Aceh Int. J. Sci. Technol., 12(3) : 327-336
December 2023
doi: 10.13170/aijst.12.3.33824

Aceh International Journal of Science and Technology
ISSN: p-2088-9860; e-2503-2398
Journal homepage: http://jurnal.usk.ac.id/aijst

Inorganic Solid Catalyst Derived from Fishbone Waste (*Katsuwonus pelamis*) for Transesterification of Coconut Oil into Biodiesel

Siti Maisarah[1], Minnati Rana Maisarah[2], Dini Satrina[2], Surya Lubis[2], Rara Mitaphonna[3], Febriani[2], Desi Novita[2], Muliadi Ramli [1,2*]

[1] Graduate School of Chemistry, Faculty of Mathematics and Natural Sciences, Graduate of Universitas Syiah Kuala, 23111, Indonesia;
[2]Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Syiah Kuala, 23111, Indonesia;
[3] Doctoral Program of Mathematics and Applied Sciences, Universitas Syiah Kuala, 23111, Indonesia.
*Corresponding author: muliadiramli@unsyiah.ac.id

Received : September 7, 2023
Received in revised from: September 19, 2023
Accepted : September 21, 2023
Online : October 31, 2023

Abstract – This research has utilized fish bone wastes of tuna (*Katsuwonus pelamis*) collected at the Lampulo fish market in Banda Aceh. Inorganic oxides have been derived from those fish bone wastes through the decomposition method at high temperatures, namely the calcination process in air atmosphere at 900^{O}C for 4 hours. The physicochemical properties of obtained inorganic oxides were characterized using XRD and SEM-EDS techniques. The characterization results indicated that the composition of the calcined fish bone contained hydroxyapatite, CaCO3, and CaO, in which the hydroxyapatite phase has been the major component. Furthermore, the calcination process positively impacts improving the physical morphology and crystalline phase of inorganic oxides. Finally, those obtained inorganic catalysts based on fish bone waste have been applied for transesterifying Coconut oil with methanol, resulting in three main compounds: trimethyl borate, methyl laurate, and methyl octanoate.

Keywords: Fishbone; *Katsuwonus pelamis*; heterogeneous catalyst; transesterification; coconut oil.

Introduction

The annual increases in fisheries industry production are also accompanied by massive increases in consumer demand, with processed fish products' demand growth rate achieving 12% per year. As fish demand and consumption grow, the volume of bone waste will also increase significantly (Nurilmala, 2014). Fishbone wastes are generated from the production process, from industry and household wastes. Due to its low economic worth, the presence of fish bone waste in the environment has been no economic gains. The presence of fish bone waste might be harmful to the environment, especially to human health, depending on its concentration and quantity.

Furthermore, because fishbone waste has no economic value, neither the government nor the community has paid much attention, which means that fishbone waste has not been properly handled or utilized. In order to prevent it from polluting the environment, there is a need for a concerted effort to utilize fish bone wastes. One of most fish bone wastes comes from tuna fish (*Katsuwonus pelamis*). This is because tuna fish is one of the most popular types of fish, easy to find, and affordable. Chemically, tuna bones are rich in minerals such as calcium, phosphorus, magnesium, iron, zinc, and others (Toppe *et al*., 2007). However, tuna meat is the only part of the fish that can be consumed, which leaves the fish bones discarded and mainly becoming waste with a huge potential to become environmental pollutants. To overcome the problem, there is a growing effort in research to further utilize bone waste as a catalyst in the transesterification reaction with used cooking oil as the feedstock, as reported by Wijianto (2016).

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A catalyst is a substance that speeds up a reaction by lowering the activation energy of the reaction so that the energy needed for the reaction to take place can be as minimal as possible. The catalyst that has been used will be recovered at the end of the reaction. However, utilizing a homogeneous catalyst has many disadvantages, as the catalyst and reactants are in the same phase and are considered difficult to separate. Whereas heterogeneous catalysts have a different phase from the reactants, are easy to recover, and can be used in other reactions. Concerning fishbone containing several metallic elements, it could be understood that heterogeneous catalysts can be derived from fishbone wastes through thermal decomposition at a specific temperature. This method, well known as the calcination method, aims to break down complex compounds into simple compounds and remove the content of carbon dioxide compounds, leading to metallic oxides forming.

