Formosa Journal of Sustainable Research (FJSR)
Vol.2, No.5, 2023: 1091-1108

Effect of Hydrogen on Cracking Reaction Products 9-Octadecenoate (Aleic Acid) Into Alkane Short Fraction Compounds

Donatus Setyawan Purwo Handoko[1*], Triyono[2]
[1]Fakultas Matematika dan Ilmu Pengetahuan Alam Universitas Jember
[2]Universitas Gadjahmada, Yogyakarta Sekip Utara, Bulaksumur, Yogyakarta
Corresponding Author: Donatus Setyawan Purwo Handoko
kreka_zeolit@yahoo.com

A R T I C L E I N F O

Keywords: Cracking, Calcination, 9-Octadecenoate

Received : 4 April
Revised : 18 April
Accepted: 19 May

A B S T R A C T

Research has been carried out on the effect of hydrogen on the cracking of 9-octadenoate into short fraction compounds. The research was conducted at the UGM physical chemistry laboratory with Prof. Dr. Triyono, MS. The research implementation that must be prepared first is the Ni/Zeolite catalyst. Ni/Zeolite catalyst preparation was carried out by immersing 500 grams of zeolite in H2O for 24 hours while stirring. Then proceed with calcination with nitrogen gas for 3 hours at a nitrogen gas flow rate of 2.5 mL/minute. After that it was cooled and continued with oxidation with oxygen gas flowing at a rate of 2.5 mL/minute for 3 hours. Then after that, cooling was carried out and then followed by acid treatment through a reflux process with 4 Molar HCl for 3 hours and every 1 hour replaced with the same solution. After that, it was followed by reflux treatment with 1M NH4Cl for 1 hour, after that it was cooled and filtered, then the oxidation process was carried out with oxygen gas for 3 hours, with an oxygen flow rate of 2.5 mL/minute. Then proceed with reduction with hydrogen gas for 3 hours at a flow rate of 2.5 mL/minute after that the catalyst is cooled. After cold, impregnation was carried out with metal Ni(NO3)2.9H2O, after impregnation a reduction process was carried out with hydrogen gas for 3 hours at a flow rate of 2.5 mL/minute, so that a Ni/zeolite catalyst was obtained

DOI: https://doi.org/10.55927/fjsr.v2i5.4160
ISSN-E: 2830-6228
https://journal.formosapublisher.org/index.php/fjsr

INTRODUCTION

Factors that affect the quality of palm oil seeds are the presence of water and impurities, free fatty acid content, peroxide value and bleaching power, melting point, solid glyceride content, refining loss, plasticity and spreadability, transparency, heavy metal content and saponification number. (Ketaren, 1986).

The main components in palm oil are palmitic acid and oleic acid which are composed of other fatty acids and these fatty acids form triglyceride bonds with glycerol. Palmitic acid is a saturated fatty acid with a chain length of 16 carbons, while oleic acid is an unsaturated fatty acid with a chain length of 18 carbons and has one double bond. Linolenic acid is also an unsaturated fatty acid and has a chain length of 18 carbon with 3 double bonds. Linolenic acid in palm oil is generally in very small amounts.

Table 1. Composition of the Main Fatty Acids in Palm Oil and Palm Kernel Oil (Pioch, 2005)

Asam lemak	**Symbols**	**Palm (%)**	**Kernels (%)**
Caprylate	C8 : 0	-	3 – 4
Kaprat	C10 : 0	-	3
Laurat	C12 : 0	< 0,2	**45 – 52**
Myristat	C14 : 0	1 – 2	**14 – 19**
Palmitate	C16 : 0	**43 – 46**	6 – 10
Stearate	C18 : 0	4 – 6	1 – 3,5
Oleic	C18 : 1	**37 – 41**	11 – 19
Linoleic	C18 : 2	9 – 12	0.5 – 2
Linolenic	C18 : 3	< 0.4	< 0.3

The physical properties of oleic acid and palmitic acid are as follows, oleic acid has a chain length of C: 18 and palmitic acid has a chain length of C: 16.

Table 2. Physical Properties of Oleic Acid and Oleic Ester(Perry's, 1997)

Compound Name	**Formula**	**MW (g/mol)**	**BP (^oC)**
Oleic acid	$C_8H_{17}CH=CH(CH_2)_7COOH$	282	285^{100}
Methyl oleate	$C_8H_{17}CH=CH(CH_2)_7COOCH_3$	296,48	190^{10}

Note: Boiling Point 190^{10} Means Boiling At 190^oc Under Pressure10 Mmhg

Based on Table 2, the content of palmitate and oleate in the form of triglycerides has the most dominant amount in palm oil. Pure palmitate in the solid phase is white, while oleate in the liquid phase is clear yellow. The difference between palmitate and oleate is due to the different types of bonds they have. Palmitate has no double bonds in its structure, whereas oleate has one double bond in its structure.

The definition of a catalyst is a substance that can accelerate the achievement of an equilibrium state of a reaction without the catalyst being permanently involved in the reaction (Augustine, 1996). Therefore, if thermodynamically a reaction cannot occur, then the presence of a catalyst in the reaction will also not cause the reaction to occur, so that it can be said that the catalyst is not a substance that starts the reaction. The catalyst plays an active role in the reaction process but does not turn into a product compound. The active role of the catalyst can be seen from the interaction between the catalyst and the reactants during the reaction, both physisorption and chemisorption (Satterfield, 1980).

