Preparation of the NiCl₂ intercalated on the bentonite as a catalyst in the cracking process of biodiesel

I W Sutapa*, S Amalia and Rosmawaty

Department of Chemistry, Faculty Mathematics and Natural Science, University of Pattimura Jl. Ir. Putuhena Poka 97233, Ambon-Maluku- Indonesia

Email: wayansutapa@fmipa.unpatti.ac.id

Abstract. Biogasoline is a benign energy product that can be produced through the process of breaking down biodiesel. The purpose of this study is to produce biogasoline from cooking oil waste through the cracking process using NiCl₂ intercalated as a catalyst. Initially, waste from cooking oil was converted into biodiesel through a process of esterification and transesterification. After biodiesel with carbon chain lengths of 16-20 was acquired, the cracking process to produce biogasoline that had carbon chain lengths of 4-12 with the aid of a catalyst was conducted. The bentonite intercalated NiCl₂ catalyst was calcined at a temperature of 600°C for 2 hours, then characterized by XRD and SEM. The results of the diffractogram with XRD showed a peak around 2θ: 44.67° which was the peak of the Ni crystalline. The cracking process of biodiesel was carried out at a temperature of 450°C and 500°C then characterized using GC-MSS. The chromatograms generated at a temperature of 450°C showed the presence of three compounds belonging to the faction of biogasoline they were methyl 6-hydroxyhexanoate (C₁₀H₁₄O₃), 4-hydroxymethyl-1,3-dioxolane (C₇H₈O₃), and methyl nonylate (C₁₀H₂₀O₂).

1. Introduction

A report from the Congressional Research Services (CRS) in 2003 to the Energy Commission in the United States Congress, stated that the level of fossil fuel use continues as it is today without increasing production efficiency, the discovery of new reserves, and the transition to alternative renewable energy sources. If this persists, then it is estimated that reserves of fossil fuel energy sources, especially petroleum, will only be sufficient for the next 30-50 years [1–5]. As a result of the increasing use of petroleum for fuel consumption and the depletion of petroleum reserves, it becomes a trigger to look for alternative and renewable energy. Biodiesel is one of the solutions to these problems [6,7]. Alternative energy sources such as hydrogen and biodiesel have caught the attention of researchers around the world. Biodiesel is an alternative fuel produced from renewable sources [8–11].

To date, the biodiesel produced from edible oil has a higher price compared to non-edible oil because it competes with consumption by the people. Therefore, now many biodiesel-based non-edible oils are being developed. Besides, biodiesel raw materials produced from non-edible oil are usually cheaper than conventional diesel fuel from petroleum. One source of non-edible oil is wasted cooking oil. In addition to increasing the added value of used cooking oil, the use of used cooking oil as a basis for making biodiesel is an excellent alternative way to handle waste [12,13].

Biodiesel production is generally carried out through a transesterifications reaction using a homogeneous catalyst (acid or base) [14–16]. Homogeneous catalysts are catalysts that have the same
phase as reactants and products and have the advantage; they do not require high temperatures and pressures in the reaction. Homogeneous catalysts that are widely used in transesterification reactions are potassium hydroxide (KOH) and sodium hydroxide (NaOH) catalysts [3,17,18]. Biodiesel produced in the transesterification reaction of vegetable oils and animal fats with homogeneous catalysts generally has a hydrocarbon chain consisting of 16-20 carbon atoms [19,20].

The long biodiesel carbon chain can be converted into gasoline (gasoline) with a shorter chain length, namely C₄-C₁₂, through the cracking process. In petroleum and chemical geology, cracking is the process by which complex organic molecules such as hydrocarbons are broken down into simple units by cutting carbon-carbon bonds in the precursors. Simply put, cracking is the process of breaking down long-chain hydrocarbons into shorter ones. The result of cracking from biodiesel called biogasoline has several advantages over gasoline derived from fossils. It is environmentally friendly and can be reproduced in a short time. One method of cracking is breaking using a catalyst [21,22].

