Purification of biodiesel from crude palm oil using multistage portable fixed bed adsorber

Pemurnian biodiesel dari minyak sawit mentah menggunakan multistage portable fixed bed adsorber

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INFO ARTIKEL	ABSTRACT

Crude biodiesel needs to go through purification process, to meet international biodiesel specifications.. The widely used adsorption column is in the form of a fixed bed column, where the contents of the column are stacked in a mess. This makes the adsorbent difficult to clean and fill, and irregular adsorbent accumulation can hinder the flow of the fluid to be purified. In this study, an adsorption column with a portable circulation system used to observe the influence of circulation and adsorption level on the purity of biodiesel. Main equipment of this research is a portable adsorption biodiesel purification device. The column consists of: top distributor, packed bed and bottom collector. The tool is made of 3D printed polymer material. At the bottom of the tower there is a buffer tank and a pump for circulating the biodiesel to be purified. The results showed that some analytical parameters based on the results of t-test, namely: density, acid value, saponification and yellowish-blue tendency, there are significant differences between the results before and after purification. For all the parameters analyzed in this study, the number of stages and fluid circulation gave significantly different results. The results of methyl ester analysis confirmed this, where the more cycles in the column, the higher the methyl ester peak produced. As number of cycles in the adsorption column increases, peak area of the methyl ester produced becomes wider, which can be explained by the ability of column and its packing materials to filter impurities present in column.

ABSTRAK

Biodiesel mentah perlu proses pemurnian yang tepat, yang harus memenuhi standar biodiesel internasional. Kolom adsorpsi yang banyak digunakan berupa kolom fixed bed, dimana isi kolom ditumpuk secara acak. Hal ini membuat adsorben sulit untuk dibersihkan dan diisi, dan akumulasi adsorben yang tidak teratur dapat menghambat cairan yang dimurnikan. Pada penelitian ini digunakan kolom adsorpsi dengan sistem sirkulasi portabel untuk melihat pengaruh sirkulasi dan tingkat adsorpsi terhadap kemurnian biodiesel. Peralatan utama yang digunakan adalah alat pemurnian biodiesel portabel. Kolom terdiri dari: distributor atas, packing bed dan kolektor bawah. Alat ini terbuat dari polimer cetak 3 dimensi. Di bagian bawah kolom terdapat buffer tank dan pompa untuk mensirkulasikan biodiesel yang akan dimurnikan. Hasil penelitian menunjukkan beberapa parameter analitik berdasarkan hasil uji-t yaitu: densitas, bilangan asam, saponifikasi dan uji warna biru kekuningan, terdapat perbedaan yang signifikan antara hasil sebelum dan sesudah pemurnian. Untuk semua parameter yang dianalisis dalam penelitian, jumlah tahapan dan sirkulasi cairan memberikan hasil yang berbeda nyata. Hasil analisis metil ester menegaskan hal tersebut, semakin banyak siklus pada kolom adsorpsi, semakin tinggi puncak metil ester dihasilkan. Dengan bertambahnya jumlah siklus dalam kolom, area puncak metil ester yang dihasilkan menjadi lebih luas, hal ini menjelaskan kemampuan kolom dan bahan isiannya untuk menyaring pengotor.

1. Introduction

Biodiesel is obtained from the transesterification of vegetable oils with short -OH chains, with or without the use of a catalyst. Many contaminants are still contained in crude biodiesel, depending on the transesterification technology used. Impurities such as residual methanol, glycerol, soap, catalysts, phospholipids, water and unreacted glycerides can still be found, which can adversely affect engine performance so that impurities must be removed(Berrios and Skelton, 2008; Shibasaki-Kitakawa et al., 2013).