The interaction of the catalyst with the reactants always refers to equilibrium, which means that not all of the reactants can turn into reaction products (Satterfield, 1980). The interaction of catalysts with reactants in general includes physisorption, chemisorption and desorption processes. The interaction between the catalyst and the reactants can produce compounds that are more active as intermediates and can increase the rate, accuracy and concentration of encounters as a result of the localization of the reactants. As a consequence of this situation, the activation energy of the reaction becomes lower.

Activation energy is the minimum energy required for a reactant to form an intermediate compound (Activated complex state), before the reactant turns into a product. Kinetics of the activation energy is affected by the presence of a catalyst. The reaction using heterogeneous catalysts includes several main steps, namely physisorption, chemisorption and desorption. The difference in the reaction path using a catalyst and without a catalyst is explained in Figure 1.

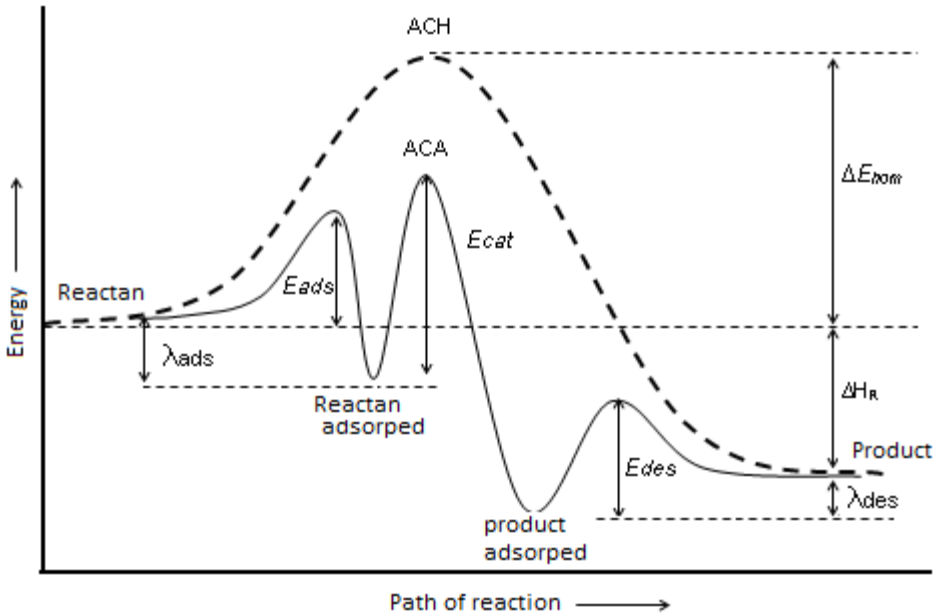


Figure 1. Energy Changes in the Catalytic Reaction Stages Compared to the Reaction Without a Catalyst (Satterfield, 1980 and Gasser, 1987)

Table 3. Heat of Adsorption of Reactants (Exothermic)

ads		Heat of adsorption of reactants (Exothermic)
ads		Activation energy in the process of physisorption of reactants on the surface of the catalyst
CH		The state of the activated complex without a catalyst (Activated complex homogenous)
CA		Adsorbate activated complex state with a catalyst (Activated complex adsorbed)
cat		Activation energy in the process of chemisorption of reactants with a catalyst to form the ACA state (Exothermic)
Edes		The activation energy for the desorption process
ΔE_{hom}		The activation energy of a reaction without a catalyst
ΔH_R		Enthalpy of uncatalyzed reaction (Exothermic process)
λdes		Enthalpy of desorption process (Endothermic)

The stages in the catalytic reaction with heterogeneous catalysts according to Wu (2005) and Augustine (1996), are as follows:

1. Diffusion (Mass transfer) of the reactants from the fluid phase to the outer surface of the catalyst.
2. Diffusion of the reactants from the pore mouth through the catalyst pores to the area around the inner surface of the catalyst.
3. Adsorption of reactants on the surface of the catalyst.
4. Reaction on the surface of the catalyst.
5. Desorption of reaction products from the surface of the catalyst.
6. Product diffusion from the inner surface through the catalyst pores to the pore mouths on the outer surface.

Diffusion (Mass transfer) of the product from the catalyst surface to the fluid phase.

Table 4. Types of Catalysts, Functions and Examples of Catalysts

No.	Catalyst Type	Function	Example
1	Logam	hydrogenation, Dehydrogenation	Fe, Ni, Pt, Ag
2	Semiconductive Oxides and Sulfides	Oxidation, Desulfurization	NiO, ZnO, MgO Bi_2O_3, MoO_3
3	Insulating oxide	Dehydration	Al_2O_3, SiO_2, MgO
4	Acid	Polymerization, Isomerization, Alkylation	H_3PO_4, H_2SO_4, SiO_2/Al_2O_3

(Satterfield, 1980)

METHOD

Tools and Materials

The equipment needed in the research were: a set of fixed bed reactors, thermocouples, thermometers, ovens, hot-plates, laboratory glassware, analytical balance (Mettler RE 200), atomic absorption spectroscopy (AAS, Varian FS 220), GC- MS (Shimadzu QP-2010S), surface area analyzer NOVA 1000 (Quantachrome Nova Win2 version 2.2), X-ray diffractometer (XRD, Shimadzu-6000).

The research materials used were: natural zeolite Wonosari from PT Prima Zeolita, materials from E.Merck and quality p.a. consists of: oleic acid (91.59 %),1-octadecanol 95 %, 1-octadekene 90 %, HCl 37 % (v/v), HF 40 % (v/v), NH4Cl, Na2SiO3, Ni(NO3) 2·6H2O 97 %. Other materials such as: oxygen, nitrogen and hydrogen gas (P.T. Samator Gas), pH paper.

