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Guided-Inquiry of Green Chemistry-Based Experiments in Biodiesel Synthesis

Risnita Vicky Listyarini^{a*}, Fransisca Ditawati Nur Pamenang^b, Noer Kurnia Dewi^c

^a Chemistry Education Study Program, Faculty of Teacher Training and Education, Sanata Dharma University, Yogyakarta, Indonesia ^b Chemistry Education Study Program, Faculty of Teacher Training and Education, Sanata Dharma University, Yogyakarta, Indonesia ^c Chemistry Education Study Program, Faculty of Teacher Training and Education, Sanata Dharma University, Yogyakarta, Indonesia

*Corresponding author: Campus III, Paingan, Maguwoharjo, Depok, Sleman, Daerah Istimewa Yogyakarta, 55282, Indonesia. E-mail addresses: risnita.vicky@usd.ac.id

article info	abstract
Article history:	The use of non-renewable energy sources such as fossil fuels is very contrary to the concept
Received: 29 March 2020	of sustainable development. The principles of green chemistry can be applied to laboratory
Received in revised form: 27 May	work through experiments based on environmental issues such as energy. Biodiesel is an
2020	alternative fuel to replace fossil fuels which can be made through the process of
Accepted: 31 May 2020	transesterification of oils. This research aims to apply the principles of green chemistry in
Available online: 30 June 2020	biodiesel synthesis experiments; designing and conducting thoughtful and efficient experiments; characterizing biodiesel products and knowing students' responses to green
Keywords:	chemistry-based experiments in biodiesel synthesis. The results showed that the principles of
Biodiesel synthesis	green chemistry were successfully applied in the synthesis of biodiesel. Biodiesel products
Green chemistry experiments	have a bright yellow colour, pH of 7, and density between 0.866-0.892 g/mL. Water contents
Guided-inquiry	range from 1.67-3.3, and acid numbers range from 0.69-4.3. 90/10 tests show the results
Laboratory work	between range 0.5-3% and kinematic viscosity from 4.195-4.995. Students showed a positive response of 75.36% in designing experimental procedures for green chemistry-based
	biodiesel synthesis.

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1. Introduction

Energy demand in the world is very dependent on the use of fossil fuels which are non-renewable energy sources. Oil consumption is projected to continue to rise until 2040 (Outlook, 2017). The use of excessive non-renewable energy sources is very contrary to the concept of sustainable development. Sustainable development is a development that can meet human needs in the present without ignoring future generations in fulfilling their needs (Eilks & Rauch, 2012). Non-renewable energy sources can be depleted if the energy consumption continues to increase, and energy management systems are less precise to anticipate the shortage of energy sources and increasing their roles in addressing the world energy needs.

The role of chemistry education is very important in sustainability for the community now and in the future. The contribution of the chemical sector in sustainable development can be made by promoting the principles of green chemistry. There are twelve principles in Green Chemistry that aim to make chemistry more efficient in energy use, to reduce generated waste, and produce innovative products made of less natural resources (Anastas & Eghbali, 2010; Anastas & Warner, 1998). The twelve principles of Green Chemistry are (1) Prevention (2) Atom Economy (3) Less hazardous chemical synthesis (4) Designing safer chemicals (5) Safer solvents and auxiliaries (6) Design for energy efficiency (7) Use of renewable feedstock (8) Reduce derivatives (9) Catalysis (10) Design for degradation (11) Real-time analysis for pollution prevention (12) Inherently safer chemistry for accident prevention (Anastas & Warner, 1998). Green chemistry is widely known since the 1990s and has been incorporated in higher education through laboratory experiment, teaching material, and courses (Gross, 2012; Hamidah et al., 2017; Kolopajlo, 2017; Listyarini, 2019; Listyarini et al., 2019)

Education in the field of chemistry is challenged to prepare young people who are competent and active to contribute to society. Students are equipped with competencies and knowledge that enable them to solve problems related to social, environmental, and sustainable development issues (Sjöström et al., 2015). Students, as part of future generations, are expected to be able to learn sustainable and environmentally friendly chemistry. Learning green chemistry more effectively and genuinely can be applied in laboratories through experiments that contain crucial environmental issues. The experiment is designed to respond to existing environmental issues in the world (Andraos & Dicks, 2012). In designing and carrying out experiments by using a guided-inquiry experiment, students are encouraged to apply the principles of green chemistry that have been learned. Inquiry experiment gives students opportunities to actively engage in learning and to experience student-centred learning (Lee, 2019; Ratnasari et al., 2015). It is expected that students can analyze the environmental effects of organic chemical synthesis and the role of sustainability and green chemistry in higher education.