Biodiesel is an alternative fuel for diesel engines synthesized from vegetable oil. Biodiesel consists of methyl esters and fatty acids produced from the reaction between vegetable oil and methanol or ethanol. Some vegetable oils often used as feedstock for biodiesel synthesis include coconut, palm, soybean, peanut, castor seed, and others. The vegetable oil used as feedstock for biodiesel synthesis in this study is coconut oil because it is easy to obtain and has low levels of free fatty acids. Utilizing renewable resources, such as coconut oil, to make biodiesel has another advantage for the environment, as it emits less carbon dioxide, is nontoxic, and does not contain sulfur or other pollutants.

Several studies have successfully produced biodiesel through transesterification reactions using heterogeneous catalysts prepared from raw natural resources or waste. For this purpose, Viriya-empikul *et al.* (2012) conducted research on the synthesis of biodiesel through the transesterification reaction of palm oil with methanol using a catalyst from the calcination of eggshell waste at a calcination temperature of 800^{O}C resulting in a biodiesel yield of 94%. Jazie *et al.* (2013) have reported on synthesizing biodiesel through the transesterification reaction of *Jatropha* oil with methanol using a catalyst from calcined animal bone wastes with an optimum calcination temperature of 900^{O}C, which has led to a biodiesel yield of 96%. Another researcher, Sirisomboonchai *et al.* (2015), synthesized biodiesel through the transesterification reaction of spent cooking oil with methanol catalyzed by Scallop shells (*Patinopecten yessoensis*), obtaining a biodiesel yield of 86%.

The availability and abundance of tuna fish bone wastes (*Katsuwonus pelamis*) in Banda Aceh has prompted researchers to utilize this waste for sound alternative energy; this study has focused on utilizing the fish bone wastes as raw material for preparing metallic oxide catalyst and its application in transesterification reaction of coconut oil into biodiesel.

Materials and Methods

Equipment and Materials

The equipment used in this study were a mortar and pestle, furnace, reflux kit, three-neck flask, thermometer, beaker, bath, magnetic stirrer, porcelain, desiccator, separating funnel, Whatman 42 filter paper, funnel, Erlenmeyer 250 mL, scales analytical, beaker, 100 mesh sieve, pycnometer, Ostwat viscometer, burette, stative, sample vials, XRD, SEM EDX, GC-MS instruments. The materials used in this study were local fish bone waste (*Katsuwonus pelamis*), coconut oil, methanol, ethanol, and aquadest.

Sample preparation

The raw material used in this experiment was fish bone wastes (Katsuwonus pelamis) obtained from the Lampulo fish local Banda Aceh market. The experimental process begins with washing the fish bones after the boiling process. The fishbone was then cut into small pieces to remove the bone materials. Then, it was dried under sunlight exposing to remove the water content. Furthermore, the bones were ground into a fine powder and sieved with a 100-mesh sieve. The powder was then calcined at 900^{O}C for 4 hours, according to the previous method reported by Wijianto (2016). After calcination, the calcined sample is stored in a desiccator to keep it dry. Then, several analyses were carried out to characterize the physical and chemical properties of the fish bone samples, such as the XRD technique used to determine the phases present in the sample. The XRD spectrum (Figure 2) of a bone sample was obtained by Cu irradiation at a voltage of 40.0 kV, a current of 30.0 mA, a scanning speed of 10.0000 ^{O}/s, and a scanning range of 10.000–80.000 (deg). The diffraction peak indices were compared using the Joint Committee on Powder Diffraction. The physical morphology and chemical composition of bone samples were characterized using the SEM-EDS technique. The obtained inorganic catalyst was further used in the transesterification reaction to synthesize biodiesel from coconut oil. For chemical analysis of the transesterification product, the obtained biodiesel in the experiment was separated, purified, and

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characterized concerning its density, viscosity, and acid number. Finally, CG-MS analysis was used to determine the chemical composition of the obtained biodiesel.

