

ORIGINAL RESEARCH

A MASS RATIO OF HIERARCHICAL H-ZSM-5 AND FATTY ACID METHYL ESTER OF CERBERA MANGHAS OIL AND THE EFFECT ON THE HYDROCARBON LIQUID PRODUCT COMPOSITION

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Abstract

Fossil fuels are classified as non-renewable resources. On the other hand, using fossil fuels has further aggravated the impact of the environmental crisis. The development of alternative fuels that are renewable and renewable is urgently needed in line with the increasing need for fuel. One alternative energy source that can be used in the development of biofuels is Cerbera manghas oil. The catalytic cracking process with 178 g of Cerbera manghas oil can be carried out using hierarchical H-ZSM-5 as a catalyst with a mass of 0.1, 0.2, 0.3, 0.4, and 0.5g at a temperature of 250°C. Gas Chromatography Analysis–Mass Spectrometry (GC-MS) was performed on Cerbera manghas oil and biofuel products. Several hydrocarbons, C7-C12 and C16-C17, with the largest content, were identified as diesel (29.14%) and gasoline (14.08%) using a catalyst mass of 0.5g. So that the use of hierarchical H-ZSM-5 catalyst in the catalytic cracking of Cerbera manghas oil can produce hydrocarbon range gasoline.

KEYWORDS:

Biofuel, Cracking, Cerbera Manghas Oil, Fatty Acid Methyl Ester, H-ZSM-5

1 | INTRODUCTION

Energy consumption in Indonesia continues to increase drastically every year, as the supply of fossil fuels in Indonesia and even the world has been running low^[1]. According to the Ministry of Energy and Mineral Resources of the Republic of Indonesia (2017), crude oil production in Indonesia from 2012 to 2017 continued to decline. Crude oil production in 2012 reached 859,743 BOPD, while in 2017, it only reached 801,024 BOPD. Suppose petroleum fuel consumption in Indonesia stays the same, and

there is no effort for a new alternative. In that case, it is estimated that petroleum reserves in Indonesia will be exhausted in the next 12 years.

The use of fossil fuel-based energy harms the Environment. The increase in the environmental crisis caused by the use of fossil fuels demands the search for alternative energy that is environmentally friendly and renewable. One alternative source of energy that can be developed to be converted into biofuels is biomass. The development of biofuels derived from alternative renewable energy sources, biomass, makes a major impact on the development of the fuel industry and environmental sustainability^[2-4].

One of the efforts made by the government to overcome the energy crisis is to develop renewable resources in the form of biofuels. The advantage of biofuels is that they can be produced quickly, are available (renewable sources), do not produce harmful emissions, are cheap, and are easy to obtain. In addition, the carbon content contained in vegetable oil triglycerides is similar to that found in fossil fuels^[5]. One of the plant candidates that have the potential to be developed as a biofuel is *Cerbera manghas* L or bintaro fruit.

Cerbera manghas seeds are non-food plants, so their use as raw materials for biodiesel will not compete with food needs. *Cerbera manghas* plants are usually used as greenery plants and dry flower crafts because *Cerbera manghas* is known to have toxins in all parts of the plant, so the community does not widely use it, and its economic value is still low. Bintaro seeds contain between 35-50 wt.%^[6]. Atabani et al.^[7] reported that *Cerbera manghas* seed oil has a content of 54 wt.% oleic acid is the main content of *Cerbera manghas* seed oil. The potential for oil yields produced from *Cerbera manghas* seeds provides a great opportunity to convert *Cerbera manghas* seed oil into biofuels or biofuels^[8, 9]. However, this conversion process requires high temperature and pressure, the use of hydrogen gas, and a longer reaction mechanism, as reported in previous studies^[6]. Prajitno et al.^[6] reported that hydrocarbon compounds containing oxygen atoms were still found in the produced biofuels. Therefore, a strategy is needed to shorten the reaction mechanism by using Fatty Acid Methyl Ester (FAME) from *Cerbera manghas* seed oil to obtain biofuel, which is equivalent to fuel oil. In addition, FAME is used as a raw material for making biofuels and a way to improve its quality as a biofuel because it contains compounds with low oxygen atoms.

The process that can be an option in the process of making biofuels for upgrading FAME is the catalytic cracking process. The catalytic cracking process is a process of breaking long-chain hydrocarbon compounds into short-chain hydrocarbon compounds. In the catalytic cracking process, solid catalysts will break high molecular weight compounds in FAME into small molecular weight compounds such as gasoline, kerosene, and diesel. The catalytic cracking process uses a cracking reactor that requires less heat than thermal cracking. In addition, catalytic cracking has a higher product conversion when compared to hydrocracking, so this process is more economical and efficient than the previous study^[8, 10, 11]. The catalytic cracking process is carried out without adding hydrogen and can occur under atmospheric pressure conditions^[2].