Research Method

1.Ni/ZSiA Catalyst Preparation

Zeolite with a pass size of 100 mesh is soaked in distilled water and washed while stirring. Then soaked with 2% HF for 30 minutes then washed with distilled water repeated up to 3 times, then dried in an oven at 120 oC for 3 hours. Then it was oxidized with oxygen at a temperature of 500 oC for 2 hours and calcined with nitrogen at a temperature of 500 oC for 2 hours with a gas flow rate of 20 mL/minute to obtain catalyst Z (Handoko, 2001).

Furthermore, the Z catalyst was washed using 2 M HClsolution with a volume ratio of zeolite: HCl solution = 1: 2, while stirring for 20 to 30 minutes (Zhang, 1999). Then the zeolite samples were washed using distilled water until pH = 6 and dried in an oven at 120 oC for 3 hours and continued with oxidation using oxygen gas at a flow rate of 20 mL/minute at 500 oC for 2 hours and calcination with nitrogen gas at a rate flow 20 mL/minute at 500 oC for 2 hours. Then 5% (w/w) Na-Silicate (Na2SiO3) was added to the Z catalyst in the beaker glass which was dissolved in distilled water and then heated at temperatures between 80 oC to 90 oC for 24 hours while stirring using a magnetic stirrer. Then the sample was cooled and continued to be oxidized with oxygen gas at a temperature of 500 oC for 2 hours at a gas flow rate of 20 mL/minute and continued with calcination with nitrogen gas at a temperature of 500 oC for 2 hours at a gas flow rate of 20 mL/minute to obtain a ZSi catalyst.

The ZSi catalyst was cooled and 2 M NH4Cl solution was added to the beaker glass with a ratio of 1: 2 (v/v) and the mixture was heated again at 90 oC for 4 hours with a magnetic stirrer (Zhang, 1999). Then cooled and continued with the oxidation process using oxygen gas at a temperature of 500 oC for 2 hours with a gas flow rate of 20 mL/minute and continued with calcination with nitrogen gas at a temperature of 500 oC for 2 hours with a gas flow rate of 20 mL/minute to obtain a ZSiA catalyst.

Impregnation of Ni metal (Ni 2 % (w/w)) on the ZSiA catalyst surface was carried out using the wet impregnation method. 9.91 g of Ni(NO3)2·6H2O salt was dissolved in 100 mL of distilled water while stirring until homogeneous, then 100 g of sample (ZSiA catalyst) was added. Then it is heated and evaporated at a temperature of 80 oC to 90 oC (At 1 atm) while stirring so that the water component will slowly evaporate. After the water component was evaporated, the sample was put into the oven at 120oC for 2 hours and continued with the oxidation process with oxygen gas at 500oC for 2 hours with a gas flow rate of 20 mL/minute and reduction at a temperature of 500oC with hydrogen gas flowing at 20 mL/minute to obtain a Ni/ZSiA catalyst (Handoko, 2001).

Each treatment stage was analyzed for metal content by AAS, acidity by gravimetric method, crystallinity by XRD and surface area by BET method.

2. Preparation of Methyl 9-octadenoate

24 mL of methanol was mixed with 2.96 mL of sulfuric acid and then added to a two-neck flask containing 100 mL of oleic acid. The mixture was refluxed for 4 hours at 60 ^{0}C and the reflux results were left overnight. Furthermore, the organic layer (Ester layer) is separated from the water layer using a separating funnel. The ester layer was washed with distilled water until neutral and dried with anhydrous Na_2SO_4. The resulting methyl 9-octadenoate was heated in an open container at a temperature of 100-120 ^{0}C and then analyzed by GC-MS.

3. Hydrogenation of Methyl 9-octadenoate with ZSiA Catalyst

ZSiA catalyst with weight variations of 5, 10 and 15 g was placed in a fixed bed reactor column and then heated to a temperature of 400 oC. Furthermore, hydrogen with a flow rate of 20, 40, 60 mL/minute was flowed through 10 g of the feed compound (Methyl 9-octadenoate) so that it passed through the catalyst. The process is carried out for 30 minutes. The products obtained were analyzed by GC-MS.

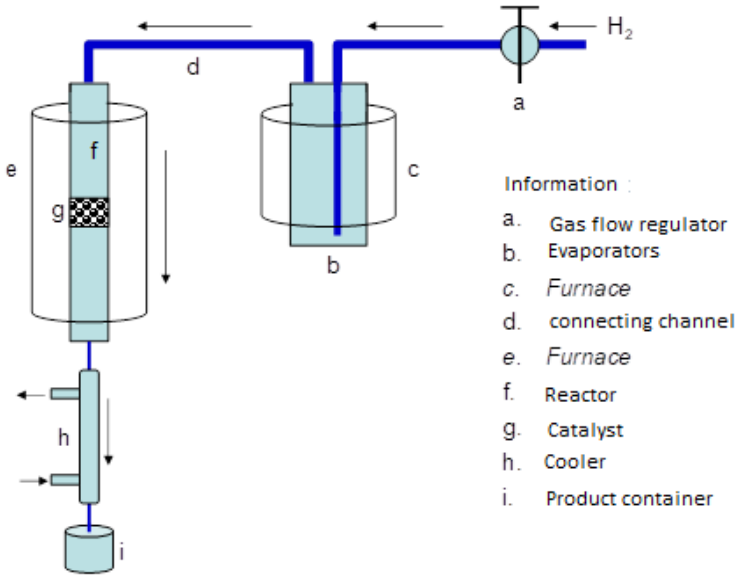


Figure 2. Fixed Bed Reactor (Equipment Used in Processing Hydrocracking)

RESULT AND DISCUSSION

1. Catalyst Crystallinity

According to Harber (1991), the requirements for zeolite materials as catalysts are surface area, Si/Al ratio, acidity, cation content and crystallinity. These properties are closely related to the pore radius, pore volume and acidity contained in the zeolite. Zeolite crystallinity is a measure of the strength of the crystal lattice in maintaining its crystalline shape. One of the requirements for a material as a catalyst is that the material must have crystalline properties and be stable when the material is used as a catalyst.