Results

The calcination process positively impacted removing CO_2 and breaking down complicated chemicals in fish bone material, resulting in simple components such as metallic oxides. Figure 1 shows that both fresh (*Katsuwonus pelamis*) (a) and the calcined fishbone catalysts were obtained through a thermal decomposition process at 900^{O} C for 4 h (b).

Figure 1. (a) Fresh fish bone (*Katsuwonus pelamis*) particles; (b) calcined fish bone particles (900^{O}C,4 h).

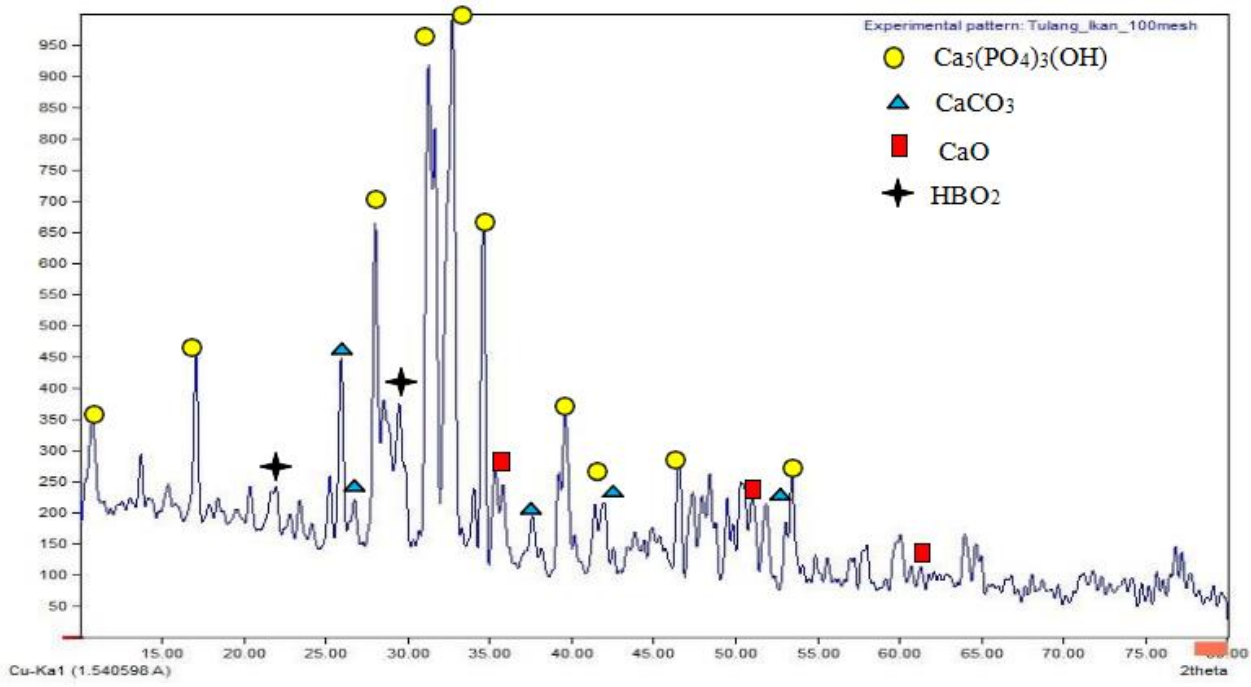

Figure 2. XRD pattern of calcined tuna bone particles (*Katsuwonus pelamis)* at 900^{O}C.

It can be understood that before undergoing the calcination process (Figure 1a), those particles have a fine texture with a light mass and a soft brown color. This indicates that the fishbone particles still contain both

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organic and inorganic compounds. After taking a calcination process, the bone particles (Fig. 1b) changed into a denser texture and a color change from brown to bright white. The color change is due to the decomposition of inorganic compounds into their oxide form. The more oxides that form, the brighter the color of the calcined particles. The resulting bright white color in the particle indicates that the main content of the material has been calcium oxides (Sugiyarto, 2003).

