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Synthesis of Nitro Ethyl Oleic from Used Cooking Oil

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Abstract: Nitro ethyl oleic has been synthesized from used cooking oil. This study aims to synthesize nitro ethyl oleic from used cooking oil. The target compounds of nitro ethyl oleic are obtained through four stages, namely the stages of purification, esterification, transesterification, and nitration. From the purification stage of used cooking oil with sago pulp activated charcoal adsorbent is obtained fragrant and delicious pure cooking oil, yellow with water content of 0.13%, acid number 1.10 mg KOH/g fat, FFA level of 1.57%, and turbidity of 2.45 NTU. From the esterification stage was obtained methyl oleic ($\rho = 0.887 \text{ g/cm}^3$) and oil (triolein) in light brown with FFA level of 0.34%. From the transesterification stage was obtained ethyl oleic (83.33% acquisition) in the form of yellow liquid ($\rho = 0.902 \text{ g/cm}^3$), fragrant, and boiling point 77°C . From the nitration stage was obtained nitro ethyl oleic (66.67% acquisition) in the form of blackish brown liquid ($\rho = 1.587 \text{ g/cm}^3$), typical of biodiesel with a boiling point of 80°C .

Keywords: esterification, transesterification, nitration, nitro ethyl oleic, used cooking oil.

1. INTRODUCTION

The limited fossil fuel resources have encouraged the development of alternative fuels. As a result, increased attention has been given to renewable resources such as vegetable oils and animal fats. One of the abundant and wasted vegetable oils is used cooking oil. Used cooking oil is used cooking oil whose color has been degraded to blackish brown due to repeated frying at high temperatures. Used cooking oil contains triglycerides, so used cooking oil can be used as raw material for making biodiesel. Biodiesel produced from renewable resources such as used cooking oil has great potential as an alternative fuel because it is environmentally friendly.

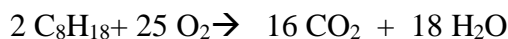
One important parameter in determining the quality of diesel oil is the octane number (Knothe, 2005; Bamgboye and Hansen, 2008). Low quality diesel oil is indicated by octane numbers of less than 48. Diesel fuel in Indonesia has 47 octane numbers (Aufar and Hendra, 2017). Low octane numbers cause knocking sounds on the engine, incomplete combustion, wasteful fuel, noisy, vibrating and removing pollutants in large quantities (Nasikin and Ade, 2003; Wardhana, 2004).

To reduce the sound of beats on the engine, the octane number of the fuel must be increased. The octane number can be increased by adding antiknock compounds (additives) to the fuel. One example of a commercially produced synthetic additive is ethyl hexyl nitrate (EHN). EHN additive is an organic nitrate compound synthesized from petroleum derivatives, so EHN is a non-renewable and expensive material (Nasikin, 2003). Therefore, we need to find other sources of raw materials to synthesize additives.

Additive substances can be synthesized from used cooking oil biodiesel (trioleic) by means of nitration. Nitration is an electrophilic substitution reaction which aims to enter a nitro (NO_2) group into a compound. Through nitration, the amount of oxygen in biodiesel increases, so nitrate alkyl esters have more oxygen. Therefore, nitration products can be referred to as additives in fuel.

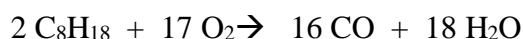
Increasing the amount of oxygen in biodiesel can improve the quality of hydrocarbon fuels because oxygen is needed in the combustion process. If enough oxygen is available, then the

combustion products of hydrocarbon fuels (thought to consist of pure octane) in the engine of the car are carbon dioxide and water (Brady, 1990).

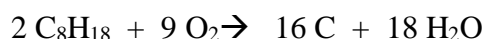


If the reaction occurs as above, the combustion reaction is called complete combustion because all oxygen reacts perfectly with octane fuel to form CO_2 and H_2O .

However, if the oxygen supply is more limited, then the combustion reaction is called incomplete combustion because the number of moles of oxygen reacting with octane fuel is very limited, so the combustion products are carbon monoxide and water.