The nature of the zeolite crystal can also affect its ability to adsorption. If a zeolite has low crystalline properties, the zeolite crystal lattice will be easily damaged and will cause clogging of the pore mouths, reduction in pore volume, and a decrease in the amount of acid. As a further consequence, the zeolite will experience a decrease in its activity as a catalyst.

The method used to analyze the crystal structure of the prepared catalyst is X-ray diffraction (XRD). The basic principle of chemical analysis used in XRD is the characteristic distance between planes (d). The position of the diffraction angle (2□) and the distance between the planes describe the type of crystal, while the intensity indicates the crystallinity of a solid (West, 1984; Sibilia, 1996). Quantitative analysis was carried out by comparing the diffractogram of natural zeolite samples with standard natural zeolite diffractograms.

The Z catalyst exhibits crystalline properties, as well as after being treated with sodium silicate (Si enrichment) and acid treatment (HF, HCl, NH4Cl) it is expected that ion exchange will occur and the formation of Bronsted acid sites (ZSiA catalyst). The treatment with impregnation of Ni metal onto the surface of the ZSiA catalyst also still exhibits crystalline properties as shown in Figure 3.

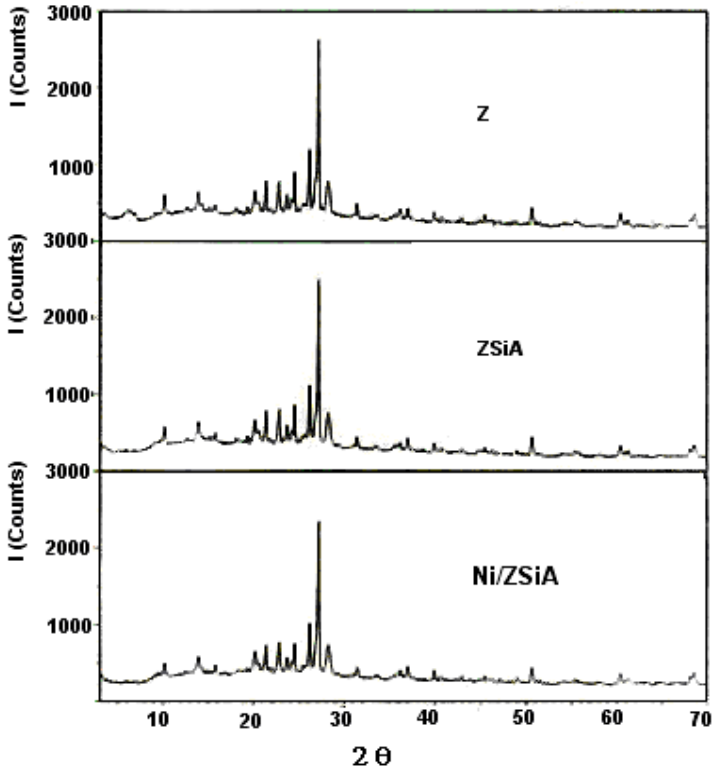


Figure 3. Diffractogram of Z, ZSiA, Ni/Zsia Catalysts

2. Metal Content in the Yield Catalyst

In the calcination treatment and Si enrichment of zeolite to acid treatment, the content of Ca and Fe metal decreased as shown in Figure 4. In the Ni/ZSiA catalyst, the content of Ca and Fe increased although relatively very little but not too significant.

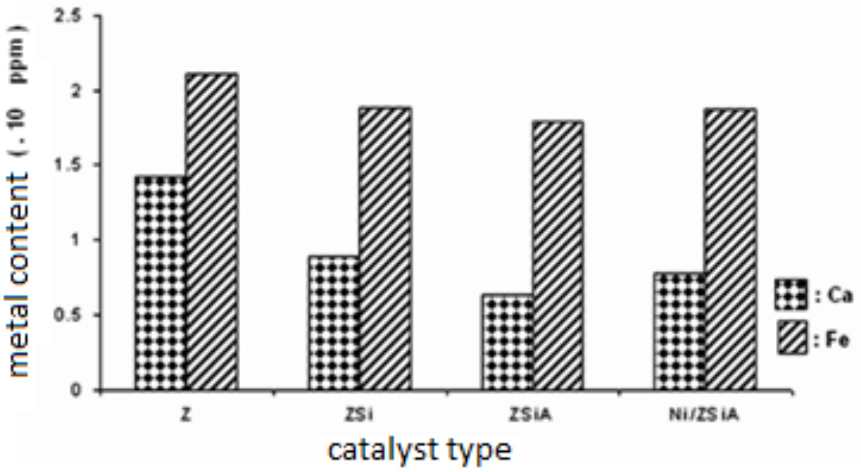


Figure 4. Ca and Fe Metal Content of Various Types of Catalysts, Z: Zeolite Catalyst, Zsi: Zeolite Enrichment Catalyst with Si (Na2SiO3), Zsia: Acid Treated Zsi Catalyst, Ni/Zsia: Zsia Catalyst Impregnated with Ni Metal

When immersing and stirring during the impregnation of Ni metal from Ni (NO3)2·6H2O salt there was an increase in Ca and Fe metals. The relatively slight increase was probably caused when the impregnation of Ni metal from Ni (NO3)2·6H2O salt used a water solvent containing Ca and Fe so that Ca and Fe metal were re-adsorbed by the zeolite, although relatively very little. Apart from these reasons, another possibility is that the basic ingredient of Ni (NO3)2·6H2O salt contains Ca as much as 50 mg/kg and Fe as much as 50 mg/kg, so when impregnation of Ni metal from Ni(NO3)2·6H2O the content of Ca and Fe in zeolite to rise again.