XRD techniques were used to investigate how the calcination process affects the crystalline phase in the fish material. Figure 2. shows the XRD pattern of calcined tuna bone particles after high-temperature treatment at 900 ^{O} C. Based on a matching process between the XRD pattern obtained in this experiment and the standard diffraction of compounds in the JCPDS system, the obtained catalyst was discovered to be composed of hydroxyapatite, $CaCO_3$, CaO, and metaboric acid. Based on Figure 2, it can be stated that the X-ray pattern of the obtained catalyst with a calcination temperature of 900^{O}C exhibits high peak intensities at 2θ of 32.5886°, 31.4205°, and 34.5866° indicating the formation of hydroxyapatite compounds. The obtained catalyst, however, still contained $CaCO_3$ compounds, as detected at 2θ of 25.9160°, 26.6800°, 37.5700°, and 41.8800°.

$CaCO_3$ peaks might be caused by the incomplete decomposition of $CaCO_3$ into CaO and CO_2. The CaO appears with low intensities at 2θ of 35.3400°, 51.8183° and 61.2700°. Meanwhile, the peaks observed at 2θ of 21.7900° and 29.4600° were linked to the formation of metaboric acid. Hydroxyapatite compounds have the highest intensity compared to other substances, which can be observed from the high peaks in the X-ray diffractogram. The high intensity of the hydroxyapatite compound indicates that it has a high level of crystallization. The level of crystallization can also be seen from the existence of sharp peaks, while the widened peak indicates that the phase of the compound is amorphous. In general, the increase in crystal formation co-occurs with the increased heating time and temperature. This experiment's XRD characterizations are similar to the previous study reported by Ratna and Jagannatham (2016), which produced hydroxyapatite from fish bones at different temperatures. The diffractogram of the XRD pattern in Figure 2 also indicated that the calcium compounds and their derivatives, including hydroxyapatite, $CaCO_3$, and CaO, have been successfully formed.

SEM analysis was used to determine the morphology of the fishbone particles without and after the calcination process at 900^{O}C. The result of SEM analysis is shown in Figure 3.

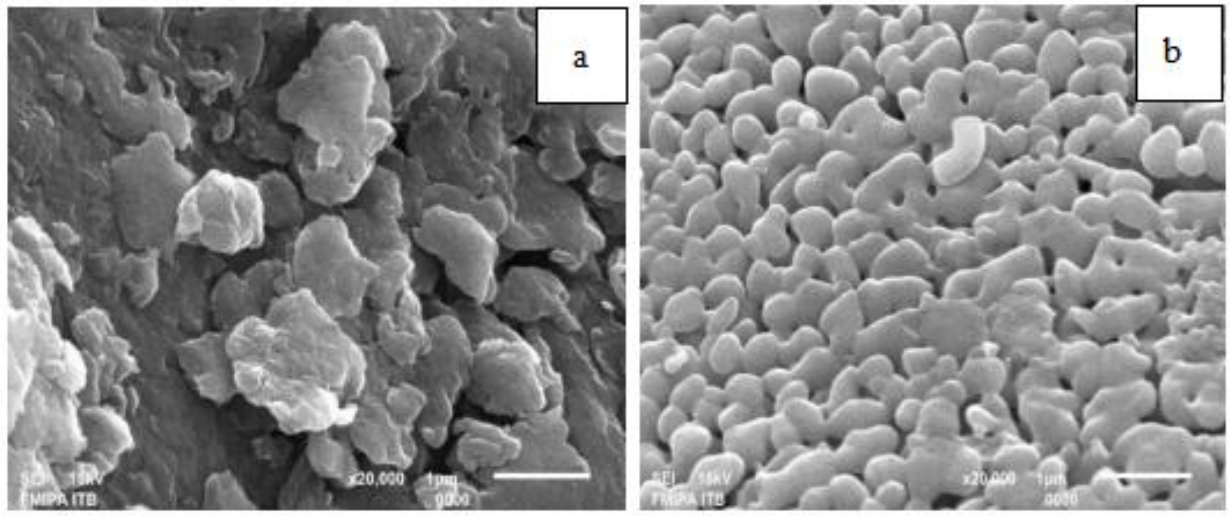

Figure 3. SEM micrographs of (a) fresh fishbone particles (without calcination) and (b) calcined fishbone particles (900 C, 4 h).