Several researchers have carried out the cracking reaction of cooking oil waste using zeolite/nickel catalysts. The natural zeolite was modified through the process: demineralization, dealumination, and activation, then followed by the impregnation of natural zeolite with NiCl₂. The determination of the optimal ratio is based on wasted cooking oil cracking products with a catalyst/sample ratio. GC-MS analysis showed C₄-C₁₅ fraction hydrocarbon compounds dominated the cracking product composition of each catalyst/sample. The optimal conversion was confirmed by catalyst/sample cracking product 10:40 w/w with the composition of pentadecane compound (C₁₅H₃₂) of 44.60% and nonane compound (C₉H₁₈) of 4.47% [23–27].

Most of the content of bentonite is montmorillonite, with a structure that has a similarity to zeolite with great potential to be used as a catalyst for cracking. The bentonite is stable at high temperatures if the chemical elements contained in bentonite must be minimized by inserting ions, molecules, or compounds into their structures called intercalation. Ions, molecules, or compounds to be intercalated should have a commercial value that is cheap, easy to obtain, and of good quality. In our research goal, the cracking catalyst produced can be used on a large scale. Finally, the biogasoline that is produced can be consumed by all levels of society. Based on the background, this study investigated the catalyst NiCl₂ intercalated in bentonite and its applications as a catalyst in the biodiesel cracking process from cooking oil waste.

2. Materials and methods

2.1 Materials

The materials used in this study were cooking oil waste, methanol (Merck), ethanol (Merck), H₂SO₄ (Merck), KOH (Merck), anhydrous Na₂SO₄, commercial bentonite, NiCl₂, CTAB (Cetyl Trimethyl Ammonium Bromide) (Merck), phenolphthalein indicators, Whatman filter-40, and distilled water.

2.2 Bentonite preparation

A total of 250 g of bentonite was sieved using a 200 mesh sieve. Subsequently, bentonite was dispersed into distilled water and stirred using a magnetic stirrer for 3 hours. The suspension obtained was filtered using filter paper. The filtering results were dried in an oven at 110°C for 2 hours. The dried sample was crushed and sieved with a size of 200 mesh and then analyzed with XRD and SEM.

2.3 Waste cooking oil preparation

A total of 500 mL of used cooking oil was heated at 120 °C to evaporate water in used cooking oil. After heating, the oil was filtered with Whatman 40 filter paper to separate larger sized solid impurities.

2.4 Intercalation process

Fifty five gram of bentonite was dispersed into 1250 mL of distilled water and stirred for 3 hours to make a bentonite suspension. After 3 hours of bentonite suspension, 10 g of quaternary salt of Cetyl
Trimethyl Ammonium Bromide (CTAB) was added and stirred for 24 hours. Next, 11 g of NiCl₂ catalyst was added to the suspension, and the stirring process was continued for another 24 hours. Then the suspension was filtered, and the sediment was dried in an oven at 110°C. After drying, the modified bentonite was crushed and sieved to 200 mesh size and then calcined at 600°C for 2 hours to obtain intercalated bentonite NiCl₂. XRD and SEM then analyzed the intercalated bentonite.

2.5 Esterification process

A total of 50 gram of sample was put into a three-neck flask, esterified with free fatty acids with methanol (comparison of oil with methanol 1: 3), added with 1 M H₂SO₄ catalyst as much as 1.25% of the total weight of oil and methanol, then refluxed at 60-65°C for 2 hours. The reaction mixture was cooled and formed in two layers, methanol and methyl ester (top layer), triglycerides (bottom layer), and then separated using a separating funnel.

2.6 Transesterification process

The separated triglyceride was then transesterified with methanol (oil and methanol ratio 1: 6), and a 1% KOH catalyst was added by the weight of the mixture of oil and methanol, refluxed at 60°C for 2 hours. The reaction mixture was cooled down, and two layers formed, methyl ester (biodiesel) in the upper layer and glycerol in the lower layer. Then the mixture was separated by a separating funnel, and then the methyl ester was evaporated. The methyl ester layer was washed with distilled water repeatedly using a separating funnel until the water layer was transparent. The final step was adding anhydrous Na₂SO₄ to bind the residual water then filtered with Whatman 40 filter paper. The resulting biodiesel product was then characterized using GC-MS.