The content of metals such as Ca, Fe, Na and Mg in uncontrolled yield catalysts can interfere with the performance of the catalyst (Harber, 1991). In the preparation of metal/carrier system catalysts, zeolite before being used as a Ni metal carrier is subjected to acid treatment which aims to reduce or eliminate the presence of unwanted metals as catalysts such as Ca, Fe, Na and Mg (Lestari, 2010), so that when Ni metal impregnation is expected to reach the maximum quantitatively.

The acid used in the preparation of the metal/carrier system catalyst is 2 M HCl which aims to dissolve the metals in the zeolite through ion exchange techniques to form Bronsted acid sites (Satterfield, 1980). As an example of the exchange of metal ions, M+ with H+ which is described in Figure 5.

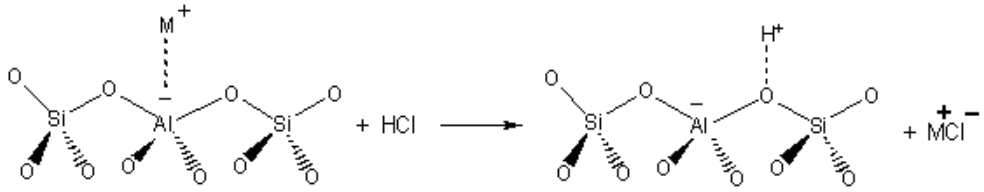


Figure 5. Exchange of M+ Ions (Metal Ions) with H+ Ions in Zeolite

3. Catalytic Hydrogenation of Methyl 9-octadenoate

The 9-octadenoate methyl compound with a boiling point of 351.4 oC before being processed using a catalyst was analyzed using GC-MS. The GC-MS chromatogram is presented in Figure 6.

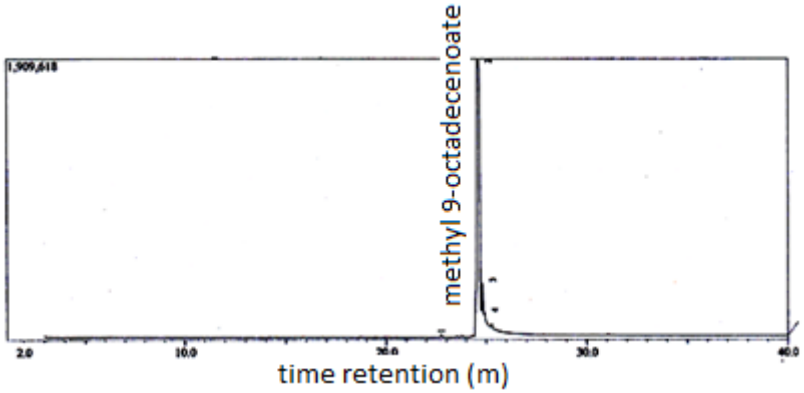


Figure 6. GC-MS Chromatogram of Standard 9-Octadenoate Methyl Compound

GC-MS chromatogram of 9-octadenoate methyl samples showed 4 dominant compounds. The most dominant peak was the peak with a retention time (tR) of 24.61 minutes (methyl 9-octadecanoate) with a relative concentration of 91.59%, a retention time of 24.81 minutes (methyl octadecanoate) with a relative concentration of 5.50% and a retention time of 24.81 minutes (methyl octadecanoate) with a relative concentration of 5.50% and retention 24.91 minutes (methyl 9.12-octadekadienoate) with a relative concentration of 2.51%.

Table 5. Methyl Ester Content Resulting from Initial 9 Octadenoic Acid Esterification (Estimated from Shimadzu GC-MS QP2010 Library Data)

Compound	t_R (m)	amout (%)
Methyl 9-ocktadecenoate	24,61	91,59
Methyl octadecanoate	24,81	5,50
Methyl 9,12-octadecadienoat	24,91	2,51
Amount		99,60

Thermal cracking at 400 oC using a fixed bed reactor system did not show any different results from the initial components. The results of the thermal process showed that 90.93% was methyl 9-octadenoate which was relatively dominant. The catalytic cracking process with ZSiA catalyst using a fixed bed system reactor operated at a temperature of 400 oC and a hydrogen flow rate of 60 mL/minute and a catalyst weight of 5 g produced 34.50% 1-octadecanol compound.

4. Conversion of Methyl 9-Octadecenoate to Alcohol

The reaction mechanism of methyl 9-octadenoate to 1-octadecanol in this reaction system is thought to follow two stages. The first step is the adsorption of methyl 9-octadenoate on the surface of the catalyst through the interaction of the Bronsted acid site with the double bond at C number 9 and the opening of the double bond (addition reaction) occurs according to the reaction mechanism according to Horiuti-Polanyi in Campbell (1988).