With a more limited supply of oxygen, combustion products are carbon and water.



2. RESEARCH METHOD

Tools

Measuring cup (Pyrex), beaker (Pyrex), erlenmeyer (Pyrex), funnel (Pyrex), separating funnel (Pyrex), spatula, vortex, volume pipette, drop pipette, vial bottle, dark bottle, stirring rod, thermometer, bottle spray, oven, statif, clamps, analytic balance, magnetic stirrer, hot plate, triple neck flask, picnometer, and FTIR Buck M500 (Scientific) spectrometer.

Materials

Used cooking oil, sago pulp activated charcoal, ethanol pa, phosphoric acid pa, potassium hydroxide pa, concentrated nitric acid pa, concentrated sulfuric acid pa, Whatman No.42 filter paper, universal indicator, aluminium foil, tissue, and aquabidest (Onelab Waterone).

Prosedures

1. Purification of Used Cooking Oil

A total of 500 mL of used cooking oil and 100 g of activated charcoal sago pulp are put into a beaker. The mixture is stirred with a magnetic stirrer for 60 minutes at 80 °C. Then the mixture was left to stand for 12 hours then the mixture was filtered with Whatman No. 42 filter paper. Pure oil is collected in beakers and activated charcoal residues are disposed (Hidayati et al., 2016).

Esterification

The oil purified used cooking oil (150 mL, 1 mole), methanol (105 mL, 20 moles), and H_2SO_4 98% (1.5 mL, 1% of the weight of oil) are put in a three neck flask equipped with a condenser and thermometer. Then the mixture was refluxed at 65°C while stirring with a magnetic stirrer for 1 hour. The mixture is put into a separating funnel and allowed to stand for 24 hours until two layers are formed. The top layer is methyl oleic and oil (triolein) as the main product, while the lower layer is a mixture of water, methanol and sulfuric acid as the side product. Then the two layers are separated from each other. Then the top layer is washed with aquabidest until the pH of the solution becomes neutral (Arita et al., 2008).

Transesterification

Methyl esters and oil (125 mL, 1 mole), ethanol (31.55 mL, 6 moles), and KOH (1.1 g, 1% of the weight of oil) are put in a three neck flask equipped with a condenser and thermometer. Then the mixture was refluxed at 78°C while stirring with a magnetic stirrer for 1 hour. The mixture is put into a separating funnel and allowed to stand for 24 hours until two layers are formed. The top layer is ethyl oleic as the main product and the bottom layer is glycerol as the side product. The ethyl oleic solution is washed with aquabidest until the pH of the solution becomes neutral. Then the ethyl oleic solution is heated until the ethyl oleic becomes free of water (Aufar and Hendra., 2017).

Nitration

Nitric acid (2.5 mL, 1 mole) and sulfuric acid (7.5 mL, 1 mole) are inserted into a three neck flask equipped with a condenser, thermometer, and separating funnel. Dry ethyl oleic (16.5 mL) is inserted into a separating funnel. Then dry ethyl oleic is added dropwise to a solution of nitric acid and sulfuric acid. Then the mixture is refluxed at room temperature while stirring with a magnetic stirrer for 4 hours. The mixture is put into a separating funnel and allowed to stand for 24 hours until two layers are formed. Furthermore, the two layers are separated and the upper layer is residual water and acid as the side product and the bottom layer is nitro ethyl oleic as the main product (Canoira et al., 2007 and Abdullah et al., 2010).

3. RESULTS AND DISCUSSION

Purification of Used Cooking Oil

The results of testing the quality of used cooking oil before and after refining using sago pulp activated charcoal are shown in Table 1.