Crude biodiesel requires a proper purification process which must meet international biodiesel standard specifications so that it is suitable for use by consumers. In addition, biodiesel purification aims to extend the shelf life of biodiesel before use. The main problems that often arise in the biodiesel washing process are emulsification and oxidation. Emulsification makes the separation process between water and biodiesel difficult. Oxidation reactions can cause polymerization which will form hydroperoxides which damage elastomeric goods (Argo and Djoyowasito, 2008). Purification of crude biodiesel usually requires 2 stages, namely wet washing and dry washing. In the wet washing process, for every 100 liters of biodiesel produced, about 20 liters of wastewater will be generated (Atadashi et al., 2011a). The water content after the wet washing process is around 0.15% which is still above the maximum standard (0.05%) so that a centrifugation or subsequent drying process is required under vacuum. Wastewater must be treated before final disposal to reduce environmental impacts due to its physicochemical characteristics which are characterized by high values of pH, COD, BOD, fats and oils, and require difficult and expensive materials to handle, thereby increasing operational costs (Atadashi et al., 2011b). Constraints in the wet washing process can be overcome by using the dry washing process. This dry washing process requires a mixing unit and the adsorbent used must be easily disposed of (Atadashi et al., 2011a). The biodiesel purification process using water (Wang et al., 2006), is carried out because the impurities have polar properties so they are easily bound by water. However, the use of water cannot bind non-polar materials (soap). In addition, the washing method with water has weaknesses, namely it requires a long processing time (up to 2.5 hours), requires large amounts of water and produces waste in the form of soap emulsion, glycerol, unreacted methanol, and large amounts of catalyst that cannot be disposed of immediately. just to the environment. The amount of liquid waste produced is about 30 percent of the amount of biodiesel produced. In addition, in this method a drying process must be carried out on the washed biodiesel to evaporate the remaining washing water contained in the biodiesel (Bryan, 2005).

The general use of adsorbents is for the adsorption of impurities in oil or triglyceride compounds (Foletto et al., 2006). Several studies have been conducted, including Widayat and Haryani (2006) regarding the adsorption process of used cooking oil with zeolite adsorbents, Purwadi et al. (1998) on the use of Indonesian natural zeolite as an adsorbent for liquid waste and fluidization media in the fluidization column, and Purwaningsih et al. (2000) regarding the utilization of palm oil shell activated charcoal as an adsorbent in plywood liquid waste.

The adsorption process can also be used in the biodiesel purification process. This has been done by Bryan (2005) who used a synthetic magnesium silicate (magnesol) adsorbent in the biodiesel purification process. However, these adsorbents generally still have to be imported. Therefore, local adsorbents are needed that can substitute for the use of these adsorbents in purifying biodiesel. Bentonite is a local mineral that has the potential to be used as industrial raw material with more than 25 applications (Murray, 1999), including the production of selective adsorbents, catalysts, and pharmaceuticals (Barrer, 1989; Moosavi, 2017; Pinnavaia, 1983).

According to Ketaren (Ketaren, 1986), the dye in the oil will be adsorbed by the surface of the adsorbent. The adsorbent will also absorb colloidal suspensions (gums and resins), free fatty acids and the results of oil oxidation such as peroxides. Adsorbents that can be used to remove impurities in oil include bentonite, activated charcoal, magnesium silicate, talc, attapulgite, aluminum silicate, and lime. The adsorbent used in the purification process consists of polar (hydrophilic) and non-polar (hydrophobic) types. Polar adsorbents include silica gel, activated alumina and some types of clay. This type of adsorbent is generally used if the dye to be removed is more polar than the liquid. Non-polar adsorbents include charcoal (carbon and coal) and activated charcoal, which are commonly used to remove less polar dyes (Kirk et al., 1978).

The adsorption of biodiesel impurities in a previous study by Saiful et. al. (Saiful et al., 2012) used the principle of magnesol membrane filtration from chitosan polymer as a medium for purification of biodiesel for total glycerol, but this membrane tends to have high selectivity so that it is not able to remove all impurities. Another membrane form developed is a silica-based hydrophobic membrane which is highly selective in the process of separating water from oil (Bambang et al., 2019).

The adsorption column that is widely used is in the form of a fixed bed column with column contents that are stacked in a mess. This makes it difficult to clean and fill the adsorbent, besides the irregular pile of adsorbents will hinder the flow of the fluid to be purified. In this study, an adsorption column with a portable and circulating system was used to see how the amount of circulation and the level of adsorption had on the purity of biodiesel.