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The calcination process at 900^{O}C was chosen because it could convert fish bone particles into oxide forms by causing H_2O and CO_2 to evaporate, as well as increasing the surface area and the activity of the catalyst. Figure 3(a) shows that those particles still have an amorphous surface due to organic compounds, and coarse aggregates form in the particles. In comparison, after taking calcination 900^{O}C, 4 h, as shown in 3 (b), it can be seen that those fish bone particles are converted into their oxide form, which can function as a catalyst. Based on the SEM micrograph, those calcined particles are homogeneous and reached the micrometer scale of particle size. The shape resembled a rod, implying the surface distribution would be even greater. Crystals have formed on the surface of the calcined catalysts and act as active sites, which is beneficial to speed up the reaction time during transesterification.

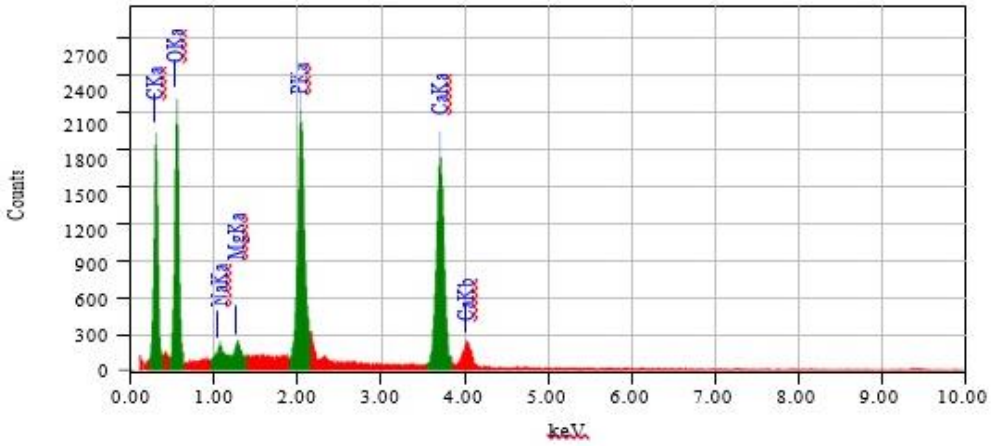

Figure 4. EDS spectra of fresh fish bone particles (no calcination treatment).

EDS analysis was also conducted to determine the element's composition in the obtained catalyst. Fig 4. shows the EDS spectra of fresh fish bone particles (no calcination). The EDS result proved that fresh fish bone particles mostly contained high carbon elements (46.87% wt/wt) while the percentage of inorganic material contained only a tiny amount.

Table 1. Elemental composition (EDS analysis) of fresh fish bone particles (no calcination treatment).

Element	(keV)	Mass (%)	Atom (%)
C	0.277	46.87	58.97
O	0.525	35.33	33.37
Na	1.041	0.33	0.22
Mg	1.253	0.33	0.21
P	2.013	7.02	3.42
Ca	3.690	10.11	3.81
Total		100.00	100.00

Calcination is a simple method to decompose solid material under a high-temperature system. In this experiment, the calcination of fresh bone particles at 900 C for 4 h changed the chemical composition of fish bone biomass, leading to metallic substances such as Ca, P, Mg, and Na. There could be that the calcination process has had a positive impact on enhancing the mass element of Ca, particularly from 10.11% to 20.11% (wt/wt). Elemental P also increased from 7.02% to 14.86%. The Mg element increased from 0.33% to 1.19%, and the Na element increased from 0.33% to 0.90%.