2.7 Cracking process

Cracking of biodiesel from used cooking oil was carried out using a cracking reactor. A total of 50 mL of sample was inserted into the reactor, followed by the inclusion of 3 g of the catalyst NiCl₂ intercalated into the upper furnace. The cracking process was carried out with variations in the temperature in the lower furnace of 450°C and 500°C, and a constant temperature in the upper furnace of 350°C. Each heat was carried out for 60 minutes. Then the product produced was analyzed using GC-MS.

3. Results and discussion

3.1 Bentonite preparation

Bentonite preparation began with the washing process of bentonite. This process was carried out by dispersing bentonite into distilled water. Bentonite was sieved using a 200 mesh sieve. Sifted bentonite was then dispersed into distilled water and stirred using a magnetic stirrer for 3 hours (figure 1). This dispersing process aimed to remove impurities and was expected to increase the surface area of bentonite. The resulting suspension was filtered using filter paper. The filtering results were dried in an oven at 110°C for 2 hours to remove water molecules found in the bentonite pore. The dried sample was crushed and sieved with a 200 mesh sieve. This process aimed to reduce the size of the bentonite so that it increased the surface area of the bentonite. This process produced clean bentonite that was ready to use. Furthermore, it was analyzed using XRD and SEM on clean (pure) bentonite samples.

3.2 Intercalation process

The bentonite intercalation process was initiated by dispersing bentonite into the water. Bentonite with a wide surface can absorb water so that hydrates are formed between layers on the bentonite, which causes the bentonite to appear to expand. Bentonite has a layered structure with the ability to swell (swelling) and has cations that can be exchanged. The presence of water molecules allows cations such as Ca²⁺, Mg²⁺, Na⁺, and K⁺ to be transferred because the water in the interlayer space can keep the
layers separate, thereby reducing the electric field between the surface of bentonite and interchangeable ions [28,29].

The intercalation process was continued by adding a cationic surfactant to the bentonite suspension. The cationic surfactant used was Cetyl Trimethyl Ammonium Bromide (CTAB) quaternary ammonium salt, and the mixture was stirred for 24 hours using a magnetic stirrer. The addition of CTAB organic molecules as quaternary ammonium served as a template. The CTAB surfactant, which included a cationic surfactant, would lead to pore formation. Templates functioned as agents that directed the bentonite crystal structure. In this case, the template was a surfactant cation functioned as a cation to neutralize the anionic framework (\([\text{SiO}_4]^{4-}\) or \([\text{AlO}_4]^{5-}\)) [30–32]. This cationic compound would replace the clay surface cations and change the clay surface to be very hydrophobic. When the aluminite and silicate solutions were mixed with the surfactants, the surfactants would form micelles to reduce surface tension, hydrophobic groups would gather, and hydrophilic surfactant heads would move away from their hydrophobic groups so that a cylindrical circle was formed.

![Figure 1. The cleaning process to produce the clean bentonite.](image)

Electrostatically, the hydrophilic group (surfactant head) would interact with the primary building unit of the bentonite. The primary building unit of a bentonite structure was a tetrahedral consisting of a central Si or Al atom surrounded by four oxygen atoms, namely (\([\text{SiO}_4]^{4-}\) or \([\text{AlO}_4]^{5-}\)) [28]. After 24 hours, NiCl\(_2\) was inserted into the mixture of bentonite and CTAB. Ni\(^{2+}\) ions would enter through hydrophobic interactions and act as catalysts. Bentonite added with CTAB surfactant formed a complex compound with a quaternary ammonium salt, which became intercalated and then bound to Ni\(^{2+}\) ions. This mixture experienced structural compaction through a heating process at a temperature of 110°C for three days and produced a cream-colored solid. The solid was crushed and sifted using a 200 mesh sieve. This sifting aimed to increase the distance between intercalated bentonite layers, which in itself also increased the surface area of the bentonite. The next step was calcination to remove surfactants left behind in the intercalated bentonite pores after the heating process. Calcination was carried out at 600°C for 2 hours, which was expected to be able to decompose CTAB so that it left bentonite pores. XRD and SEM characterized the bentonite product.

