runs. Also, by visual evidence, the biodiesel produced from the runs was close to the color of the standard biodiesel.

Figure 5. The 3/27 results from the trials.

Figure 6. Product results from the trials.

The properties of the actual run samples tested on the Department of Energy were shown on Table 4 while the comparison to other existing studies were shown in Table 5.

Table 4. Comparison of the biodiesel characteristics with the reference values.

Properties	Test Method	Sample result 1	Sample result 2	Reference values
Density at 25°C (kg/m^3)	PNS ASTM D1298	871	870	860-880
Kinematic viscosity at 40 °C (mm^2/s)	ASTM D445	10.6	10.6	1.9-6.0
Flash point by PM °C)	ASTM D93	127	129	93.0 min
H_2O (% vol)	ASTM E203	0.15	0.16	0.05 max
Total glycerine (% mass)	EN14105 modified	0.21	0.18	0.24
Free glycerine (% mass)	EN 14105 modified	0.17	0.14	0.02
Sulfur (ppm)	ASTM D5453	<3	<3	15 max

Source: Department of Energy, Biomass Division

Table 5. Comparison of biodiesel characteristics with related studies.

Properties	[1]	[9]	[10]
Density at 25 °C (kg/m^3)	ND	883	870
Kinematic viscosity at 40 °C (mm^2/s)	3.00	4.3	3.5
Flash point by PM (°C)	103	174	150
H_2O (% vol)	0.14	0.0564	0.16
Total glycerine (% mass)	0.05	0.005	0.24
Free glycerine (% mass)	ND	0.121	0.20
Sulfur (ppm)	5	18.4	0.05

Density is the weight per unit volume. Oils that are denser contain more energy. The study of [9,10] indicated the similarity of the properties to existing biodiesel.

Viscosity is an essential property of a fuel because it indicates the ability of a material to flow. Thus, it affects the operation of the fuel injection equipment and spray atomization. The obtained value of the kinematic viscosity was relatively higher than those from the existing studies. It poses a challenge for the utilization of the fuel but having the biodiesel blended with other fuels can easily lower its viscosity thus preventing problems in fuel injection.

Flash point of a fuel is the temperature at which it will ignite when exposed to a flame or a spark. The flash point of the biodiesel is higher than that of diesel fuel, which is safe for transport, handling and storage purposes. The values

obtained were close to the related studies which may indicate that the biodiesel is safe for storage, transport and handling.

H_2O can be present in the fuel in two forms, either dissolved or suspended. While biodiesel is generally considered to be insoluble in H_2O, it actually takes up considerably more H_2O than diesel fuel. The H_2O content from the results were almost the same with [1,10] studies. The study of [9] have the lowest value among the studies. The H_2O content can easily be reduced upon increasing the duration of the purifying stage of the process in order to evaporate the excess moisture.

Total glycerin is a measurement of how much triglyceride remains unconverted into methyl esters. The total glycerol can be less than 0.25 % of the final biodiesel product. The values followed the existing standard similar to the related [10], however the results from [1,9] were relatively lower.

Free glycerol refers to the amount of glycerol that is left in the finished biodiesel. The content of free glycerol in biodiesel is dependent on the production process. The high yield of glycerol in biodiesel may be because of the insufficient separation during washing of the product. Glycerol is essentially insoluble in biodiesel so almost all of the glycerol is easily removed by settling. Free glycerol may remain either as suspended droplets or as the very small amount that is dissolved in the biodiesel. It can be less than 0.24 % of the final biodiesel product. The result conformed to the standard values, similar with the related studies.

Lastly, sulfur content was also analyzed for the samples. The most probable reason for the observed values of sulfur content is the various foods that cooking oil comes in contact with. High sulfur levels in the fuel might result in the production of H_2SO4 and sulfate compounds in the engine. H_2SO4 causes corrosion in the engine, while sulfates lead to increased particulate matter emissions. The values obtained were both <3 ppm indicating less sulfur present in the product. It was also lower than [1,9] studies but was higher than [10].

