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The Synthesis of Methyl Ester Nitrate from Ketapang Seed Oil (Terminalia catappa L.)

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Abstract
The synthesis of methyl ester nitrate (MEN) from ketapang oil (*Terminalia catappa* L.) have been carried out. This study aims to determine the yield and the characteristics of MEN. In this study, ketapang seed oil was obtained from the soxhlet extraction process followed by an evaporation process to separate the oil from the solvent. MEN can be produced from ketapang seed oil by esterification to convert all of FFA became ester, followed by transesterification that intended to produce ester from triglycerides and nitration that is reaction of esther and HNO_3 to create MEN. Evaporated oil is esterified using methanol with a mole ratio of oil: methanol (1: 6), then the transesterification process using methanol with a mole ratio (1:15) gives a yield of 86%. The transesterification product was then nitrated using HNO_3 and H_2SO_4 for 4 hours with a yield of 83%. Characterization of methyl ester using GC-MS characterization showed the presence of methyl palmitoleate (C17H32O2), methyl palmitate ($C_{19}H_{34}O_2$), methyl oleate ($C_{19}H_{36}O_2$), methyl 13-octadecanoic ($C_{19}H_{36}O_2$), methyl stearate ($C_{19}H_{38}O_2$), and methyl 18-nonadecanoic ($C_{21}H_{42}O_2$). Characterization MEN using a FTIR spectrophotometer showed the presence of a C-ONO_2 group at wave number 1550 cm^{-1}, NO_2 group at wave number 1365 cm^{-1} and a C-N group at wave number 1118 cm^{-1}. It shows that MEN can be synthesized from ketapang seed oil.

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INTRODUCTION

The quality of the world's crude oil is currently decreasing along with the lack of fossil fuel reserves characterised by an increase in density, sulphur content, carbon content, residual fraction and a decrease in the percentage of light fraction and cetane number. High sulphur content will cause corrosion due to the formation of acids produced from sulphur oxides and water in cold or wet conditions (Rufaida & Waldjinah, 2012). Incomplete combustion that results in the absence of CO_2 and H_2O can cause exhaust emissions that are very harmful to health. Low quality diesel oil is indicated by a cetane number that is less than 48 (Musta et al., 2021). Based on this, improving the quality of diesel fuel with the approach of increasing the cetane number value needs to be done by adding an additive (Aufar & Hendra, 2017). Additives are compounds that are added in small volumes to certain materials, including diesel fuel to increase the cetane number value (Astam et al., 2019). Additives for diesel fuel have many uses and effects as they can improve engine performance and reduce exhaust emissions (Saputra et al., 2013).

One of the synthetic additives that has been produced commercially is Ethyl Hexyl Nitrate (EHN). Nasikin in Musta et al. (2021) stated that the disadvantage of EHN is that the basic material used to synthezise the compound comes from non-renewable materials, namely petroleum (petrochemicals). In addition, the price of EHN is relatively expensive because the synthesis path is quite long. Therefore, alternative sources of raw materials are needed for the synthesis of diesel additives from vegetable oils, one of which is by nitrating methyl ester (ME) or biodiesel.

The source of raw materials that can be used as ME is ketapang seed oil (*Terminalia catappa* Linn.) (Rasyid & Nasir, 2020). Ketapang is a type of plant that can grow well in tropical climates such as Indonesia (Listiani et al., 2024). Research by Ariyani et al. (2020) stated that ketapang seeds contain 50%-60% oil. Napitupulu et al. (2021) in their research said that the composition of fatty acids that make up triglycerides consists of palmitic acid (35.63%), palmitoleic (8.6%), stearic (4.3%), oleic (38.0%) and linoleic (21.0%). The high content of palmitic acid in ester form is similar to the palmitic acid content of palm oil. The fatty acid content of ketapang seeds has the potential to be utilised as a ME (Napitupulu et al., 2021).