2. Methods

The biodiesel used in this study was derived from the esterification reaction of crude palm oil carried out at a mini palm oil mill in Indonesia. The main equipment in this research is an adsorption-based biodiesel purification unit with a portable form as shown in figure 1. The purification column is in the form of a column with a number of plates as a place for the adsorber

material and increases the residence time so that the purification process can take place perfectly. The column consists of 3 parts, namely: top distributor, packing bed, and bottom collector (Bimantio et al., 2020). The tool is made of polymer material resulting from 3D printing. At the bottom of the column there is a buffer basin and a pump that is used for circulating the flow of biodiesel to be purified.

Purification is carried out by flowing crude biodiesel to the purification unit. In the purification unit, purification will be carried out with the independent variable, namely the total height of the adsorbent, 15 cm (sample code: A), 30 cm (sample code: B), and 45 cm (sample code: C). From each height, biodiesel circulation will be carried out as the second independent variable, where at each variable height, the circulation flow will be carried out 1 (sample code: 1), 5 (sample code: 5), 10 (sample code: 10), and 15 (sample code: 15) times to see the saturation level of the adsorbent. So, a total of 12 samples will be obtained. The filling materials used in packing bed include: zeolite, silica gel, ceramic ring, activated charcoal, and foam. Parameters analyzed included: density, color, acid number, saponification number, viscosity, water content, and methyl ester. These parameters are compared to variables using statistical tests such as t-test and ANOVA using SPSS software.
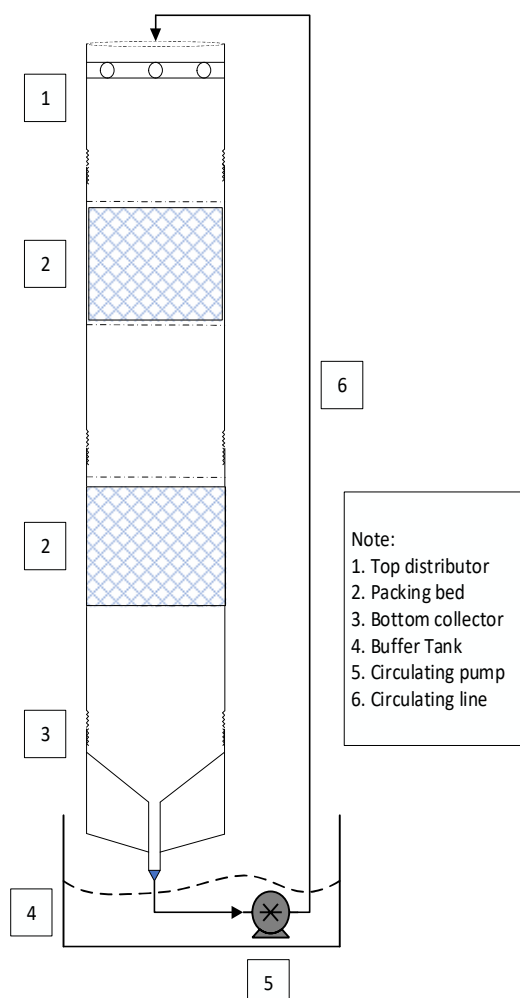

Note:
1. Top distributor
2. Packing bed
3. Bottom collector
4. Buffer Tank
5. Circulating pump
6. Circulating line

Figure 1. Biodiesel purification scheme using a portable adsorption column

3. Result and discussion
3.1 T-test

To find out whether the biodiesel purification process carried out can cause changes to the characteristics of the biodiesel, a T test was carried out between the samples before and after the adsorption process. Some of the observed characteristics were density, brightness (L*), red-green color tendency (a*), blue-yellow color tendency (b*), acid number, saponification number, viscosity and water content. The adsorbents used were ceramic ring, silica gel, sponge, activated charcoal and zeolite. T-test results for all parameters are presented in table 1.

Density is an important characteristic of a fuel. Density can indicate the amount of mass of fuel injected into the fuel chamber per unit volume (Pratas et al., 2011). The T test showed that the mean density of biodiesel after purification was significantly different from the density of biodiesel before it was purified. Based on the data, the density of biodiesel has decreased. The density of biodiesel is influenced by the presence of impurities in it. These impurities include: residual glycerol, catalyst, and residual methanol. The purification process can reduce the number of impurities so as to reduce the density value.