Hierarchical H-ZSM-5 is one of the effective acid catalysts for cracking fatty acids into kinds of paraffin, olefins, and aromatic compounds. The results obtained from using H-ZSM-5 hierarchical zeolite catalysts are higher product yields than other zeolite catalysts^[12]. Hierarchical H-ZSM-5 has a three-dimensional shape containing aluminum, silicon, and oxygen as the main structure. The hierarchical H-ZSM-5 has similarities with the H-ZSM-5 but has a different pore size. The pore size in hierarchical H-ZSM-5, which is larger than H-ZSM-5, is due to the hierarchical H-ZSM-5 obtained from the desacralization process of H-ZSM-5 in alkaline solutions.

This study was conducted to determine the effect of the mass of the hierarchical H-ZSM-5 catalyst on the hydrocarbon liquid product composition and the yield product produced in the catalytic cracking process of Fatty Acid Methyl Ester (FAME) of *Cerbera manghas* oil. The raw materials for *Cerbera manghas* seed oil, FAME, and the resulting liquid product were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) to determine hydrocarbon compounds and hydrocarbon groups in range fuel oil.

2 | PREVIOUS RESEARCHES

More previous studies discussed catalytic cracking of Fatty Acid Methyl Ester *Cerbera manghas* oil, as shown in Table 1.

3 | MATERIAL AND METHOD

TABLE 1 Previous research studies.

Researcher	Title Study	Method	Product
Arumugam et al. ^[12]	Hierarchical HZSM-5 for Catalytic Cracking of Oleic Acid to Biofuels	Hydrothermal	Silanization on the nanozeolite surface before aging experienced mesoporous and hierarchical systems formation on ZSM-5. The presence of mesopores leads to the increased access of large lipids to acid sites and promotes deoxygenation within the hierarchical zeolite framework to short-chain hydrocarbons (65% versus 20–25% for conventional HZSM-5).
Hendra et al. ^[13]	Karakteristik Biodiesel Biji Bintaro (Cerbera manghas L) dengan Proses Modifikasi	Process Modification	The physical and chemical properties of bintaro oil, which is processed into biodiesel, meet Indonesian standards (BSN-2006) except for water content. Modification of the process in the manufacture of biodiesel from bintaro seed oil with the addition of 1.5% bentonite during degumming and 1.5% zeolite during esterification and in the transesterification process accompanied by the addition of the catalyst mixture of 20% (v/v) methanol and 0.6% (w/v) KOH was able to reduce the acid number of Bintaro seed oil from 6.33 mg base/g to 0.47 mg base/g. This acid number complies with SNI 04-7182-2006. This acid number complies with SNI 04-7182-2006.

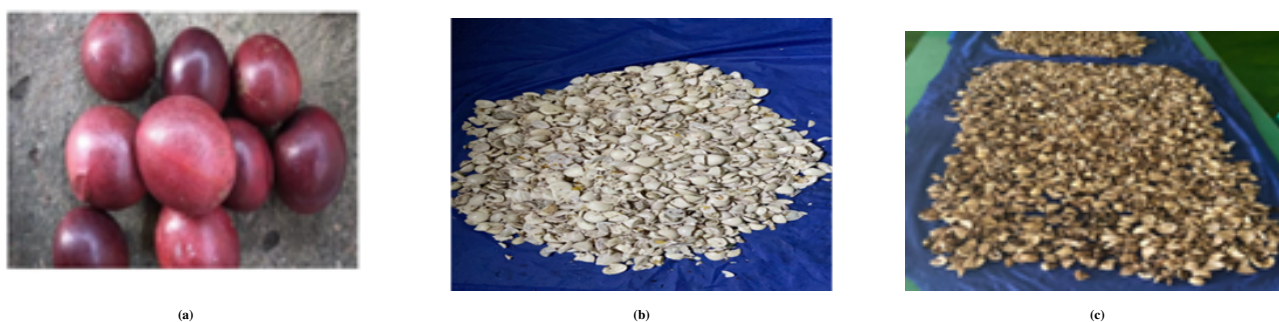


FIGURE 1 The drying of (a) Cerbera manghas fruit; (b) fresh and (c) dried Cerbera manghas seeds.

3.1 | Chemicals

The materials used in this study were bintaro fruit seeds, methanol (water $\leq 0.003\%$), potassium hydroxide ($geq85\%$), sulfuric acid (32%), phosphoric acid (85%), and hierarchical H-ZSM-5 (hH-ZSM-5). A previous study reported the characterization of hH-ZSM-5 used in this study^[14].