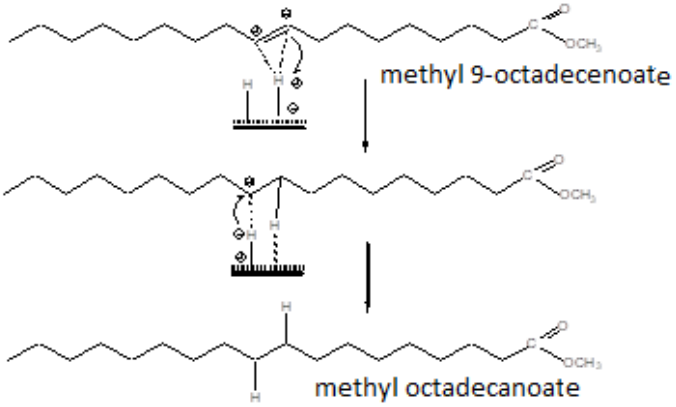


Figure 7. Changes from Methyl 9-Octadenoate to Methyl Octadecanoate

The second step is the reduction of the ester group into an alcohol group (Fessenden and Fessenden, 1986) as shown below, so that 1-octadecanol is produced. Both stages can occur as the feed passes through the catalyst.

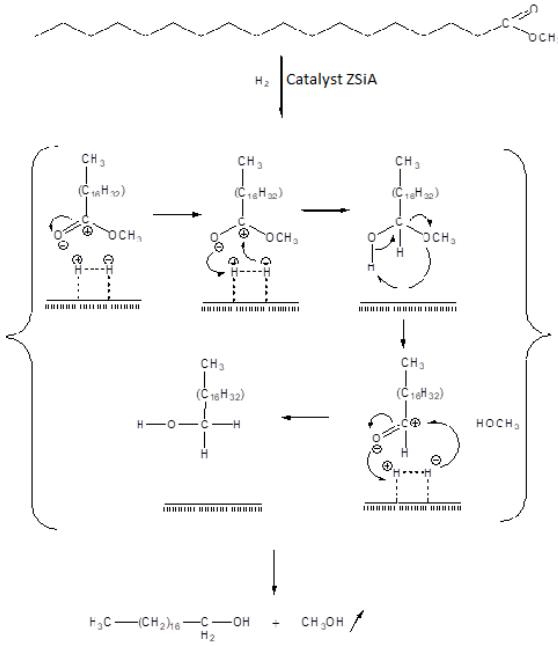


Figure 8. Conversion of Methyl Octadecanoate to Alcohol

5. Convert 1 Octadecanol to 1-Octadecene

The amount of hydrogen is quite abundant and the physisorption process between hydrogen and the 1-octadecanol functional group is expected to be quite effective. In the experiment, researchers were unable to increase the pressure due to leaks in the reactor and relatively high operating conditions, so it was very dangerous.

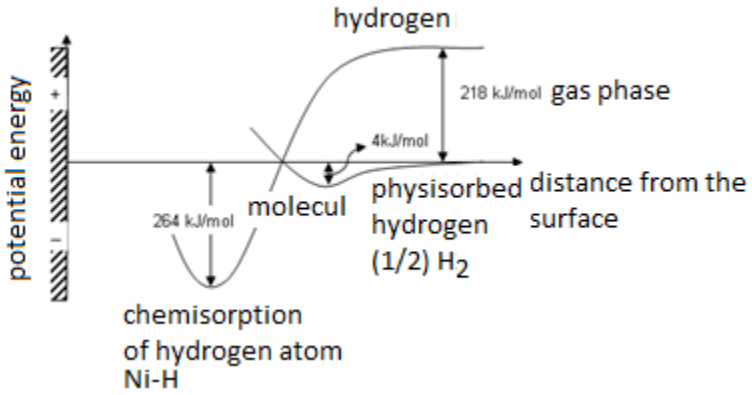


Figure 9. Potential Energy Diagram (Campbell, 1988)

The less optimum adsorption of hydrogen on the catalyst surface due to insufficient pressure also causes the quantity of hydrogen on the catalyst surface to quantitatively decrease, so that the probability of a collision between the adsorbed hydrogen and the 1-octadecanol functional group also decreases, thus the resulting alkanes and alkenes are 49. 60%. The process of collisions can take place more easily if it starts with a physisorption process, then continues with a chemisorption process.

The catalytic hydrogenation of 1-octadecanol with ZSiA catalyst in a fixed bed reactor at a temperature of 400 ^{o}C resulted in alkanes and alkenes with a chain length < C18 of 49.60%. The mechanism of the catalytic hydrogenation of 1-octadecanol is thought to follow two main steps, namely reduction of the alcohol group to an alkene and breaking of alkene bonds to form alkanes and shorter chain alkenes.

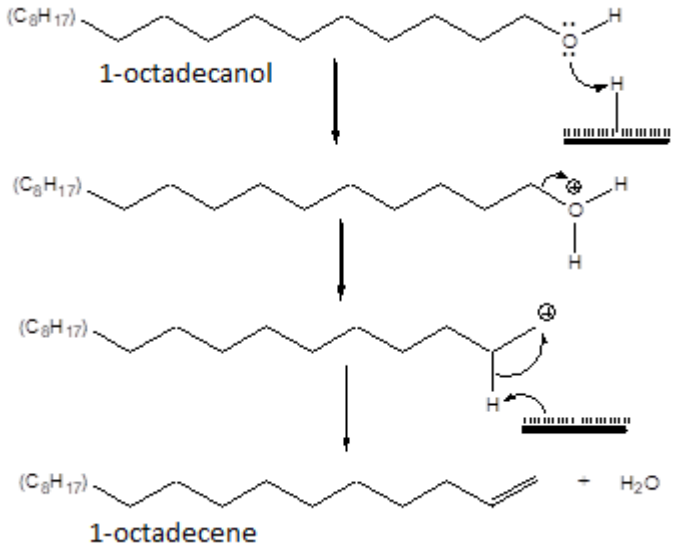


Figure 10. Formation of 1-Octadecene from Alcohol

According to Campbell (1988), the 1-octadekene compound then formed its isomers, namely 5-octadekene and 9-octadekene with relative concentrations of 14.37 and 10.40%.