One issue that can be associated with green chemistry-based learning is the issue of energy. As the supply of fossil fuels keeps decreasing, the use of renewable fuels must be increased to respond to the world's needs. Biodiesel is a renewable energy source that is environmentally friendly and can be used on diesel engines with some minor modifications. Biodiesel is promoted as an energy source that can replace fossil fuels. Biodiesel is produced from vegetable oils such as palm oil, rapeseed, soybean and sunflower seed oil and animal fats using the transesterification process (Mekhilef et al., 2011). Biodiesel from palm oil has great potential as a source of energy in the future. Indonesia, one of the world's palm oil-

producing countries with production in 2017/2018, ranges from 38.5 million tons. This makes Indonesia a source of raw materials for biodiesel production.

Several studies regarding biodiesel synthesis for undergraduate courses have been published in recent years (Akers et al., 2006; Behnia et al., 2011). Biodiesel synthesis in undergraduate courses discusses biodiesel synthesis made of different seed (Goldstein, 2014) and problem-based green chemistry biodiesel synthesis experiment (Gross et al., 2016). However, to the best of authors' knowledge, there is no guided-inquiry of a green chemistrybased experiment in biodiesel synthesis. This research was intended to apply the principles of green chemistry in biodiesel synthesis experiments. This research instructed students to design and conduct thoughtful and efficient experiments and characterize biodiesel products. Then, the students' responses to designing green chemistry-based experiments in biodiesel synthesis were collected.

2. Method

Experimental design

Green Chemistry-based experiment on biodiesel synthesis was designed for second-year students of the Chemical Education Study Program, Sanata Dharma University. This laboratory experiment was an integrated practicum in the Organic Chemistry Laboratory work course.

Before starting the experiment, students were given an introductory explanation to biodiesel, and the use of biodiesel as an alternative fuel that could be renewed and its impact on the environment. Students were instructed to apply the principles of green chemistry in biodiesel synthesis. Students were directed to read the references and investigate the factors that influence biodiesel synthesis.

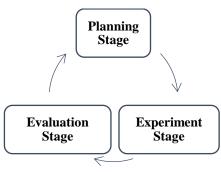


Figure 1. Experimental design

Planning Stage

At this stage, students were actively involved in studying the literature and designing experiments on biodiesel synthesis methods and the factors that influenced the success of biodiesel synthesis. Each group prepared an experimental design. The observations were conducted to check students' readiness to design a green chemistry-based experiment on biodiesel synthesis. Several questions were provided for guiding the students as follows.

- 1) What can green chemistry principles be applied in the biodiesel synthesis experiment?
- 2) What factors influence the success of biodiesel synthesis?
- 3) Search for journals or literature that supports and designs a thoughtful and efficient method for synthesizing biodiesel!

Biodiesel synthesis could be done by a variety of methods (Aziz et al., 2011) and the ratio of materials used (Astuti, 2008). In the next step, the students were asked to fill this table adapted from Gross et al. (2016). The students were asked to submit their experimental design using the guided-questions in three days prior to the laboratory work. This time limit was set to allow students to receive feedback or suggestion from the lecturers.

Table 1. Synthesis of Biodiesel from vegetable oils

Oil type	Oil type Oil volume (mL)		Catalyst mass per litre of oil (g)

Experiment Stage

At this stage, students conducted biodiesel synthesis based on the experimental designs that had been prepared by characterizing the synthesis of biodiesel and explaining the synthesis results. Observations were made on the performance of students was observed when they were practising in the learning process and explaining their synthesis products

Evaluation Stage

At this stage, each student conducted a self-evaluation using a questionnaire for cooperative skills, scientific skills, and scientific attitudes. Furthermore, these data were analyzed by descriptive analysis.

Biodiesel synthesis

Biodiesel synthesis method was designed by students who then discussed and reviewed it together by using the method in Aziz et al. (2011) as reference. The students used different variables for biodiesel synthesis. The synthesis began by measuring the volume of oil and methanol used. Methanol was added to vegetable oils into beakers. The catalyst used was weighed and added into a beaker, then it was heated at 50 °C for 30 minutes. The mixture was

allowed to settle at the room temperature and transferred to a separating funnel. After that, the mixture was separated into two layers—the top layer was biodiesel, and the bottom layer was glycerol. The lower layer was removed from the separating funnel so that there was only biodiesel in the separating funnel. Biodiesel was washed with distilled water in a separating funnel then the water layer was removed, and biodiesel was stored. Biodiesel products were ready to be characterized.