3.3 Characterization using x-ray diffraction (XRD)
Characterization with XRD in this study aimed to provide information about the crystallization of a particular mineral. The results obtained can also be used to determine the magnitude of the shift in distance between layers of silicate clay. If intercalation occurred, we would see a change in the
distance between the layers marked by variations in bass spacing. The pure bentonite diffractogram can be seen in figure 2.

Figure 2 shows that the pure bentonite used in this study contained montmorillonite selected at an angle of 2θ: 6.05° (d: 14.57 Å); 12.12° (d: 7.29 Å); 20.32° (d: 4.36 Å); 62.16° (d: 1.49 Å) and 73.89° (d: 1.28 Å). Other peaks that appeared at an angle of 2θ: 35.72° (d: 2.51 Å) and 55.52° (d: 1.65 Å) were the peaks resulted from quartz, while the feldspar peaks appeared at an angle of 2θ: 22.02° (d: 4.03 Å). The XRD diffractogram results show the bentonite used could provide enough cavities to continue the NiCl₂ intercalation process. This condition was characterized by a reasonably high montmorillonite content in the bentonite sample used. Montmorillonite is a material consisting of silica and alumina arranged octahedrally and tetrahedrally.

![Figure 2. XRD diffractogram of bentonite.](image)

![Figure 3. XRD Diffractogram of bentonite intercalated NiCl₂ before calcination (a), and bentonite intercalated NiCl₂ after calcination (b).](image)
Figure 3 shows the crystallization of bentonite intercalated NiCl$_2$ before calcination. It can be seen that the intensity of peak sharpness was compared to the peak of pure bentonite diffractogram. Two new diffraction peaks appeared as Ni diffraction peaks, namely at a value of 20 at 44.96° with a basal spacing of (d: 2.01 Å). The new peak appeared as a Ni peak, which was intercalated in the bentonite structure. Figure 3 shows the peaks with low intensity on the diffractogram before calcination was reduced. It indicates that the CTAB surfactant decomposed. In this diffractogram, a new peak appeared with a good sharpness intensity at a value of 20 at 44.67° with a basal spacing (d: 2.03 Å). This peak shows the Ni crystalline phase, which experienced a shift in the 2θ number.

3.4 Characterization using Scanning Electron Microscope (SEM)

The results of micrographs with SEM at a magnification of 15,000x are shown in figure 4. In figure 4 (a) it can be seen that the porosity of pure bentonite before intercalation was relatively large compared to the porosity in intercalated bentonite NiCl$_2$ in figure 4 (b). It can be explained by the appearance of a black pore basin that was indicated as part of the pore of the bentonite. In contrast, the micrograph results in figure 4(b) show fewer pore basins compared to figure 4(a).

![Figure 4](image)

Figure 4. The bentonite micrographs (a). Pure bentonite; (b) NiCl$_2$ intercalated before calcination; (c) NiCl$_2$ intercalated after calcination.

This is presumably because, at the time of intercalation, a portion of the bentonite pore was covered by the surfactant and catalyst being embedded. As a result of the partial closure of pores in intercalated bentonite, the N$_2$ gas that passed through the pore was getting smaller so that the data from the gas absorption analysis showed a reduced surface area value. Figure 4(c) shows the porosity of NiCl$_2$ intercalated after calcination, showing a wider pore surface because the surfactant had decomposed in the calcination process.