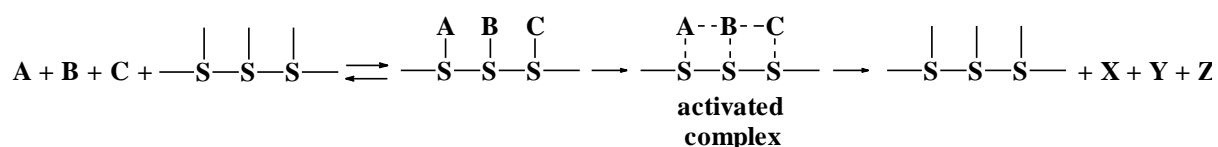
Table 1 The results of testing the quality of used cooking oil before and after purification using sago pulp activated charcoal

Parameter	Used cooking oil before purification	Used cooking oil after purification	SNI 01-3741-2013
Smell and taste	Fishy and unpleasant	Fragrant and delicious	Normal
Color	Blackish brown	Yellow	Normal
Water content (%)	2,35	0,13	Max. 0,15
Acid number (mg KOH/g fat)	2,95	1,10	Max. 0,60
Level of FFA(%)	2,75	1,57	Max. 0,30
Turbidity (NTU)	8,15	2,45	-

The results of testing the quality of used cooking oil above shows that the quality of used cooking oil increases after purification. This is shown by the decrease in turbidity of used cooking oil from 8.15 NTU to 2.45 NTU. In addition, the unpleasant smell of used cooking oil has become fragrant and delicious. Similarly, the blackish brown color of used cooking oil has also turned

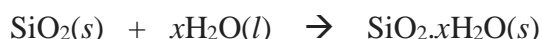
yellow. This happens because the particles that cause turbidity, unpleasant fishy smell, and blackish brown color on used cooking oil have been absorbed by the adsorbent of sago pulp activated charcoal. This adsorption process can occur because the results of the analysis by the Brunauer-Emmett-Teller (BET) method show that the sago pulp activated charcoal adsorbent has a surface area of 174.81 m²/g. According to Syauqiah et al. (2011), one of the factors influencing this adsorption process is the surface area of the adsorbent. The more surface area of the adsorbent, the more substances can be adsorbed.

From the data in Table 1 above shows that the water content of used cooking oil decreases from 2.35% before refining to 0.13% after purification. According to Langmuir-Hinshelwood in Laidler and Meiser (1995), if a solid surface catalyzes a process involving a reaction between three molecules adsorbed on the surface site of the adsorbent, one of the possible mechanisms is



where S represents the surface site of the adsorbent, A, B, and C representing the adsorbed substance, and X, Y, and Z represent the reaction products. In the first stage the three molecules A, B, and C become adsorbed on the surface site of the neighboring adsorbent. Then the reaction occurs through an activated complex to produce reaction products.

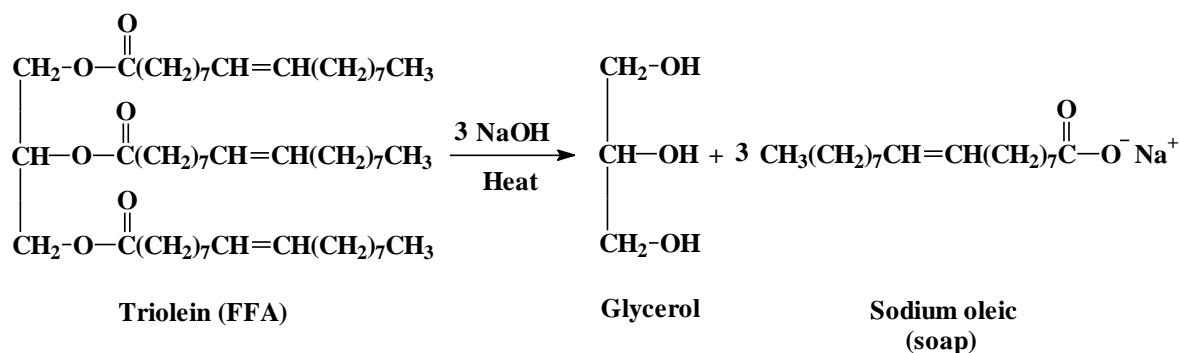
The results of X-ray Fluorescence (XRF) analysis showed that the highest component of metal oxide compounds in sago pulp activated charcoal was silica, SiO₂, which was 8.31%. Silica is a porous solid, this porous structure is related to surface area. The smaller the size of the pores, the more surface area so that the ability of adsorbents to adsorb increases. According to Atkins and Beran (1992) silica has a very large surface area (around 700 m²/g) and this is useful as a drying material, because silica can absorb water molecules to form silica gel deposits.