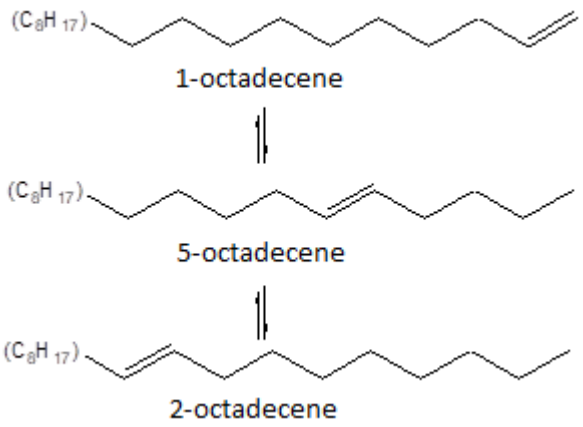
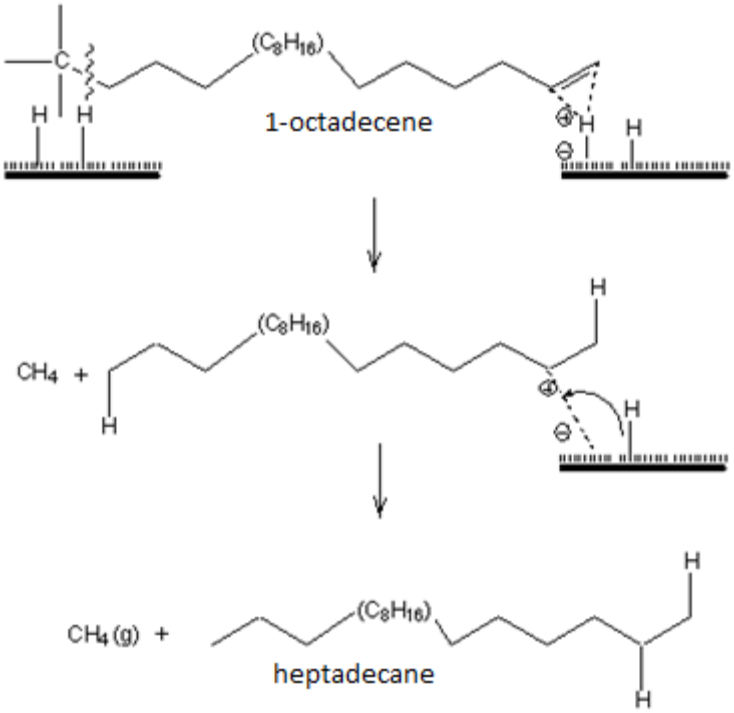


Figure 11. Isomers of 1-Octadekene

Furthermore, the 1-octadekenes, 5-octadekenes and 9-octadekenes undergo further cracking into shorter alkanes and alkenes.



Picture 12. Termination of the 1-Octadecene Bond to Become a Shorter Compound

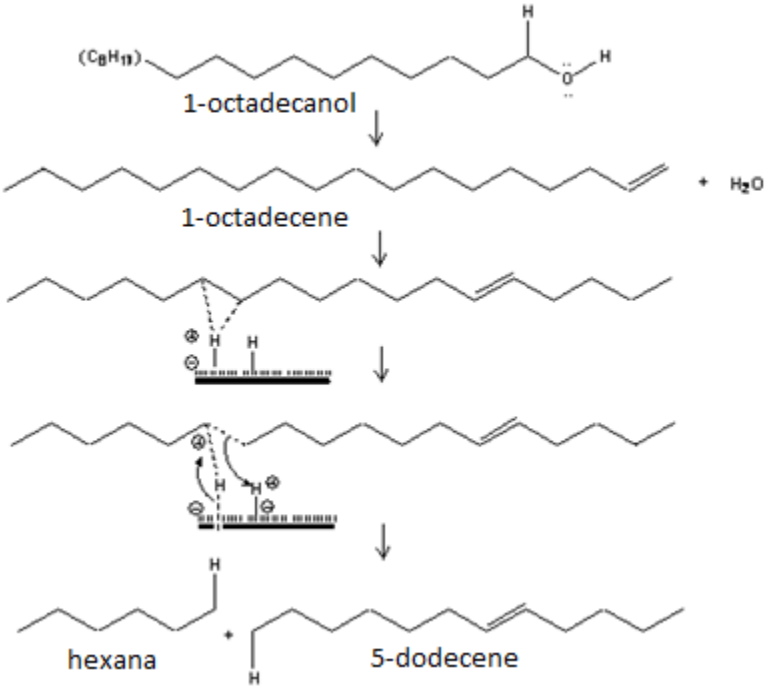
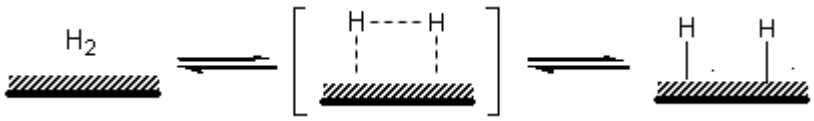