The biodiesel purification process did not produce a significant change in the brightness (L*) and the red-green color (a*) of the biodiesel. However, the purification process was able to produce a significant difference to the yellow-blue color tendency (b*). A positive b value indicates the intensity of the yellow color in the material. The lower mean value of b* after purification indicates that biodiesel has decreased the intensity of the yellow color. The decrease in intensity indicates the color is getting more and more bright yellow as shown in figure 2. One of the reasons for this change is the presence of activated carbon as an adsorbent. The use of activated carbon as an adsorbent can change the color of biodiesel from brownish red to bright yellow (Ngernyen et al., 2020a).

The acid number indicates the impurity in the form of acid in biodiesel. Free fatty acids, carboxylic acids are some examples of acids that can be an impurity. Table 1 shows that there are significant differences in the value of the acid number before and after purification. The adsorption process that occurs during purification is able to trap these acids thereby reducing the acid number. The use of zeolite as an adsorbent material can actively adsorb impurities contained in biodiesel (Dwi et al., 2018).

Purification of biodiesel resulted in a significant difference in the amount of saponification. The saponification rate in biodiesel after purification is higher than before purification. The higher the number of saponification indicates that biodiesel has a relatively low molecular mass (Febriani and Dewi, 2012). The adsorbent used can trap impurities that have a high molecular mass. The use of zeolite is known to increase the rate of saponification due to its ability to adsorb impurities and free fatty acids (Purnama et al., 2014).

Figure 2. Sample of biodiesel purification result (most left: blank sample; most right: sample A15)

Biodiesel viscosity that is too thick will have difficulty flowing, pumping and ignition, while if it is too dilute it will be difficult to burn (Leonardo et al., 2015). The purification process does not have a significant effect on the viscosity value of biodiesel. The viscosity shown in this study is absolute viscosity. Kinematic viscosity can be obtained from absolute viscosity divided by density. The result is that the kinematic viscosity before purification is 19.49 mm2/s and the average after purification is 18.83 mm2/s. In accordance with the requirements of SNI 7182:2015

regarding biodiesel quality requirements, the required kinematic viscosity value is 2.3-6.0 mm2/s. The value of viscosity is inversely proportional to its density. The higher the density, the lower the viscosity, and vice versa (Lapuerta et al., 2010).

The purification process did not cause significant changes in the water content of biodiesel. The use of adsorbent material has not been able to reduce the water content. High water content in biodiesel can form acid, foam and reduce the heat of combustion (Aziz et al., 2012).

Table 1.
T-Test results of testing parameters before and after biodiesel purification

Sample	Density [g/cm³]	Color L	Color a	Color b	Acid Number [mg/g]	Saponification [mg/g]	Viscosity [cp]	Water Content [%]
Average Before	0.8850	24.76	11.26	6.31	1.7665	151.0318	17.25	0.22045
Average After	0.8819	23.76	11.3475	4.8467	1.3327	182.7633	16.6042	0.2670
Significance	$6.33e^{-8}$	0.051	0.68	0.006	$1.67e^{-10}$	$1.62e^{-10}$	0.151	0.089
Difference	Yes	No	No	Yes	Yes	Yes	No	No

3.2 Two way anova test

The parameters of the height/number of trays and the number of circulations were tested further to see if each parameter had an effect on the test results and whether

there was an interaction between the two parameters. The results of statistical tests using the two-way ANOVA method are as presented in the tabulation of table 2.

Table 2.
Tabulation of two way anova test results

Sample	Test	Color L	Color a	Color b	Acid Number [mg/g]	Saponification [mg/g]	Viscosity [cp]	Water Content [%]
Tray	Difference	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Recycle	Difference	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Tray*Recycle	Interaction	Yes	Yes	Yes	No	Yes	Yes	No

The results of the ANOVA test for the color brightness parameter (L*) showed a direct effect on the number of trays on the color brightness indicated by the F value of 82.509 (high F) and Sig 9.705e-8 (low Sig). The R square value of 0.966 indicates that the variability of the number of trays and the duration of circulation affect the brightness of the color by 96.6%.

Meanwhile, the results of the ANOVA test for magenta/red showed that there was a direct effect on the number of trays on the magenta/red color, indicated by the F value of 28.678 (high F) and Sig 2.683e-5 (low Sig). The R square value of 0.904 indicates that the variability of the number of trays and the duration of circulation affect the magenta/red color trend by 90.4%.