3.2 | Procedure

3.2.1 | Extraction of Cerbera Manghas Oil

Cerbera manghas fruit is also known as buto-buto or bintaro in Jambi, Indonesia. As a raw material, Cerbera manghas seeds obtained from plants growing in Muaro Jambi and Merangin, Jambi Province, was dried in sunlight for five days. Drying was carried out to reduce the moisture content in Cerbera manghas seeds. Fresh and dried seeds are shown in Fig. 1. Preparation of Cerbera manghas seeds before drying was carried out by separating the seeds and flesh of the fruit for washing. The washing process was carried out to reduce the gum content and remove impurities carried away during the stripping process. Dried Cerbera manghas seeds were cut and extracted using an oil press machine to obtain seed oil. This extraction process produced 6.5 liters as raw material from 12.6 kg of dried seeds. The degumming process of Cerbera manghas oil is carried out by adding 85% of H_3PO_4 as much as 0.2% by weight oil and heated and stirred at 70°C for 30 minutes. Distilled water was added to a solution at 55°C as much as 10% (v/v) and heated for 10 min. The degumming process was carried out to obtain gum- and water-free Cerbera manghas oil through separation using a separatory funnel for 24 hours.

3.2.2 | Esterification-Transesterification of Cerbera Manghas Oil

Cerbera manghas oil was processed with a mixture of 49.5 mL methanol and 5.5 mL H_2SO_4 catalyst. The mixture is heated at 60–70°C for 2 hours. The reflux results are then separated using a separatory funnel for 24 hours to obtain the esterified oil.

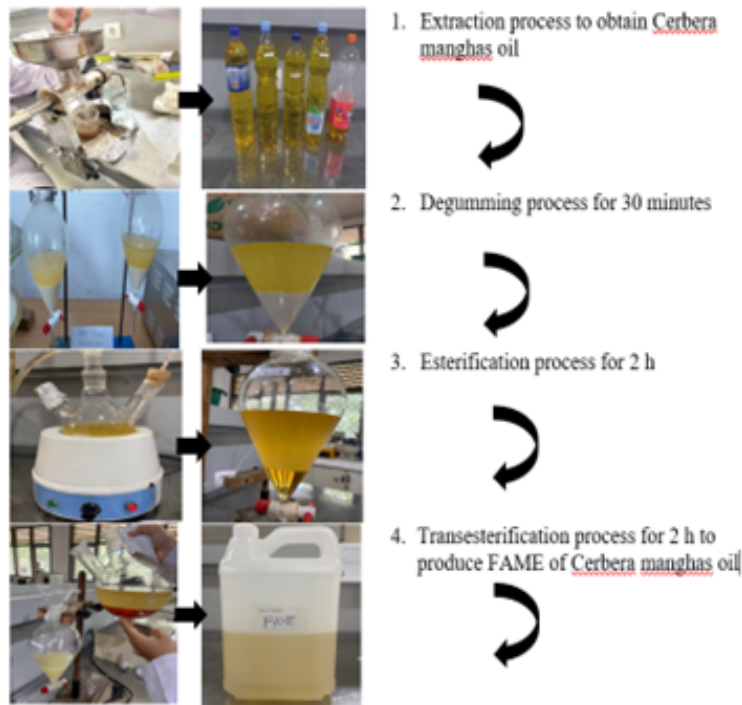


FIGURE 2 FAME production process stages in this study.

Cerbera manghas oil was transesterified oil on 961 mL of esterified oil in a 2000 mL three-neck flask. At this stage, a mixture of oil, methanol, and KOH of 1% by weight oil. The ratio of methanol to oil is 12:1. The transesterification process continues at a temperature of 60-70°C for 2 hours. FAME of *Cerbera manghas* oil was obtained through a separation process from transesterification using a separatory funnel for 24 hours. FAME purification was carried out by washing distilled water until a neutral pH was obtained. The washed FAME is then heated and stirred at 110°C until it has a clear and non-foaming color. The result can be named with FAME of *Cerbera manghas* oil as raw material in catalytic cracking. The density of FAME was measured, and the hydrocarbon composition of FAME was determined by Gas Chromatography-Mass Spectrometry (GC-MS). The water content, density, and hydrocarbon composition value are based on the standard value of biodiesel with SNI 04-7182-2006 so that it can be continued to the next stage, namely the catalytic cracking process.