From the data in Table 1 above it also shows that the acid number of used cooking oil decreases from 2.95 mg KOH/g fat before refining to 1.10 mg KOH/g fat after purification. Similarly, levels of free fatty acids (FFA) decreased from 2.75% before refining to 1.57% after purification. However, both acid numbers and FFA used cooking oil after purification have not met the SNI 01-3741-2013 quality standard, which is a maximum of 0.6 mg KOH/g fatty acid number and a maximum of 0.30% FFA levels. If the FFA level is <0.50%, the process of making ethyl ester can only go through the transesterification stage. Conversely, if the FFA level is >0.50%, then the process of making ethyl ester must go through two stages, namely esterification and transesterification (Hashatan et al., 2012). From the analysis of the FFA levels of used cooking oil after purification above, it shows that used cooking oil has not met the requirements to be used as raw material for making ethyl ester with the transesterification stage. Therefore, the process of making ethyl ester must go through two stages, namely esterification and transesterification.

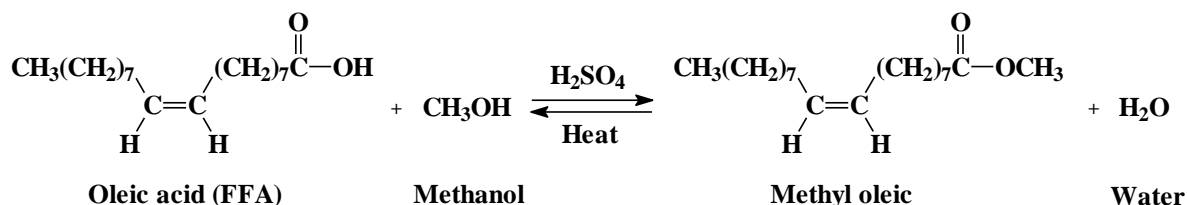
Esterification

Used cooking oil contains 84% oleic acid as free fatty acid (FFA) (Ouellette, 2018). The esterification reaction is needed because the FFA level of used cooking oil (1.57%) > 0.50%. If used cooking oil with an FFA level of > 0.50% is directly transesterified with a base catalyst, then triolein or FFA will be hydrolyzed by base (saponification) to form soap and glycerol (Matta et al., 2016).



The formation of large amounts of soap can inhibit the separation of glycerol from methyl esters due to the formation of emulsions during washing.

The method used for making esters is Fischer esterification, which is a condensation reaction of alcohol with carboxylic acid catalyzed by acid (Carey, 1992). Esters were obtained by refluxing oleic acid as free fatty acid (FFA) from used cooking oil with methanol using H₂SO₄ 98% as catalyst.



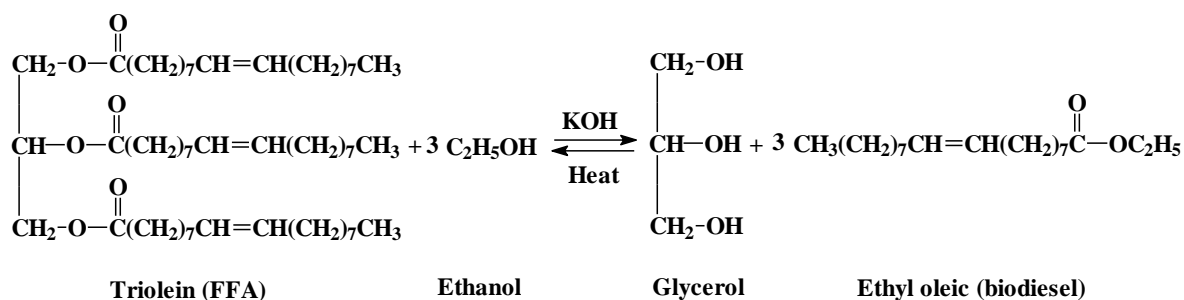
The above equilibrium can be pushed towards the formation of the product by using excess alcohol or carboxylic acid, or by removing water when it is formed. Therefore, the mol ratio of oleic acid (FFA) and methanol used in this esterification reaction is 1: 20. The use of excess methanol aims to avoid the occurrence of back reactions and bind water formed from the reaction, so that more methyl oleic products are formed in the reaction.