For the yellow color, the ANOVA test results show that there is a direct effect on the number of trays on the clear yellow color, indicated by the F value of 338.125 (high F) and Sig 2.809e-11 (low Sig). R square value of 0.991 indicates that the variability of the number of trays and the duration of circulation affect the tendency of clear yellow color by 99.1%.

The biodiesel purification process did not produce a significant change in the brightness (L*) and the red-green color (a*) of the biodiesel. However, the purification process was able to produce a significant difference to the yellow-blue color tendency (b*). A positive b value indicates the intensity of the yellow color in the material. The large number of trays used can increase the contact between biodiesel components and impurities (residual glycerol and impurities) so as to facilitate separation based on density. In addition, the use of activated carbon as an adsorbent material can change the color of biodiesel from brownish red to bright yellow (Ngernyen et al., 2020b).

The results of the ANOVA test on the acid number parameter showed that there was a direct effect on the duration of circulation on the acid number value indicated by the F value of 32.230 (high F) and Sig 5.053e-6 (low Sig). The R square value of 0.923 indicates that the variability of the number of trays and the duration of circulation affect the amount of biodiesel acidity by 92.3%. The duration of biodiesel purification circulation tends to affect the acid value because the longer the biodiesel deposition, the better the separation between the glycerol and biodiesel phases. In addition, the use of zeolite as one of the active adsorbent materials can adsorb impurities contained in biodiesel (Udyani et al., 2018).

The results of the ANOVA test showed that there was a direct effect on the duration of circulation on the value of the saponification rate indicated by the F value of 131.634 (high F) and Sig 1,601e-7 (low Sig). The R square value of 0.983 indicates that the variability of the number of trays and the duration of circulation affect the amount of biodiesel saponification rate of 98.3%. The duration of biodiesel purification circulation tends to affect the acid number because the longer the biodiesel deposition, the better the separation between the glycerol and biodiesel phases, so that the saponification rate will

be higher. In addition, the use of zeolite is known to increase the rate of saponification due to its ability to adsorb impurities and free fatty acids (Purnama et al., 2014).

The number of trays on the viscosity value shows a direct effect as indicated by the F value of 962.161 (high F) and Sig 5.665e-14 (low Sig). The value of R square of 0.999 indicates that the variability of the number of trays and the duration of circulation affect the amount of biodiesel viscosity by 99.9%. The large number of trays used can increase the contact between biodiesel components and impurities (residual glycerol and impurities) making it easier to separate based on density, where the amount of viscosity is directly proportional to its density (Damayanti et al., 2018).

The ANOVA test showed that there was a direct effect on the duration of circulation on the water content, indicated by the F value of 6.178 (high F) and Sig 0.0088 (low Sig). The value of R square of 0.746 indicates that the variability of the number of trays and the duration of circulation does not significantly affect the amount of water content of biodiesel by 74.6%. The use of adsorbent material is more decisive in reducing the water content, where the type of adsorbent used has not been able to reduce the water content. High water content in biodiesel can form acid, foam and reduce the heat of combustion (Aziz et al., 2012).