3.2.3 | Catalytic Cracking of FAME

FAME of 200 mL was fed into the cracking reactor with the addition of a hierarchical HZSM-5 catalyst (hH-ZSM-5). The mass variations of the catalysts used were 0.1, 0.2, 0.3, 0.4, and 0.5 g. The cracking process was carried out at 250°C for 2 hours. After reaching the reaction time, the reactor was cooled until it reached room temperature. Condensed liquid products were collected and recorded as light fractions. At the same time, the remaining FAME in the reactor was recorded as heavy oil. The volume of each product was calculated to determine the yield of the biofuel product produced. The content and percentage of hydrocarbons and the distribution of hydrocarbons based on the amount of carbon in the biofuel products produced are known through Gas Chromatography-Mass Spectrometry (GC-MS) analysis using Wiley275 and NIST02 mass spectral library of data. Agilent HP 6890 GC equipped with capillary columns 0.25 mm in diameter, column length 30 mm, and film thickness 0.25 μm , Agilent model 19091S-433 (HP-5MS, penylmethylsiloxane 5%). As reported by Barron et al.(10), Hydrocarbon compounds (i.e.paraffins, isoparaffins, cycloparaffins, aromatic, olefins) are grouped in gasoline-range hydrocarbons (C5-C9), kerosene range hydrocarbons (C10-C13), and gasoil range hydrocarbons (C14-C22) as reported by Barrón et al.^[15]

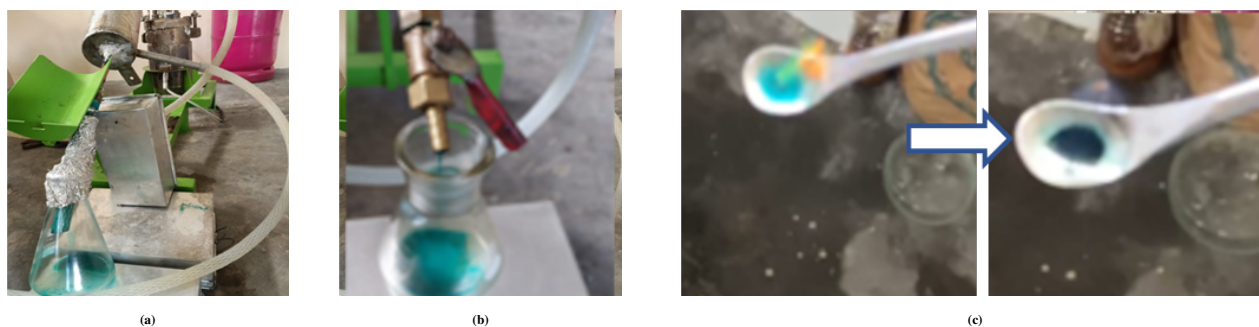


FIGURE 3 Catalytic cracking process (a), cracking liquid products (b), flame test on cracking liquid products (c).

4 | RESULTS AND DISCUSSION

Seed oil produced from the extraction process was degummed, esterified, and transesterified. All of these stages can be seen in Fig. 2 .

Esterification is one of the stages in the manufacture of biodiesel or FAME, which aims to reduce the number of free fatty acids in vegetable oils. The reaction between methanol and fatty acids occurs in this esterification process. Methanol was chosen as the reactant alcohol because it is polar and has the shortest carbon atom structure in the alcohol group. Using methanol can accelerate the reaction of methanolysis with fatty acids.

This esterification produces a product with two very different layers, separating it easily. The top layer consists of glycerol and the remaining acid and methanol. Then the bottom layer founded as a mixture of methyl esters and a little impurity that is still followed, as shown in Fig. 2 . After being separated, the volume of oil from the esterification process was obtained as much as 4.5 liters. Furthermore, this oil was processed again at the transesterification stage. Finally, the FAME of *Cerbera manghas* oil was obtained as much as 4.26 liters.

4.1 | Catalytic Cracking of Fatty Acid Methyl Ester (FAME)

The stages of the catalytic cracking process and the liquid product produced can be seen in Fig. 3 . GC-MS analysis was carried out on five samples produced using five masses of the hierarchical H-ZSM-5 catalyst.

One of the samples of the liquid cracking product is tested by burning the fire above the liquid, as shown in Fig. 3 . At first, the flame looked red, slowly showing a blue flame and leaving black combustion products. This shows that this liquid product still needs to be carried out at the next cracking stage to maximize the hydrocarbon compounds that are equivalent to the hydrocarbon composition of fuel oil resulting from petroleum processing.

4.2 | Characterization of FAME *Cerbera Manghas* Oil

Drying of *Cerbera manghas* seeds was carried out for five days using sunlight. The extraction process of *Cerbera manghas* oil is obtained using an oil press machine against dried *Cerbera manghas* seeds. *Cerbera manghas* oil is produced through degumming, esterification, and transesterification to produce FAME. Density analysis and Gass Chromatography-Mass Spectrometry have been tested on *Cerbera manghas* oil and FAME of *Cerbera manghas* oil. Based on SNI 7182:2015, the quality standard of FAME density in Indonesia is 0.85-0.89 g/cm³. The results were carried out by the ASTM D 1298 test method. FAME *Cerbera manghas* oil produced in this study was 0.89 g/cm³. The density value obtained was following FAME quality standards in Indonesia.