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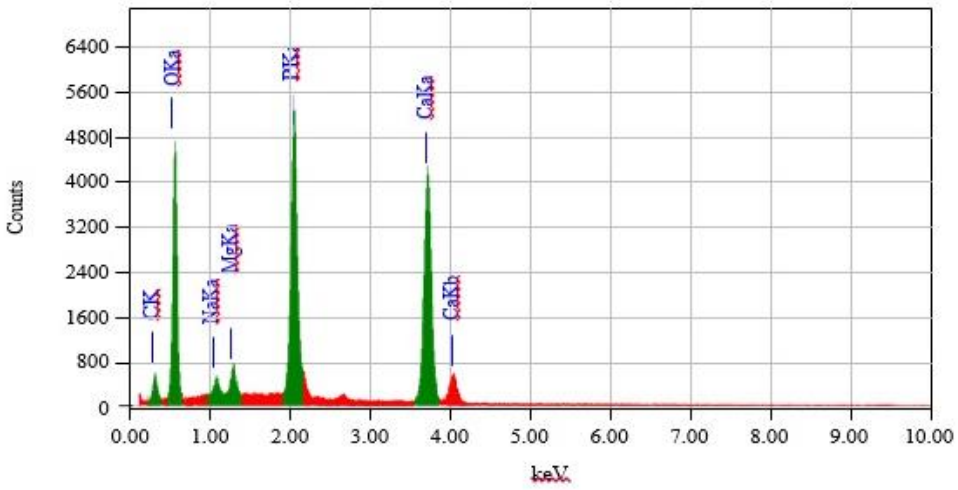

Figure 5. EDS spectra of calcined fish bone particles (calcined fish bone particles)

This is because the bond, on the contrary, after the calcination process, the mass of organic materials such as carbon elements drops dramatically from 46.87% to 11.27%. Between carbon and oxygen, it weakens during the heating process at high temperatures, allowing hydrogen gas to quickly reduce oxygen and escape from carbon, causing the mass of carbon to decrease.

Table 2. The EDS analysis of calcined fish bone particles (calcined fish particles)

Element	(keV)	Massa (%)	Atom (%)
C K	0.277	11.27	17.92
O K	0.525	51.64	61.64
Na K	1.041	0.90	0.75
Mg K	1.253	1.19	0.93
P K	2.013	14.86	9.16
Ca K	3.690	20.14	9.60
Total		100.00	100.00

Discussion

Application of obtained catalyst for transesterification of coconut oil into biodiesel

The biodiesel synthesis process began with the analysis of coconut oil as the feedstock in this experiment. The coconut oil used was obtained from the Commercial Store in Banda Aceh. The quality of coconut oil was determined through various parameters, such as acid number, density, and viscosity, as shown in Table 3.

Table 3. Properties of coconut oil used raw biomass in this experiment.

No.	Parameter	Method	Result
1.	Color	-	Transparent
2.	Acid Number	ASTM-D6640	0.06 mg KOH/g
3.	Viscosity	ASTM D-445	10.22 mm^2/s
4.	Density	ASTM D-1298	0.90 Kg/m^3

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Based on the information in Table 3, coconut oil has a low free fatty acid content of 0.06 mg KOH/g, a density of 0.90 kg/m3, and a viscosity of 10.22 mm2/s. The low content of free fatty acids in the oil sample gave an advantage because coconut oil could be directly used for biodiesel synthesis. The high free fatty acids content in raw oil samples is highly directed to side products, namely saponification reaction will take place in the system. Another factor that can cause saponification reactions is the high water content in the reactants, so methanol has a relatively high purity of 99%. Was used in this experiment.

The transesterification reaction was demonstrated by reacting coconut oil with methanol employing different inorganic catalysts, namely fresh fishbone catalysts and calcined fishbone catalysts, which were prepared previously in this experiment. The catalysis experiment was conducted due to scientific information regarding the catalyst's activity. Theoretically, the transesterification product yielded a layers system after being transferred into a separatory funnel, in which the top layer was indicated as methyl ester, and the bottom layer was glycerol. The glycerol was removed from the system in a clear-colored solution called crude biodiesel. The biodiesel was taken for further purification using warm water at 50 °C. The expected biodiesel of methyl ester was analyzed using GC-MS.

The obtained biodiesel presulted from coconut oil catalyzed by inorganic catalyst derived from fish bone has two-layer heterogeneous mixture. In contrast, the top layer was discovered to be methanol rather than methyl esters. This was proven by measuring the density using a pycnometer, which noticed that the density of the solution was similar to the density of methanol, which was 0.792 g/cm3.