3.3 Methyl esther analysis

The biodiesel used in this study was analyzed for its content after the filtration process with a Portable Fixed Bed Composite Adsorber using Gas Chromatography – Mass Spectrometry (GCMS). The analysis was carried out at the Organic Chemistry Laboratory, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Gadjah Mada University. The results of the GCMS analysis used helium as a carrier gas. There are 4 peaks in the chromatography results which indicate the presence of substances that are suspected to be methyl esters as shown in figure 3. The types of methyl esters detected were: methyl caprate, methyl myristate, methyl laurate, methyl stearate, methyl oleate, methyl petroselinate, and methyl lignocerate.
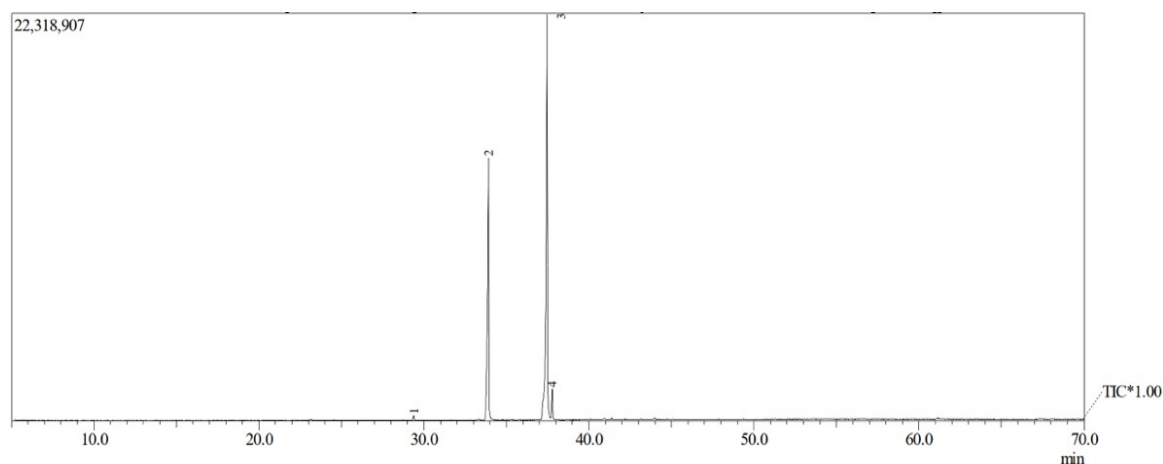

Figure 3. GCMS results from adsorption biodiesel samples (Sample C15)

Table 3.
Peak height results of GCMS analysis of purified biodiesel samples

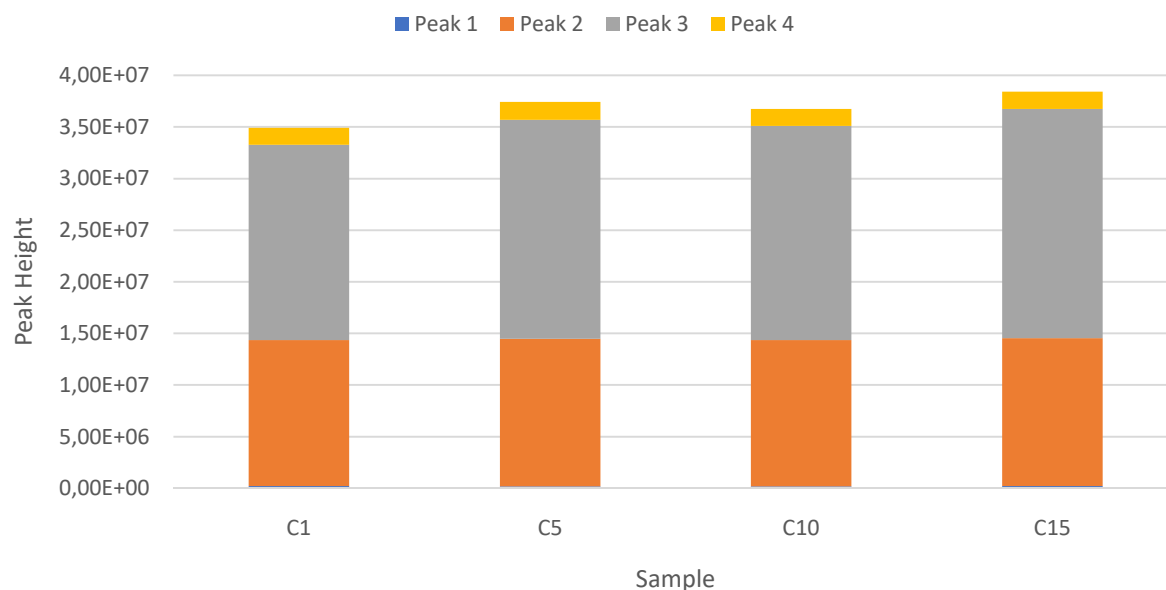|	Sample			
	C1	C5	C10	C15
Peak 1	223,413	184,162	208,338	229,648
Peak 2	14,133,706	14,298,671	14,153,400	14,317,028
Peak 3	18,933,697	21,209,642	20,755,772	22,208,983
Peak 4	1,622,378	1,745,891	1,624,636	1,671,268
Total	34,913,194	37,438,366	3,674,2146	3,842,6927

Figure 4. Comparison of methyl esters peak heights for filtration result samples

In Table 3 and Figure 4 it can be seen that the increase in the total peak height from the GCMS analysis results, that the more the circulation in the adsorber column, the higher the methyl ester peak produced, this can be interpreted as the ability of the column and its filling material to filter impurities in biodiesel. The higher the total peak height, the more methyl ester content in the filtered biodiesel sample.