3.5 Esterification of cooking oil waste

The esterification process was carried out by reacting methanol with cooking oil waste (ratio of oil and methanol 1: 3) and by adding 1 M H$_2$SO$_4$ catalyst as much as 1.25% of the total weight of oil and methanol. The mixture was refluxed for 2 hours at 60-65°C. The results obtained from the esterification reaction was a mixture of methyl ester and methanol in the upper layer and triglycerides in the lower layer (figure 5). The esterification reaction occurred from a carboxylic acid with alcohol with the help of an acid catalyst to form esters and water. The problem that arose with the use of acid catalysts was the formation of water in the mixture so that the addition of excess alcohol was carried out because the water formed would dissolve in methanol and not inhibit the reaction process. The acid catalyst used in this case was sulfuric acid because it relatively quickly converted free fatty acids into alkyl esters [33].

3.6 Transesterification of waste cooking oil triglycerides

The transesterification of used cooking oil triglycerides was carried out after the esterification reaction. The transesterification reaction was carried out by reacting used cooking oil triglycerides and
methanol in a 1:6 mole ratio and adding KOH catalyst as much as 1% of the total weight of oil and methanol, for 2 hours at a temperature of 60-65ºC. This condition was based on the optimization process that was conducted in another study. Next was the evaporation process of methyl ester (biodiesel) to remove the remaining methanol.

Figure 5. Esterification of the waste cooking oil route.

Figure 6. Transesterification of the waste cooking oil route.

The method of washing methyl esters (biodiesel) was done by using distilled water until the bottom layer was transparent. The washing product consisted of two layers, namely the top layer in the form of methyl ester (biodiesel) and the bottom layer was distilled water and the remaining glycerol (figure 6). The methyl ester (biodiesel) layer was above because the specific gravity of the water was higher than the methyl ester (biodiesel). The separated methyl esters (biodiesel) were then given anhydrous Na₂SO₄ which served to bind the remnants of water that was still contained in methyl esters (biodiesel). It was then filtered using Whatman 40 paper and characterized using GC-MS to determine the compounds contained in the methyl esters.

3.7 Biodiesel characterization using GC-MS
GC-MS analysis was performed on methyl esters (biodiesel) resulting from the transesterification reaction with a KOH catalyst. Through GC-MS, it was found that the type of methyl esters contained in methyl esters (biodiesel) from used cooking oil was produced from the transesterification reaction. The number of peaks indicates the number of transesterified methyl esters, each of which was completely separated, resulting in peaks with different retention times. The top that came out first was the ester with a short carbon chain. The column (stationary phase) used is non-polar, while in general, the ester was polar. Short-chain esters were polar from long-chain esters. The methyl ester with a longer chain would be held in the column while the short-chain ester would escape with the mobile phase-out of the column. The content of methyl esters resulting from GC-MS analysis on used cooking oil methyl esters can be seen in figure 7 and table 1.
Figure 7. Methyl ester chromatogram of biodiesel from cooking oil waste.

| No. | Compounds       | Retention time (minute) | % Areas |
|-----|-----------------|-------------------------|---------|
| 1   | Methyl palmitate| 17.76                   | 32.40   |
| 2   | Methyl oleate   | 22.14                   | 25.88   |
| 3   | Methyl stearate | 22.75                   | 11.38   |

Table 1. Methyl ester content of biodiesel from waste cooking oil.

In figure 3, the chromatogram GC-MS test results showed six peaks, but only three peaks were detected as fatty acid methyl esters (table 1). The methyl esters were methyl palmitate (C_{17}H_{34}O_{2}) with the largest area of 32.40% with a retention time of 17.76 minutes (m/z = 270), methyl oleate (C_{19}H_{36}O_{2}) with a percentage area of 25.88% and a retention time of 22.14 minutes (m/z = 296), Methyl stearate (C_{19}H_{38}O_{2}) with a percentage area of 11.38% and a retention time of 22.75 minutes and (m/z = 298).