The results of esterification of used cooking oil are bifasa shaped liquids. The upper phase is methyl oleic ($\rho = 0.887 \text{ g/cm}^3$) (Alamsyah et al., 2011) and light brown oil (triolein). While the lower phase is water ($\rho = 1,000 \text{ g/cm}^3$) (Wikipedia, 2019), the remaining methanol and acid catalyst are dark brown. Furthermore, FFA levels of oil (triolein) and methyl oleic were tested again. From the results of testing the oil (triolein) and methyl oleic obtained 0.34% FFA levels. This shows that FFA levels of oil (triolein) and methyl oleic have met the requirements to proceed to the transesterification stage.

Transesterification

Transesterification reactions are interconversion reactions of esters, triesters or triglycerides to esters through heating with alcohol and acid or alkaline catalysts (Solomons, 1988). The

transesterification reaction in this study aims to convert triolein (FFA) to ethyl oleic (biodiesel) and glycerol through reaction with ethanol and potassium hydroxide as catalysts.



From the above equilibrium reaction it appears that the ratio of the number of moles of triolein and ethanol is 1: 3. Therefore, to shift equilibrium towards the product, the mole ratio of triolein and ethanol used is 1: 6.

The result of transesterification of used cooking oil is a bifasa shaped liquid. The lower phase is glycerol ($\rho = 1.253 \text{ g/cm}^3$) (Aziz et al., 2018) in blackish brown color. Whereas the upper phase is ethyl oleic ($\rho = 0.902 \text{ g/cm}^3$) which is yellow (83.33% gain), fragrant, and boiling point 77°C .

Nitration

The nitration of used cooking oil is bifasashaped liquid. The upper phase is the remaining water ($\rho = 1,000 \text{ g/cm}^3$) and yellow acid. Whereas the lower phase is the nitro ethyl oleic ($\rho = 1,587 \text{ g/cm}^3$) which is blackish brown (66.67% gain), smells of biodiesel, and boiling point is 80°C .

The results of the interpretation of the FTIR spectrum of ethyl oleic (biodiesel) and nitro ethyl oleic are shown in Table 2. The FTIR spectrum analysis aims to determine changes in functional groups that occur before and after the nitration process. From the FTIR spectrum data in Table 2 shows that ethyl oleic and nitro ethyl oleic both have functional group absorption types that are typical of unsaturated fatty acid esters. This we can see in the presence of three absorption bands of vinylenes or ethenyl ($-\text{CH}=\text{CH}-$) groups, which are medium absorption bands from $=\text{CH}_2$ and $\text{C}=\text{C}$ stretch appearing at 3010.95 and 1635.64 cm^{-1} , and absorption bands the strength of the CH out-of-plane deformation at 730.15 cm^{-1} . Strong absorption bands from the C-H stretch of methylene ($-\text{CH}_2-$) and methyl ($-\text{CH}_3$) groups appear at 2929.09 and 2854.65 cm^{-1} , respectively. While the absorption band is very strong from the ester (RCOOR') group, which is the $\text{C}=\text{O}$ stretch, $\text{C}-\text{O}-\text{C}$ antisym stretch, and $\text{O}-\text{C}-\text{O}$ bend appear on 1743.65 , 1244.09 and 1192.01 , and 671.23 cm^{-1} , respectively.