Biodiesel characterization

Colour test

The colour of biodiesel was tested through visual observation

pH test

The biodiesel pH produced was tested using a universal indicator. The standard pH of biodiesel ranges from pH 7.

Density test

Pycnometers were used for density testing. Initially, the weight of the empty pycnometer was weighed. Then, pycnometer weight and water were weighed to calculate the pycnometer volume. Afterwards, weighing the pycnometer and biodiesel was to check the volume of biodiesel. Finally, biodiesel density could be calculated.

Water content test

Biodiesel was weighed and put into a petri dish (A). Afterwards, the biodiesel was heated in an oven at 105 °C for 2.5 hours. In the next step, biodiesel was taken out from the oven after 2.5 hours. Biodiesel (B) was weighed, and the reduction in oil weight was considered as the weight of water that evaporated from the oil.

Acid number test

Biodiesel was weighed as much as 2 grams in a 50-ml Erlenmeyer. The mixture was added with 10 ml of isopropyl alcohol. The mixture was titrated with 0.1 N KOH solution by using phenolphthalein as an indicator.

Free fatty acid number (FFA) =
$$\frac{Volume of KOH x Mw KOH x N KOH}{biodiesel mass}$$
.....(1)

90/10 Test

90/10 test was conducted based on the method invented by Gross et al. (2016) to detect the amount of unreacted vegetable oil. 1 mL of the biodiesel product was added to the conical tube centrifuge and followed by 9 mL of up to 10 mL of methanol. The tube was shaken vigorously, and the layers were allowed to settle. The oil that did not rotate would go down to

the bottom. The volume of unreacted oil was multiplied by 10 for the approximate percentage (%) of reaction imperfections.

Viscosity test

The biodiesel viscosity test was conducted at the Petroleum Gas and Coal Technology Laboratory, Department of Chemical Engineering, Faculty of Engineering, Gadjah Mada University using ASTM D 445 testing method.

Data Analysis

The data obtained from this study were data of experimental results as well as students' evaluation data. From the characterization of biodiesel were obtained data on colour, pH, density, viscosity, water content, the acid content of each biodiesel product. The questionnaire was used to determine the student's response to designing green chemistry-based experiments in biodiesel synthesis.

3. Result and Discussion

The chemistry laboratory work experiment is traditionally conducted using the "cooking book" method. This experiment provided a series of procedures in which students learned the practical skill and proved the theory they had learned previously in theory class (Laredo, 2013). However, this chemistry guidebook experiment limited the freedom to plan and design the experiment (Pamenang et al., 2020). In this study, the green chemistry-based experiment of biodiesel synthesis was conducted using guided-inquiry experiments. According to Lee (2019), guided-inquiry questions was suitable for an organic chemistry course. Students got an opportunity to develop their higher-order thinking skills and activities to engage in a learning activity (Mistry et al., 2016). Therefore, in this study, several questions were provided as guidelines for students to design green chemistry-based experiment of biodiesel synthesis. In the process, students were required to be actively involved in conducting literature studies and designing experiments. Several studies had stated that inquiry-based experiments in organic chemistry enhance students' conceptual understanding and participation in class (Mistry et al., 2016; Okoth et al., 2019; Supasorn, 2012).

The implemented stages of the experimental design were literature studies conducted by students to enable students to get an overview of biodiesel synthesis and to determine the tools, materials, and methods used. Thus, the experiments designed can run effectively and efficiently. The experimental understanding was enhanced because of guided-inquiry

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challenges the students in all steps of experimental procedures, including searching the literature, planning, and experimenting (Green et al., 2004; Supasorn, 2012). The students were asked to submit their laboratory in three days before laboratory hours for suggestions given by the lecturer. This step was necessary because students could get confirmation and suggestions from the lecturer (Supasorn, 2012).