Fig. 4 (a) shows that the compounds with a retention time from 15.6 to 18.26 min in the *Cerbera manghas* oil disappear after the catalytic cracking reaction. 9-octadecenoic acid (oleic acid) of 50.98% area was detected in the largest content at a retention time of 17.87, and Palmitic acid of 0.11% area was detected in a retention time of 22.59 min. The same was also found by Marlinda et al.(3) that oleic acid of 76.21% area is the main content in *Cerbera manghas* oil.

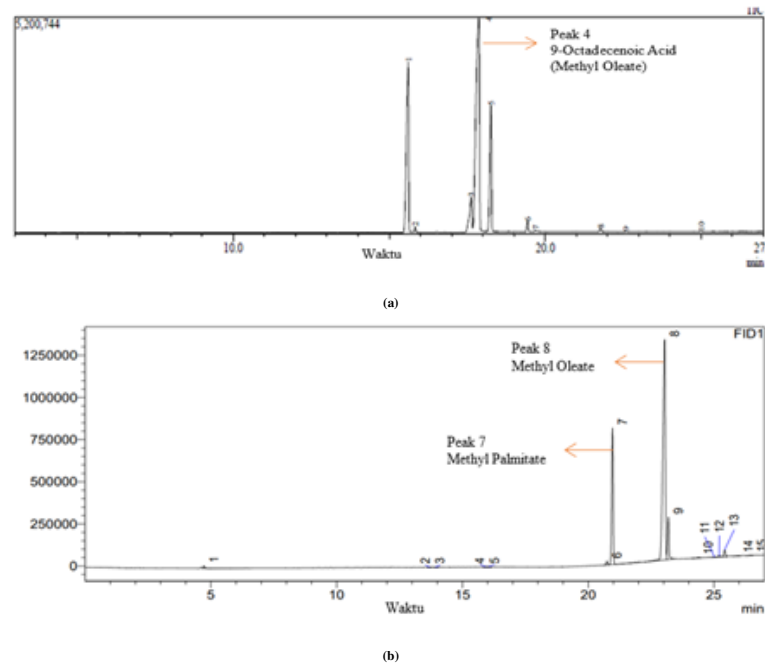


FIGURE 4 GC-MS spectra of (a) Cerbera manghas Oil, and (b) FAME of Cerbera manghas Oil.

Fig. 4 (b) shows chromatograms of FAME produced in the transesterification reaction. The hydrocarbon compounds in FAME appear in retention time from 20.96 to 23 min. The FAME was dominated by methyl oleate of 63.99% area detected in a retention time of 23.038 min and 7.334% area detected in a retention time of 23.186 min. As for methyl palmitate, 24.054% area was detected in a retention time of 20.96 min. In SNI 7182-2015 for biodiesel standards, It is stated that the methyl ester content is at least 96.5%. In this study, the methyl ester content reached 100%, so the FAME produced follows this standard.

4.3 | Analysis of Biofuel

Fig. 5 shows that the number of shorter-chain hydrocarbon compounds hydrocarbon components of the liquid product increases at a retention time of 2-9 minutes. When the compounds disappear at a retention time of 20-23 minutes, as shown in Fig. 4 (b), other hydrocarbon compounds appear at a retention time of 18-28 min after the cracking reaction. The liquid products still contain methyl nonanoate ($C_{10}H_{20}O_2$), methyl decanoate ($C_{11}H_{22}O_2$), methyl 14-methylpentadecanoate ($C_{17}H_{34}O_2$), and 14-Octadecenoic acid, methyl ester ($C_{19}H_{36}O_2$) in the range of 11–30% area which detected in retention time of 12.57, 17.9, 20.6 and 20.5 min, respectively.

Fig. 5 shows that methyl 14-methylpentadecanoate still has high content, i.e., 29.14, 16.18, and 20.51% area, respectively. However, pentadecane was detected at a retention time of 22.4 minutes, as shown in Fig. 5 (a,c), about 3.5% area. In addition, methyl decanoate of 12.22 and 11.21% area are presented in Fig. 5 (d) and Fig. 5 (e), respectively. The higher pentadecane content is about 7% area and a small amount of compounds (0.03 – 0.6% area), i.e., hexane (C_7H_{16}), nonane (C_9H_{20}), decane ($C_{10}H_{22}$), undecane ($C_{11}H_{24}$) were found in the use of a catalyst mass of 0.4 and 0.5 g. However, 14-Octadecenoic acid methyl ester was still found in high content, around 14.95% area at the retention time of 20.5 minutes, as presented in Fig. 5 (e). The compounds were classified as oxygenated compounds. This phenomenon explains that the hH-ZSM-5 catalyst plays an important role in a cracking reaction.