Table 2. The results of interpretation of the FTIR spectrum of ethyl oleic (biodiesel) and nitro ethyl oleic

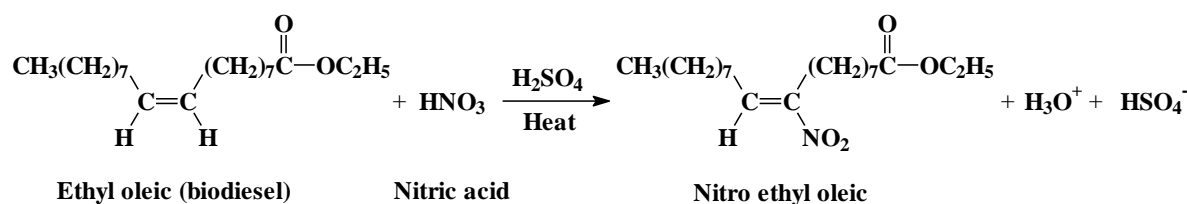
No	Frequency (cm ⁻¹) and Intensities		Frequency Ranges (cm ⁻¹)*	Group or Class	Type of Vibrations
	Ethyl oleic (biodiesel)	Nitro ethyl oleic			
1	3010.95 (m)	3010.95 (m)	3040 - 3010 (m)	Vinylene or Ethenyl -CH=CH-	=CH ₂ stretch
2	1635.64 (m)	1635.64 (m)	1665 - 1635 (m)		C=C stretch
3	730.15 (s)	730.15 (s)	730 - 665 (s)		CH out-of-plane deformation
4	-	1550.77 (vs)	1570 - 1550 (vs)	Nitro -NO ₂	NO ₂ stretches in aliphatic nitro compounds
		1365.60 (vs)	1380 - 1360 (vs)		
5	-	835.13 (s)	920 - 830 (s)		C-N stretch
6	-	630.75 (m)	650 - 600 (m)		NO ₂ bend in aliphatic compounds
7	2929.09 (s)	2929.09 (s)	2940 - 2920 (s)	Methylene -CH ₂ -	C-H stretch
8	2854.65 (s)	2854.65 (s)	2970 - 2850 (s)	Methyl -CH ₃	C-H stretch
9	1743.65 (vs)	1743.65 (vs)	1765 - 1720 (vs)	Esters RCOOR'	C=O stretch
10	1244.09 and 1192.01 (vs)	1244.09 and 1192.01 (vs)	1290 - 1180 (vs)		C-O-C antisym stretch
11	671.23 (s)	671.23 (s)	675 - 575 (s)		O-C-O bend

°Notes: vbr = very broad; vs = very strong; v = variable; s = strong; m = medium; w = weak.

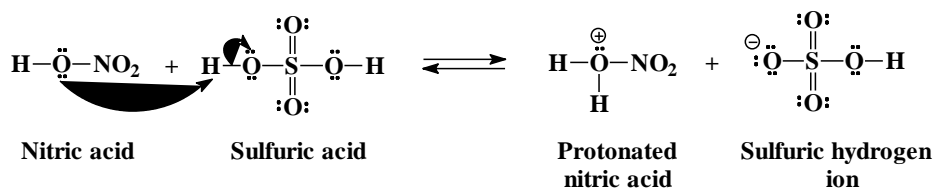
*Sources: (Kemp, 1991); (Lambert et al., 2011); (Sastrohamidjojo, 1992)

The FTIR spectrum data in Table 2 also shows that there are several different nitro ethyl oleic absorption bands from ethyl oleic. This we can see with the presence of very strong absorption bands from the NO₂ stretches in aliphatic nitro compounds that appeared at 1550.77 and 1365.60 cm⁻¹. Whereas the strong absorption bands that appear at 835.13 cm⁻¹ and moderate at 630.75 cm⁻¹ come from the C-N stretch and NO₂ bend in aliphatic compounds, respectively. The appearance of these four absorption bands on the reaction product shows that the nitration reaction of ethyl oleic has occurred. These absorption bands are also in accordance with the results of research from Abdullah et al (2010; 2016), and Canoira et al (2007).

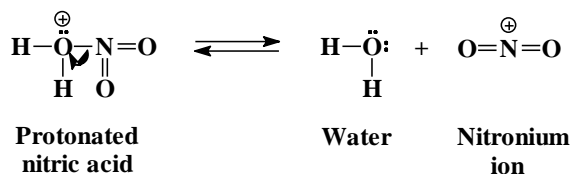
Based on the interpretation of the FTIR spectrum above, we can write down the possibility of the reaction mechanism for the formation of nitro ethyl oleic compounds as follows. Nitration reaction is the reaction of the entry of a nitro group into a ethyl oleic using a mixture of reactants of concentrated nitric acid and concentrated sulfuric acid.