Figure 6. Obtained Biodiesel

It may be assumed that the transesterification product catalyzed by calcined fish bone particles is still not accorded to SNI. Meanwhile, based on the Indonesian National Standard (SNI) biodiesel quality, the density value should be in the range of 0.850 - 0.890 g/cm3.

Finally, the technique analysis was carried out to get scientific information regarding the composition of the obtained biodiesel. The GC-MS analysis proved that the obtained biodiesel was contained the fatty acid methyl ester based on comparing the retention time and mass spectra, which directly observed in the experiment with library data of the mass spectra of compounds—figure 7 shows the GC chromatogram of obtained biodiesel.

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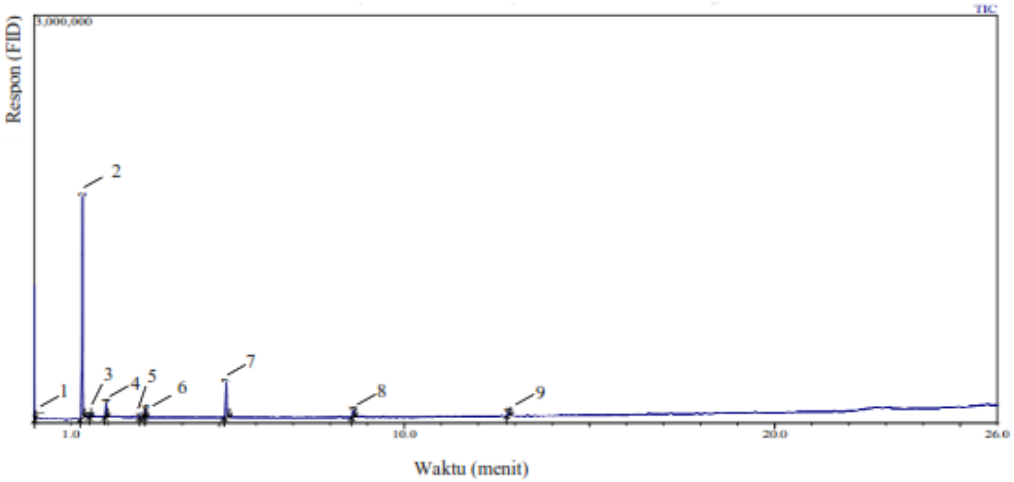

Figure 7. GC Chromatogram of obtained biodiesel in this experiment

The GC analysis noticed that nine peaks correspond to methyl esters compounds, which they consist of Desin-2-ol with a retention time of 0.019 minutes, trimethyl borate with a retention time of 73.12 minutes, methyl caproate with a retention time of 0.37 minutes, methyl octanoate with a retention time of 3.71 minutes, dodecyl ether with a retention time of 0.46, methyl caprate with a retention time of 2.34 minutes, methyl laurate with a retention time of 14.33 minutes, methyl myristate with a retention time of 3.21 minutes, and methyl palmitate with a retention time of 1.89 minutes. The percentage of chemical components contained in biodiesel that have been analyzed using MS is shown in Table 4.

Table 4. The GC data and chemical composition of obtained biodiesel based on MS analysis

Peak No.	Retention Time	Relative Area (%)	Compound
1	0.019	0.57	Desin-2-ol
2	1.314	73.12	Trimetil borat
3	1.520	0.37	Metil caproate
4	1.948	3.71	Metil octanoate
5	2.870	0.46	Diisodesil eter
6	3.006	2.34	Metil kaprat
7	5.187	14.33	Metil laurat
8	8.625	3.21	Metil myristate
9	12.814	1.89	Metil palmitate