Fig. 6 shows the hydrocarbon compounds obtained in hydrocarbon liquid product resulting from the catalytic cracking of FAME Cerbera manghas oil for 2 hours at 250°C at a time retention range of 17-22 min. Seven hydrocarbon compounds are found in products such as paraffin, cycloparaffins, olefins, cycloolefins, aromatic hydrocarbons, carboxylic acids, and compounds with oxygen content. It illustrates the amount of carbon of hydrocarbon compounds produced in the product cracking of FAME Cerbera manghas oil. In the use of catalysts hierarchical HZSM-5 0.1 g, 0.2 g, and 0.3 g produce hydrocarbon compounds belonging to the solar or light diesel group with carbon amounts C16-C17. Meanwhile, the use of hH-ZSM-5 produces biofuels

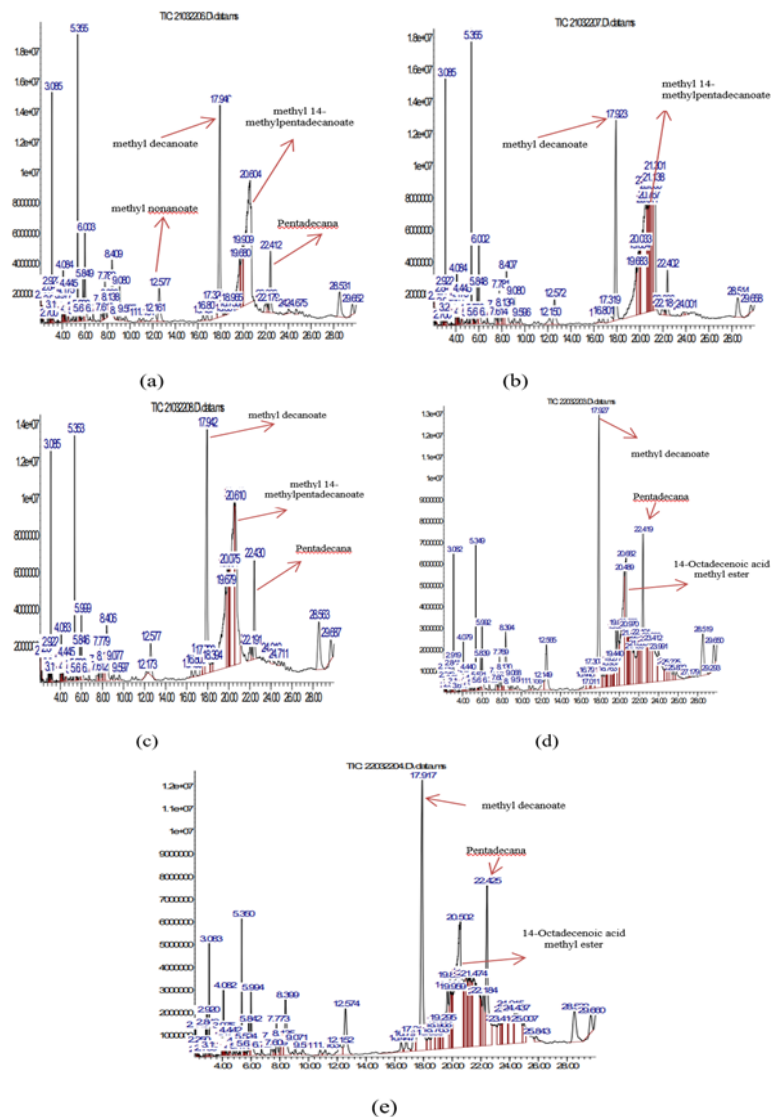


FIGURE 5 GC-MS spectra of cracking liquid product for hH-ZSM-5 mass (g): (a) 0.1 (b) 0.2 (c) 0.3 (d) 0.4 (e) 0.5 (operation condition: 200 mL of FAME, FAME density of 0.89 g/cm³ and 250°C in the catalytic cracking process).

with hydrocarbon compounds that are classified as gasoline groups (C7-C12). The performance of the hierarchical HZSM-5 has shown a tendency to produce gasoline range hydrocarbons in the use of catalysts mass 0.4 and 0.5 g.