Figure 13. Conversion Of 1-Octadecanol to Shorter Compounds

Then hexane decomposes into gaseous compounds or volatile compounds as follows,

Figure 14. Conversion of Hexane to Compounds with Shorter Chains

With this dissociation and migration process, the probability of an effective collision of the adsorbed hydrogen with the adsorbed 1-octadekene compound on the surface of the catalyst will be even greater. The interaction of hydrogen with 1-octadecene can be illustrated in Figure below. Kinetics can be explained that the more hydrogen adsorbed on the surface of the catalyst can cause the reaction rate constant to increase.



$$H_2 + surface \rightleftharpoons [\ 2H\cdot + \cdot surface\ ] \rightleftharpoons H - surface$$

$$k = \frac{[H - Surface]}{[H_2][Surface]}$$

$$k = e^{-\Delta H / RT}$$

Figure 15. The Interaction of Hydrogen

At a hydrogen flow rate of 10 mL/minute at a reaction temperature of 450 oC it is estimated that relatively many hydrogen free radicals are formed, so that the probability of interaction between free radicals and 1-octadecene compounds can increase quantitatively and produce reaction products which are characterized by increasing gains reaction product.

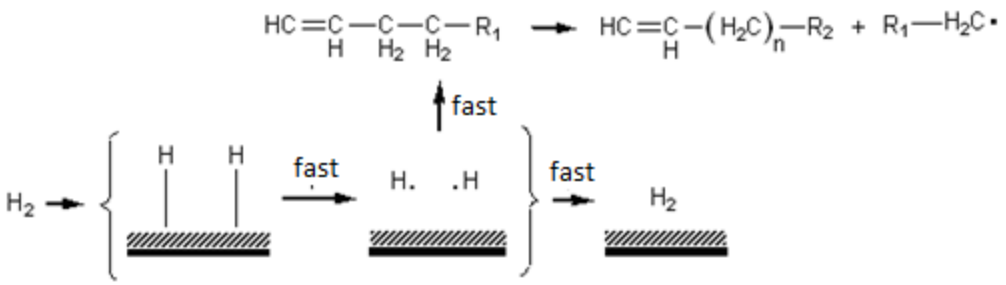


Figure 16. Events of the Formation of H Free Radicals and Attack the 1-Octadekene Compound

At a reaction temperature of 450 oC and a hydrogen flow rate of 10 mL/min, it is estimated that the reaction time is relatively fast, so that radical recombination is possible, so the reaction probability and product production are faster.

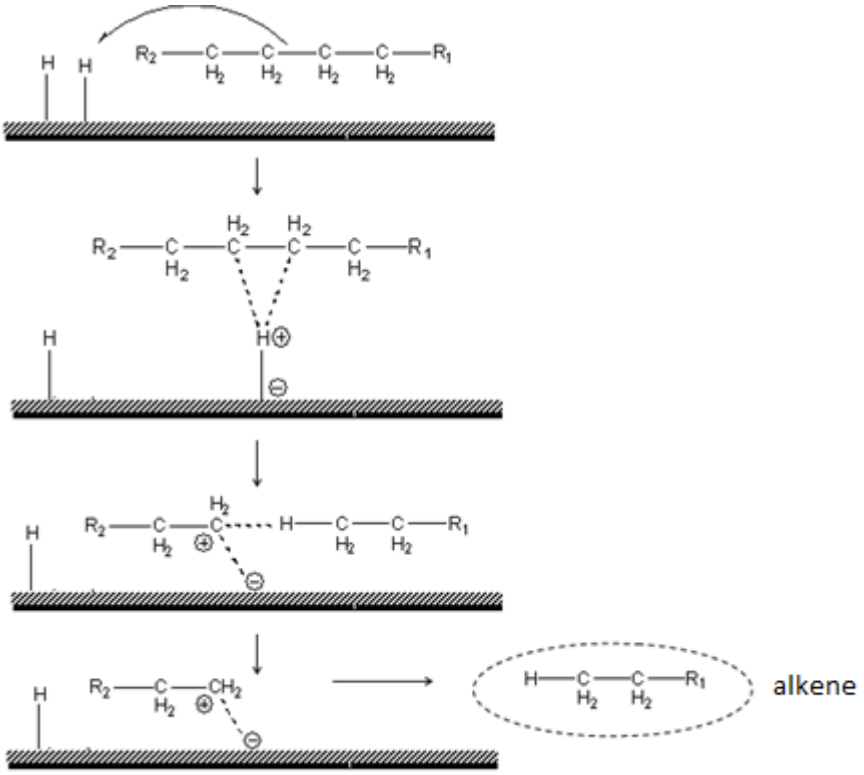


Figure 16. Mechanism of Cracking Reaction on the Surface of the Catalyst in the Formation of Short Chain Alkanes

The process of breaking alkane compounds obtained from the interaction between the surface of the catalyst which is in the upper layer can interact again with the surface of the catalyst which is in the lower layer in line with the feed flow. It is possible for this interaction to produce even shorter alkane compounds and possibly to obtain other forms of compounds such as cyclic, aromatic or isomeric forms.

CONCLUSION

Based on the results of the discussion, it can be concluded that the hydrogenation of the products of the 9-octadenoate cracking reaction (oleic acid) into short fraction alkanes and alkenes with the higher the hydrogen rate, the less product will be produced and if the hydrogen flow rate is too slow, the less product will be produced. Moderate hydrogen flow rate is 10 mL/min.

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