Table 4 shows that the biodiesel product is dominated by trimethyl borate at 73.12%. Trimethyl borate is a chemical compound with the formula $B(OCH_3)_3$. Trimethyl borate can be formed through condensation of boric acid and alcohol. Boric acid can also react with glycerol while reacting with glycerol, the acidity of boric acid increases. The increased acidity causes the reactions between boric acid and glyceride and boric acid and glycerol to become faster, producing more products. As a result, the high content of trimethyl borate may be due to the reaction of metaboric acid and glycerol. Based on the XRD data (Figure 2), the calcined fish bone (catalyst) still contains metaboric acid, so the compound reacts with alcohol to form trimethyl borate. Apart from trimethyl borate, the other most significant component in the biodiesel is methyl ester, with a total percentage of 25.85% (wt/wt). It was noticed referred to the GC-MS analysis whereas the major compounds were methyl caproate (0.37%), methyl octanoate (3.71%), methyl caprate (2.34%), methyl laurate (14.33%), methyl meristate (3.21%), and methyl palmitate (1.89%). Among the methyl esters identified, methyl laurate has the highest percentage, 14.33%. The quality of biodiesel resulting from the transesterification of coconut oil catalyzed by fish bone catalysts follows previous research reported by Jeyarani *et al.* (2009), who confirmed that methyl laurate has the

highest content in biodiesel compounds that are produced from coconut oil, which reaches up to 47.3% (wt/wt)—considering another study reported by Kouzu *et al.*, (2007) using CaO, Ca $(OH)_2$, and $CaCO_3$ catalysts to transesterification soybean oil into methyl esters. The reaction was carried out using a refluxing system with methanol producing methyl ester yields of 93% while using CaO catalyst, 12% for Ca $(OH)_2$ catalyst, and no methyl ester product was formed while $CaCO_3$ catalyst was used. The shallow methyl ester content is in this study because there are still many CaCO3 compounds in the calcined fishbone catalyst (they have not been completely converted into CaO. $CaCO_3$), which is poorly active as a catalyst. The properties of biodiesel produced through the transesterification reaction with calcined fish bone catalyst are determined using various parameters such as acid number, density, and viscosity, then compared using the ASTM D-1298 and ASTM standard methods, as shown in Table 5.

Table 5. Properties of obtained biodiesel using calcined fish bone catalyst

No.	Method	Parameter	Standard	Product (%)
1	ASTM-D6640	Acid number	Max.0.8 mg KOH/g	0.0331 mg KOH/g
2	ASTM D-1298	Density	0.85-0.90 g/cm^3	0.8248 g/cm^3
3	ASTM D445	Viscosity	2.3-6.0 mm^2/s	2.46 mm^2/s

The Indonesian National Standard (SNI-04-7182-2006) for the kinematic viscosity and acid number of biodiesel at 40^{o}C should be no higher than 2.3 – 6.0 mm^2/s and 0,8 mg KOH/g, respectively. The viscosity of the obtained biodiesel was found to be 2.46 mm2/s, whereas the acid number was 0.0331 mg KOH/g, indicating that the synthesized biodiesel complied with the quality standards. However, there is a significant difference between the density of the obtained biodiesel and the standards. According to the Indonesian National Standard (SNI-04-7182-2006), the density of biodiesel measured at 40^{o}C should be in the range of 850–890 kg/m^3. Unfortunately, the density of the obtained biodiesel was 0.8248 g/cm3, which is lower than the SNI value. This might be due to the biodiesel only consisting of a low concentration of methyl ester, which led to low-density value, and it is still unable to comply with the SNI standard.

Conclusion

Fish bones (*Katsuwonus pelamis*) catalyst mostly contain hydroxyapatite compounds, which are the dominant substance compared to $CaCO_3$ and CaO crystalline phases. Concerning GC-MS analysis, the inorganic catalyst prepared from fish bone waste has successfully transistorized coconut oil lead to biodiesel, which contains design-2-ol, trimethyl borate, methyl caproate, methyl octanoate, dodecyl ether, methyl caprate, methyl laurate, methyl meristate, and methyl palmitate. The acid number and viscosity of biodiesel resulting in this experiment follow the Indonesian National Standard SNI-04-7182-2006 and ASTM provisions.

Acknowledgment

The author would like to highly thank the Indonesian Ministry of Research, Technology, Higher Education and The University of Syiah Kuala, Banda Aceh, for supporting this research work financially.

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