In this biodiesel synthesis process, the green chemistry principles were implemented. The principles of designing safe synthesis processes (3) could be seen as the safety procedures of synthesis. These principles were applied to ensure that students did not use any concentrated or dangerous material and excessive heating and to check whether the students used safety equipment to minimize the potential major hazards. The principle of designing safe chemical products (4) could be seen from the nature of biodiesel products which were more environmentally friendly than fossil fuel. Chemical products should be synthesized by minimizing their toxicity (Saleh & Koller, 2018). The principle of using renewable feedstock (7) was seen from the main ingredient of biodiesel derived from oils of plants. According to the principles of green chemistry, renewable resources like plant biomass or microbial was promising for the development of next-generation fuels (Saleh & Koller., 2018). Principle using catalysts (9) were seen from the basic catalysts and acids used to maximize the synthesis process. In terms of the catalysis, it was necessary to discuss biocatalysts such enzymes because of their highly selective characteristic. The principle of designing chemicals that were easily degraded (10) could be seen from the nature of biodiesel which could be degraded in combustion reaction into carbon dioxide and water which were the end product (Saleh & Koller, 2018).

Biodiesel synthesis

Students were instructed to fill in a worksheet containing methods, amount of vegetable oil, amount of alcohol, and amount of catalyst. The worksheets were used and collected through the LMS (Learning Management System) owned by Sanata Dharma University. Examples of filled worksheet tables were presented in Table 2. The students had filled with information obtained from literature studies.

Table 2. Example worksheet composition of biodiesel synthesis from vegetable oils

Oil Type	Oil Volume (mL)	Catalyst type	Catalyst mass per litre of oil (g) (g/L)
Palm oil	100 mL	NaOH	1 g/L

Students also determined the variables that affected biodiesel synthesis and choose one variable used in biodiesel synthesis. Table 3 contains the variables chosen by students in biodiesel synthesis.

Group	Variable
1	Oil Type: Palm oil vs Corn oil
2	Type of catalyst: NaOH vs KOH
3	Amount of NaOH catalyst (corn oil)
4	Reaction temperature: heating vs room temperature

Table 3. Variables were chosen by students in biodiesel synthesis

The synthesis of biodiesel from vegetable oils applied several green chemical principles. Based on the table above, the types of oil used were palm oil and corn oil with alkaline catalysts in the form of NaOH and KOH as well as variations in the amount of NaOH catalyst and reaction temperature. Biodiesel synthesis from vegetable oils used a transesterification reaction. The transesterification reaction was a new ester reaction that had an exchange of fatty acid positions. Triglycerides from vegetable oils could be converted into alkyl esters (biodiesel) and glycerol assisted by alcohol (Pawoko, 2009). In this synthesis, the type of alcohol used was methanol. Methanol reacted with a basic catalyst to produce a methoxide solution. When the methoxide solution reacted with oils containing triglycerides, a transesterification reaction occurred. The methoxide ion was a strong nucleophile so that it could replace the carbonyl group in fatty acids (Sudrajat et al., 2010). After the students had synthesized biodiesel, the characterization of biodiesel including colour, pH, density, acid number, water content, 90/10 test, and kinematic viscosity was conducted.

Colour test

Biodiesel from synthesis had a clear yellow colour. All biodiesel produced by synthesis had a colour similarity, as shown in Figure 2. Biodiesel is yellow, which often changes into brown because of storage time (Ferrari et al., 2011).

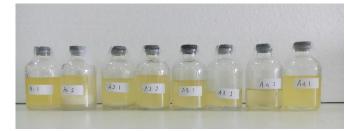


Figure 2. Biodiesel from vegetable oil

pH test

The biodiesel produced was also tested for pH using a universal indicator. Based on the study of (Freedman, 1986), the standard pH of biodiesel must be neutral or 7. If the synthesized biodiesel product still had a basic pH, it showed that the biodiesel still contains the remaining alkaline catalyst, methanol, or other impurities. So, the pH of biodiesel becomes neutral, or 7 was washed using distilled water. Washing the remaining impurities with distilled water was intended to remove soap, alkaline catalyst, methanol, or other impurities (Atadashi et al., 2011; Muniyappa, 1996). In these experiments, students had washed by spraying distilled water on biodiesel in a separating funnel several times. The use of distilled water was chosen because distilled water is a safe and effective solvent in the washing process.