The same thing also happened for the peak area, as shown in Table 4 and Figure 5. With the increasing number of circulations in the adsorber column, the wider the peak area of the methyl ester produced, this can be interpreted as the ability of the column and its filling material to filter out impurities present in the column. The wider the peak area, the more methyl ester content in the filtered biodiesel.

Table 4.
Results of peak area GCMS analysis of purified biodiesel samples

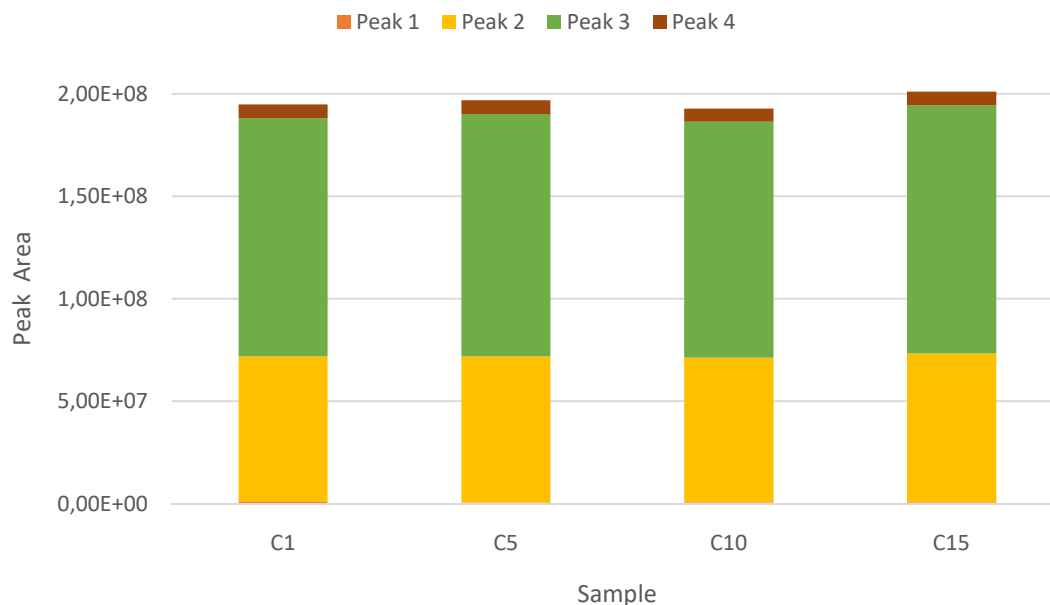|	Sample			
	C1	C5	C10	C15
Peak 1	846,866	511,005	574,901	676,196
Peak 2	71,013,242	71,449,720	70,646,788	72,671,402
Peak 3	116,341,970	118,210,832	115,213,339	121,056,670
Peak 4	6,645,178	6,719,260	6,359,479	6,792,267
Total	194,847,256	196,890,817	192,794,507	201,196,535

Figure 5. Comparison of methyl esters peak areas for filtration results

4 Conclusions

The biodiesel purification process carried out using multistage portable fixed bed adsorber can cause changes to the characteristics of the biodiesel. There is significant difference result between before and after purification for some analyzed parameter based on t-test result, namely: density, acid number, saponification, and yellow-blue color tendency. Number of stage and fluid circulation gave significant different result for all parameter analyzed in this research. This is confirmed by the results of methyl ester analysis, where the more the circulation in the adsorber column, the higher the methyl ester peak produced. With the increasing number of circulations in the adsorber column, the wider the peak area of the methyl ester produced, this can be interpreted as the ability of the column and its filling material to filter out impurities present in the column. The types of methyl esters detected were: methyl caprate, methyl myristate, methyl laurate, methyl stearate, methyl oleate, methyl petorselinate, and methyl lignoserat.

Acknowledgements

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