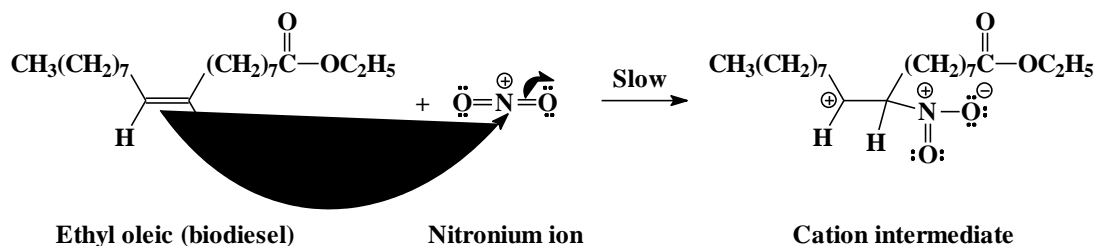
Concentrated sulfuric acid increases the rate of the reaction by increasing the concentration of the electrophile, the nitronium ion (NO_2^+). The mechanism of the nitration reaction begins with nitric acid acts as a base and accepts a proton from the stronger acid, namely sulfuric acid.



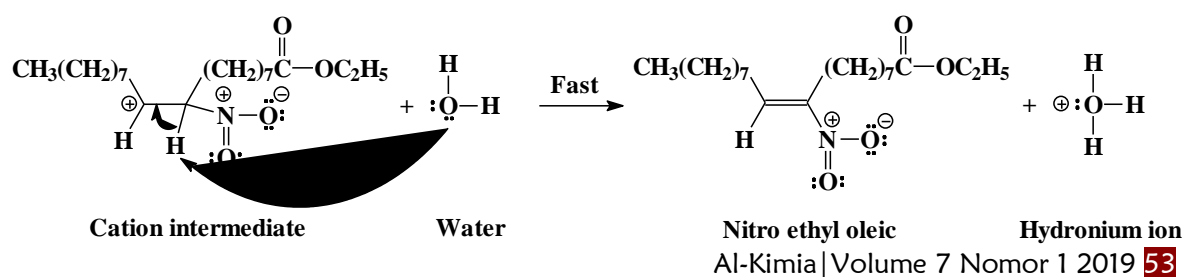
Furthermore the protonated nitric acid dissociates and produces a nitronium ion as reactive electrophile.



Then the electrophilic nitronium ion reacts with the nucleophilic C=C from ethyl oleate to form intermediate cations.



In the final stage the water functions as a base to remove the proton from the $sp^3\text{C}$ bearing the nitro group and reforms the C=C and becomes nitro ethyl oleic.



4. CONCLUSION

Conclusion

Nitro ethyl oleic can be synthesized from used cooking oil through four stages, namely the stages of purification, esterification, transesterification, and nitration. From the purification stage of used cooking oil with sago pulp activated charcoal adsorbent is obtained fragrant and delicious pure cooking oil, yellow with water content of 0.13%, acid number 1.10 mg KOH/g fat, FFA level of 1.57%, and turbidity of 2.45 NTU. From the esterification stage was obtained methyl oleic ($\rho = 0.887 \text{ g/cm}^3$) and oil (triolein) in light brown with FFA level of 0.34%. From the transesterification stage was obtained ethyl oleic (83.33% acquisition) in the form of yellow liquid ($\rho = 0.902 \text{ g/cm}^3$), fragrant, and boiling point 77°C . From the nitration stage was obtained nitro ethyl oleic (66.67% acquisition) in the form of blackish brown liquid ($\rho = 1.587 \text{ g/cm}^3$), typical of biodiesel with a boiling point of 80°C .

Suggestion

Further research is needed on the quality of the ethyl ester nitrate additive from used cooking oil and testing this additive on a single cylinder engine.

Acknowledgements

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