4. Conclusions

The characteristics of the waste cooking oil were a good indication that it can be efficiently converted to biodiesel. The biodiesel reactor was designed with the careful consideration of the reactor components and its material specification had conformed to the proposed design. Producing smaller bubbles to create more surface area signifies the best aeration flow rate and achieved a higher yield of the biodiesel. The settling time after the reaction considerably decreased contributing to less energy utilization and the automation of the reactor reduced the need for manual labor and greatly improved the reactor's operation. The automated portable biodiesel reactor can produce a higher yield of the biodiesel conforming to the ASTM standards as compared to the manual portable biodiesel reactor.

It is further recommended that a condenser be added to the reactor for the recovery of the CH_3OH during the reaction. Automation of the developed prototype is highly advisable to decrease the manual labor. Using powdered form of NaOH is suggested to improve the mixing of the catalyst. For the methoxide tank to become more efficient, the stirrer must be improved, and a heater can be added. It is also appropriate to source out for other available nozzles for the methoxide spray.

References

[1] Cinco WM, Evangelista KJ, Matibag MC. Design and development of a continuous flow biodiesel reactor - separator [Undergraduate thesis]. Batangas City: Batangas State University; 2015.

[2] Xuan TW, Fahmi MI, Jing ZH. Investigation into the optimization of biodiesel production process [Undergraduate thesis]. Singapore: Singapore Polytechnic; 2017.

[3] Teng G, Gao L, Xiao G, Liu H. Transesterification of soybean oil to biodiesel over heterogeneous solid base catalyst. Energy & Fuels [Internet]. 2009 Sep 17;23(9):4630-4. Available from: https://doi.org/10.1021/ef9003736

[4] Chuah LF, Aziz AR, Yusup S, Bokhari A, Klemeš JJ, Abdullah MZ. Performance and emission of diesel engine fuelled by waste cooking oil methyl ester derived from palm olein using hydrodynamic cavitation. Clean Technol Environ Policy [Internet]. 2015 Apr 9;17(8):2229-41. Available from: https://doi.org/10.1007/s10098-015-0957-2

[5] Kim TH, Han JK. Viscosity characteristics of waste cooking oil with ultrasonic energy irradiation. J Biosyst Eng [Internet]. 2012 Dec 31;37(6):429-33. Available from: https://doi.org/10.5307/jbe.2012.37.6.429

[6] Chhetri A, Watts K, Islam M. Waste cooking oil as an alternate feedstock for biodiesel production. Energies [Internet]. 2008 Apr 10;1(1):3-18. Available from: https://doi.org/10.3390/en1010003

[7] Mahgoub HA, Salih NA, Mohammed AA. Suitable condition of biodiesel production from waste cooking oil–al-baha city – KSA. Int J Multidiscip Curr Res. 2015 May 26;3:447-51.

[8] Yusuff AS, Gbadamosi AO, Popoola LT. Biodiesel production from transesterified waste cooking oil by zinc-modified anthill catalyst: parametric optimization and biodiesel properties improvement. J Environ Chem Eng [Internet]. 2021 Apr;9(2):104955. Available from: https://doi.org/10.1016/j.jece.2020.104955

[9] Tsoutsos T, Tournaki S, Gkouskos Z, Paraíba O, Giglio F, García PQ, Braga J, Adrianos H, Filice M. Quality characteristics of biodiesel produced from used cooking oil in southern europe. ChemEngineering [Internet]. 2019 Feb 16;3(1):19. Available from: https://doi.org/10.3390/chemengineering3010019

[10] Atabani AE, Silitonga AS, Badruddin IA, Mahlia TM, Masjuki HH, Mekhilef S. A comprehensive review on biodiesel as an alternative energy resource and its characteristics. Renew Sustain Energy Rev [Internet]. 2012 May;16(4):2070-93. Available from: https://doi.org/10.1016/j.rser.2012.01.003

Acknowledgment

The researchers would like to express their gratitude and appreciation to Batangas State University for allowing them to maximize its full potential as an experimental laboratory workplace and funding the research; To Singapore Polytechnic for sharing this idea a breakthrough last 2018 and to all the people who interceded for the success of this research.