4.3.1 | Effect of Catalyst Mass on Hydrocarbon Liquid Product Composition

Hancsó et al.^[16] stated that determining the composition of the hydrocarbons of the liquid products resulting from cracking is important for evaluating the properties of the contained biofuels. Fig. 6 shows the hydrocarbon content found in the liquid product against the use of the different masses of the hH-ZSM-5 catalyst. It is seen that with the mass catalyst use of 0.1 g, the resulting liquid product contains methyl ester of 75.06% area and cycloparaffin of 10.89% area. In addition, there was a hydrocarbon group, i.e., paraffin, olefin, cycloolefin, and aromatic, of 6.98, 5.25, 0.88, and 0.66% area, respectively. The group of compounds with the smallest area was an oxygen content of 0.28% area.

In liquid products using an hH-ZSM-5 catalyst of 0.2 g, there was a decrease in the number of paraffin, cycloparaffin, olefin, cycloolefin, and aromatic compounds. Meanwhile, methyl ester and compounds with oxygen content increased to 82.98 and 0.38% area, respectively. When the mass of the catalyst used was 0.3 g, there was a decrease in cycloparaffin, cycloolefin,

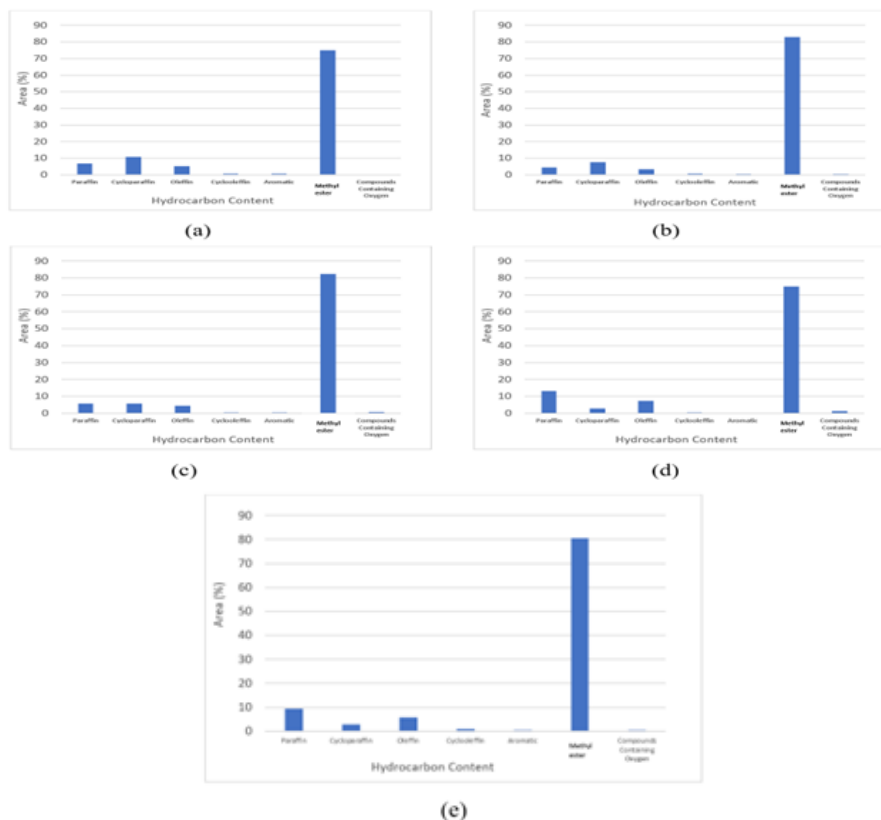


FIGURE 6 Effect of catalyst mass on hydrocarbon liquid product composition (operation condition: 200 mL of FAME, FAME density of 0.89 g/cm³ and 250°C in the catalytic cracking process).

aromatic, and methyl ester compounds. While the other group, paraffin, and olefins, there was an increase in the mass of the catalyst of 0.2 g. Compounds with oxygen content occur a significant increase exceeding percentage area at a mass of 0.1 g, which is 0.73% area.

Compared to a catalyst mass of 0.4 g, there was a decrease in cycloparaffin, cycloolefin, aromatic, and carboxylic acid compounds. Meanwhile, paraffin, olefins, and compounds with oxygen content again increased in the amount marked by an increase to 13.15, 7.24, and 1.23% area. In using a catalyst mass of 0.5 g, there was a decrease in cycloparaffin again, which was 2.72% area. Paraffin, olefins, and methyl ester have an increase when compared to a catalyst mass of 0.4 g. This phenomenon indicates that the performance of hH-ZSM-5 increases with increasing catalyst mass used towards the formation of hydrocarbon compounds in the gasoline range through the breaking of long-chain hydrocarbons into short-chain hydrocarbons. The acidity of the catalyst plays an important role in breaking the C-C, C=C, C=O, and C-O bonds in FAME.