No	Group	рН	Density (g/mL)	Water content (%)	Acid number	90/10 Test (%)	Viscosity (mm²/s)
1	Palm oil	7	0.891	2.333	0.699	1	4.195
	corn oil	7	0.893	3	0.699	1	578.8
2	NaOH catalyst	7	0.866	2.326	0.467	1	4.418
	KOH catalyst	7	0.892	2.333	0.467	3	4.765
3	Amount of catalyst 1 (c.o)*	7	0.895	1.333	3.615	0.5	4.613
	Amount of catalyst 2 (c.o)*	7	0.9	2	4.373	1	4.995
4	Room temperature	7	0.891	3.3	2.798	0.5	4.563
	Heating 50 °C	7	0.892	1.67	2.798	3	4.760

Table 4. Characterization of biodiesel: pH, density, water content, acid content, 90/10 test, viscosity

*Notes: (c.o) is corn oil

Density test

Density testing was performed using a pycnometer. According to SNI 7182:2015 standard, a good biodiesel density is between 0.850 and 0.890 g/mL. According to Pratas et al. (2011) and Baroutian et al. (2008), biodiesel had a density from 0.8215 to 0.8759 g/mL. The biodiesel density test results were presented in Table 4. All synthesized biodiesel was presented in the density range for biodiesel, according to SNI 7182: 2015.

Water content

Water content testing was done using an oven to find out the water content in the synthesized biodiesel. Biodiesel was heated for a certain length of time, and its mass changes were calculated to compare the changes before and after roasting. The results of the water content test were presented in Table 4. According to SNI 7182: 2015, the maximum water and

sediment content contained in biodiesel was 0.05%. The water contents of the biodiesel synthesized by students were in the range between 1.33 and 3.3%. This biodiesel exceeded the maximum limit of water content set by SNI 7182: 2015. This was maybe because there were remaining water contents when washing biodiesel.

Acid number

Biodiesel contained fatty acid methyl esters (FAME) and also free fatty acids that could be calculated in acid levels. These acid levels were defined in the number of milligrams of potassium hydroxide needed to neutralize 1 gram of biodiesel samples. According to ASTM D664 and European Biodiesel Standard (Mahajan, 2006), the number of acid number limit was 0.50. The value of the acid number obtained in biodiesel exceeded the standard of ASTM D664. This could be influenced by the titration process carried out in which the titration process was stopped when the endpoint of the titration was obtained—the pink solution. Oil or biodiesel titration had a few obstacles because the pink colour obtained was unstable, so it turned back to have no colour and led to the exact endpoint of the titration. Biodiesel synthesis was carried out only on a laboratory experimental work and not for commercial purposes. This synthesis was preferred for learning purposes.

90/10 test

The 90/10 test was a test conducted by (Gross et al., 2016) to detect the amount of unreacted biodiesel. This illustrated the perfection of the biodiesel reaction. If all biodiesel products reacted, the biodiesel product could be considered to pass 90/10 testing. The results of the 90/10 test were presented in Table 4. The results showed that almost all biodiesel reacts with the remaining biodiesel only around 0.5 - 3%. It could be concluded that almost all of the biodiesel reacts in biodiesel reaction.

Kinematic viscosity test

Kinematic viscosity testing was conducted at the Petroleum Gas and Coal Technology Laboratory, Department of Chemical Engineering, Faculty of Engineering, Universitas Gadjah Mada. Kinematic viscosity testing (mm²/s) was carried out at 40 °C by the ASTM D445 method. The kinematic viscosity of the ASTM D 445 method was measured by noting the time taken by oil to flow through capillary holes under the force of gravity. Based on ASTM D445 and EN ISO 3104, the kinematic viscosity testing of biodiesel from vegetable oils with

various variables were in the range of kinematic viscosity values at 40 °C according to ASTM D445 and EN ISO 3104.

Students' responses to learning activities

In the learning process of biodiesel synthesis, students found significant differences between guided-inquiry learning experiments and traditional laboratory work activities. Traditional laboratory work provided a series of experimental procedure design, so the students only conducted experiments based on given procedures. In contrast, the guided-inquiry learning experiments required the students to actively involved in conducting literature studies and designing experiments (Mistry et al., 2016).

The questionnaire was used to determine students' perceptions and responses to the guidedinquiry learning process. Student responses could be considered positive if $\geq 60\%$ of students chose the option of AGREE, or $\leq 40\%$ of them chose to disagree with the option of DISAGREE. The questionnaire sheet consisted of 16 questions and each of which had three options, namely "agree", "quite agree", and "disagree". This questionnaire was filled in by eighteen students. The questionnaire was used to determine the student's response to designing green chemistry-based experiments in biodiesel synthesis.