3.8 Biodiesel cracking process
Catalyst cracking is a way to break down complex hydrocarbons into simpler molecules so that they can improve the quality and also reduce the amount of residue produced. Cracking biodiesel from used cooking oil in isothermal conditions was carried out using a cracking reactor. A total of 50 mL of sample was put into the sample tube, followed by the inclusion of three gram of the intercalated catalyst NiCl\(_2\) into the catalyst tube. The temperature of the upper furnace was fixed at 350°C. The cracking process was carried out with temperature variations in the lower furnaces of 450°C and 500°C. This temperature selection was based on the optimum conditions of trial and error tests carried out on the reactor.

3.9 Results of cracking process using GC-MS
GC-MS chromatogram analysis provided information on the number of different component compounds between the two temperature variations. Chromatogram results can be seen in figure 8. From the results of the chromatogram at 450°C, there were 11 peaks. The carbon chain breaking in biodiesel resulted in shorter carbon chain compounds. Among these 11 peaks, there were three peaks of compounds belonging to the gasoline fraction, they were thought to be methyl 6-hydroxacapaproic (C_{17}H_{34}O_{3}), 4-hydroxymethyl-1,3-dioxolane (C_{6}H_{12}O_{3}), and methyl nonylate (C_{10}H_{20}O_{2}).

Figure 9 shows that the carbon chain was cut from biodiesel (C_{16-20}) to biogasoline (C_{4-12}). The five short-chain compounds were successfully tracked well due to the acidity of the bentonite catalyst which had been intercalated with NiCl\(_2\). The more active the acidic side of the catalyst was used, the more product was produced. The following chromatogram results of cracking at 500°C can be seen in
The results of the GC-MS chromatogram at 500°C produced seven peak compounds. These compounds underwent carbon chain cuts that were not too far from the biodiesel carbon chain (C_{16-20}).

Three compounds underwent carbon chain-breaking, they were compounds dodecane (C_{12}H_{26}), methyl dodecanoic (C_{13}H_{26}O_{2}), and methyl tridecanoic (C_{14}H_{28}O_{2}). The only dodecane belonged to the gasoline fraction, while methyl dodecanoic and methyl tridecanoic belonged to the kerosene fraction which had a carbon chain length of 13-15 (C_{13-15}). Product conversions dominated by large molecular weight compounds occurred because of the polymerization and re-polymerization reactions. The polymerization reaction could occur between free radicals and carbon molecules that had double bonds to produce products with a higher number of C atoms. In contrast, the re-polymerization reaction was influenced by the length of cracking reaction time and the abundance of products produced. It would cause the catalyst to be deactivated by an abundant product so that it could cover the active site on the catalyst [24]. The GC-MS results obtained showed similarities in the products produced using the cracking process in other studies [27].

4. Conclusion
The biodiesel product contents resulted from cooking oil waste were methyl palmitate (C_{17}H_{34}O_{2}), methyl oleate (C_{19}H_{36}O_{2}), and methyl stearate (C_{19}H_{38}O_{2}). Based on the diffractogram of the intercalated bentonite NiCl$_2$ before the calcination stage, a new peak appeared as a Ni peak, namely at a value of 2θ 44.96°. After the calcination process, it was seen that the Ni peak shifted from 2θ to 44.66°. The results of the morphological study of intercalated bentonite NiCl$_2$ using SEM showed the NiCl$_2$ catalyst had covered a portion of the surface of the bentonite pore. Based on the results of GC-MS analysis on the cracking process at 450°C, it was found that there was a cutting of the carbon chain from biodiesel (C_{16-20}) to biogasoline (C_{4-12}). The compounds were thought to include methyl 6-hydroxy caproic (C_{6}H_{14}O_{3}), 4-hydroxymethyl-1,3-dioxolane (C_{4}H_{8}O_{3}), and methyl nonylate
(C\textsubscript{10}H\textsubscript{20}O\textsubscript{2}). The results of GC-MS chromatograms at 500 °C found that three compounds underwent carbon chain cutting. Those compounds were suspected as dodecane (C\textsubscript{12}H\textsubscript{26}), methyl dodecanoic (C\textsubscript{13}H\textsubscript{26}O\textsubscript{2}), and methyl tridecanoic (C\textsubscript{14}H\textsubscript{28}O\textsubscript{2}).

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