The occurrence of an increase in the mass of paraffin when there is an increase in the mass of the hierarchical H-ZSM-5 catalyst is used in accordance with the study by Jimmy et al.^[17]. This is because the more hierarchical catalysts HZSM-5 used, the larger the active surface that interacts with the reactants. According to Jimmy et al.^[17] and Marlinda et al.^[18], the more active the surface area of the hierarchical HZSM-5 catalyst, the better the performance of the catalyst used.

4.3.2 | Effect of Catalyst Mass on Product Yield

Using different catalyst masses to FAME affects the yield of the liquid product. The number of catalysts mass-added plays a role in improving the quality of the products produced. The addition of catalysts will increase the chances of reactants to react more to produce a product. The effect of the catalyst mass on the yield of gasoline, gasoil, or kerosene on liquid products resulting from the cracking of FAME Cerbera manghas oil can be seen in Fig. 7 .

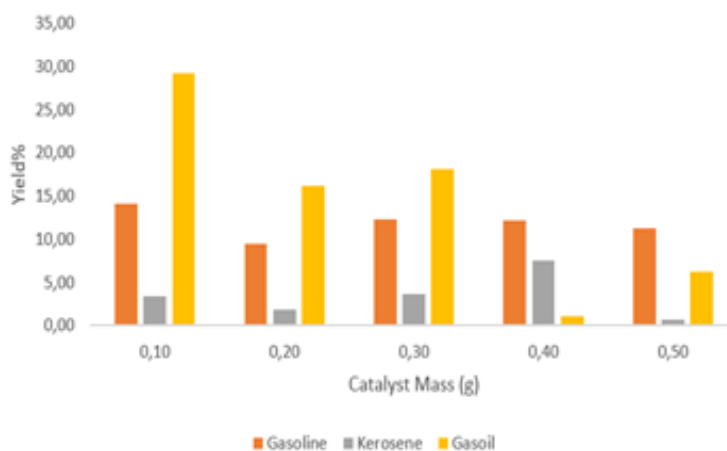


FIGURE 7 The yield of gasoline/kerosene/gasoil on hydrocarbon liquid product.

This can be seen in the hierarchical HZSM-5 catalyst mass of 0.1, 0.2, and 0.3 g, producing gasoil as the largest content. The use of catalyst mass of 0.4 and 0.5 g shows gasoline as the largest content in the liquid product produced. Table 1 shows the yield of each component produced in the liquid product resulting from the FAME cracking of Cerbera manghas oil.

TABLE 2 The yield of hydrocarbon content in liquid product from catalytic cracking of FAME Cerbera manghas Oil.

Catalyst Mass (g)	Hydrocarbon Content (% area)*					
	Light Gas C1-C4	Nafta C5-C7	Gasoline C7-C12	Kerosene C13-C15	Gasoil C16-C17	Heavy Oil C20-C70
0.1	0.03%	2.77%	14.08%	3.35%	29.14%	0.11%
0.2	0.21%	2.29%	9.51%	1.86%	16.18%	0.48%
0.3	0.02%	1.91%	12.30%	3.70%	18.07%	1.17%
0.4	0.19%	0.87%	12.22%	7.56%	1.03%	9.54%
0.5	0.21%	0.24%	11.21%	0.64%	6.28%	5.00%

*GC-MS analysis

Table 2 shows the results of the GC-MS analysis of liquid products grouped into several groups of hydrocarbon compounds based on the number of C atoms, namely C1-C4 (light gas), C5-C7 (naphtha), C7-C12 (gasoline), C13-C15 (paraffin), C16-C17 (diesel) and C20-C70 (fuel oil). It shows that overall the composition of the hydrocarbons in the liquid product hydrocarbons as a result of cracking changes with each addition of catalyst mass. The composition of the largest fraction of hydrocarbon compounds using a catalyst mass of 0.1, 0.2, and 0.3 g was obtained from the diesel fraction (C16-C17). While using 0.4 and 0.5 g of catalyst, it was found that the largest fraction was gasoline (C7-C12). If the target liquid product is in the gasoline fraction, then a catalyst mass of 0.5 g can be used and tried again to use catalyst mass > 0.5 g to get the optimal gasoline fraction. It should be noted that using a mass of 0.5 g, the fractions of gasoline and diesel with the highest amount of messages were obtained, namely 14.08% area for the gasoline fraction and 29.14% area for the diesel fraction.

5 | CONCLUSION

The effect of the variation in the ratio of hH-ZSM-5 catalyst mass to the composition of the formed hydrocarbons can increase the paraffin content and hydrocarbon compounds in the gasoline range (C7-C12) at a low cracking temperature of 250°C. Even so, the high methyl ester content was still found for each variation of the catalyst mass. Therefore, a new strategy is needed to improve the performance of this catalyst by adding transition metals to the catalyst pores and using hydrogen gas for the hydrogenation process.

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CREDIT

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