		Percentage of	Positive/		
No	Statement	Agree	<u>Quite</u> Agree	Disagree	Negative Response
1	I am interested in learning where I am asked to arrange the design for the experiment.	78.125	21.875	0	Positive
2	I am always eager to follow all the processes in preparing experimental designs.	78.125	21.875	0	Positive
3	I feel happy to follow the experiment because it is an experimental design that is obtained independently.	81.25	18.75	0	Positive
4	I always earnestly follow the laboratory work course.	78.125	18.75	3.125	Positive
5	I understand that the given material is about biodiesel synthesis because I am required to read more to be able to develop the most appropriate biodiesel synthesis experimental design.	65.625	31.25	3.125	Positive
6	I feel that I understand more deeply the basic concepts of the experiment because I have to design my experiment procedure as the research procedure is arranged from the beginning.	65.625	28.125	6.25	Positive
7	I understand the principles of green chemistry	81.25	18.75	0	Positive

Table 5. The results of the questionnaire responses of students in designing experimental procedures

		Percentage of Student Response (%)			Positive/
No	Statement	Agree	Quite Agree	Disagree	Negative Response
	that are applied in biodiesel synthesis.				
8	I have no difficulty in designing the biodiesel synthesis based on the principles of green chemistry.	28.125	65.625	6.25	Negative
9	I am interested in developing working procedures for biodiesel synthesis that are thoughtful and efficient in terms of material used.	75	25	0	Positive
10	I understand the factors that influence the success of biodiesel synthesis.	78.125	18.75	3.125	Positive
11	I am interested in finding literature on the composition of required materials in biodiesel synthesis.	68.75	28.125	3.125	Positive
12	I am motivated to find the most optimal composition of ingredients in biodiesel synthesis.	75	25	0	Positive
13	I feel that creating experimental designs of biodiesel synthesis increases my understanding and insight into organic chemistry.	90.625	9.375	0	Positive
14	I was motivated to find new research based on green chemistry.	87.5	12.5	0	Positive
15	I am motivated to design and develop new research with the concept of green chemistry principles.	90.625	9.375	0	Positive
16	Experiments on the synthesis of biodiesel based on green chemistry improved my skills in understanding current environmental problems.	90.625	9.375	0	Positive
Aver	Average		23.05	1.56	
Num	Number of positive responses				
Num	Number of negative responses				

Based on the overall results, 75.36% of students gave a positive response to the activity of designing experimental procedures while the remaining 1.56% of students showed a negative response. Positive responses were 15 questions, and the number of negative responses was 1 question. In particular, 81.25% of students felt happy to join the experiment because the experiment was designed independently by them. This finding was in line with the finding of Gross et al. (2016) in which students found the inquiry-based experiment was challenging yet engaging. Besides, 90.625% of students felt that creating of biodiesel synthesis experimental

designs helped them increase their understanding and insight into organic chemistry. This result showed that designing an experiment, students needed to find some relevant literature and this activity helped them to get a deeper understanding of the given topic. It can be concluded that the laboratory book manual is no longer the main source for laboratory work (Supasorn, 2012). 81.25% of students confirmed that they understood the principles of green chemistry that are applied in biodiesel synthesis. Also, 90.625% of students felt motivated to design and develop new research methods incorporating the concept of the principles of green chemistry. Experiments on the synthesis of biodiesel based on green chemistry improved the skills in understanding current environmental problems which were confirmed by 90.625% of students. This finding showed that the experiment helped students to be aware of sustainable developments and environmental problems (Listyarini, 2019). Lastly, it can be concluded that the response of students to the experimental procedures of green chemistry-based biodiesel synthesis is positive.

4. Conclusion

Based on the findings and discussion above, it could be concluded that the principle of green chemistry was successfully applied in the synthesis of biodiesel. Students were used green chemical principles, namely, designing safe synthesis processes (3), designing safe chemical products (4), using renewable feedstock (7), using catalysts (9), and designing chemicals which are easily degraded (10) designing biodiesel synthesis effectively and efficiently. Biodiesel products had a clear yellow colour, pH of 7, and density between 0.866-0.892 g/mL. Water contents ranged from 1.67-3.3, and acid numbers ranged from 0.69 - 4.3. In addition, 90/10 test show results ranged between 0.5 and 3% while kinematic viscosity ranged from 4.195 to 4.995. Students showed a positive response of 75.36% in designing experimental procedures for green chemistry-based biodiesel synthesis. In the future, it is promising to use guided inquiry on green chemistry-based experiments in other topics of organic chemistry in undergraduate courses. This will enhance the engagement of students since students have to actively design their experiment.

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