Research related to ME nitrate from vegetable oils that have been carried out include kapok seed oil (Cahyono & Tjahjani, 2014), coconut and jatropha oil (Aufar & Hendra, 2017), waste cooking oil (Nascimento et al., 2022), palm oil (Mardawati et al., 2019), nyamplung seed oil (Astam et al., 2019), palm oil liquid waste (Arita et al., 2020) and mahogani seed oil (Musta et al., 2021). All of that publications have a different oil source with this research.

METHOD

Sample Preparation

Ketapang fruit samples were obtained from Lakansai Village, North Kulisusu District, North Buton Regency, Southeast Sulawesi. The ketapang fruit was separated between the seeds and the fruit shell. The seeds were then aerated at room temperature for two to three weeks until they were completely dry (to a constant weight). Furthermore, ketapang seeds were mashed using a smoothing machine (blender) so as to obtain fine ketapang seeds (ketapang seed powder) (Muderawan & Daiwataningsih, 2016).

Determination of Free Fatty Acid (FFA) Content of Ketapang Seed Oil

7.5 g of Ketapang seed oil was weighed and put into an Erlenmeyer flask and 50 mL of 95% neutral alcohol was added which had been heated using reverse cooling while stirring until homogeneous. Next, 2-3 drops of phenolphthalein indicator were added and then titrated with 0.5 N KOH. The addition of KOH caused the phenolphthalein indicator to react by changing color to pink. The equation for calculating the FFA percentage is (Ilyas et al., 2023):

$$\%FFA = \frac{A \times N \times MR}{m} \times 100\%$$

A = Amount of KOH used for titration (mL)
N = Normality KOH
MR = Molecular mass of oil (g/mL)
m = mass of oil sample (gram)

Isolation the sample

The soxhletation process was carried out by making a sleeve with a sample weight of 125 g and measuring the n-hexane solution using a 250 mL measuring cup in a ratio of 4:1 of the

sample weight. The n-hexane solution was put into a 500 mL round bottom flask. Then, the solution was heated at 60-80^{0}C for 8 hours, cooled and filtered with filter paper. Next, it was evaporated to obtain pure oil and solvent. The pure oil obtained was determined by density using a pycnometer. The empty pycnometer was weighed, then the sample were inserted into the empty pycnometer, then the weight of the pycnometer containing the sample were weighed (Saputra et al., 2017).

Esterification

The esterification process was carried out by reacting methanol and evaporated ketapang oil with a mole ratio (1:15) and adding 1% sulfuric acid (H_2SO_4) as a catalyst. The evaporated oil was put in a two-neck flask that has been equipped with a condenser and heater. Furthermore, the sample was heated at a temperature of 65^{0}C while slowly adding methanol and sulfuric acid (H_2SO_4) while stirring using a magnetic stirrer for a hour. The reaction mixture was cooled then put into a separatory funnel and allowed to stand for 24 hours to form two phases. The lower phase was taken and then it washed with warm distilled water until the pH was neutral (Mohan et al., 2016).

Transesterification

Esterified oil and methanol (1:6) were reacted with a KOH catalyst of 1% (w/v) by weight of esterified oil (Damayanti & Bariroh, 2013). The esterified oil was put into a two-neck flask equipped with a condenser and heater while stirring. Next, the sample was heated at 60^{0}C while slowly adding methanol and KOH while stirring using a magnetic stirrer for a hour. Furthermore, the reaction was stopped and cooled, then put into a separating funnel and allowed to stand for 24 hours to form two phases. The upper phase is taken and then washed with warm distilled water until the pH is neutral. Moreover, the ME was heated at 105-110^{0}C to remove the remaining water that was still mixed into the ME (Nurliana et al., 2021).

Nitration

A total of 1.5 mL of nitric acid was reacted with 15 mL of sulfuric acid in a 250 mL round bottom flask. Sulfuric acid and nitric acid that had been mixed were put into a 250 mL round bottom flask then 16.5 mL of ME was added to the nitric and sulfuric acid solution drop by drop, then the solution was refluxed for 4 hours at 11℃-15^{0}C with stirring using a magnetic stirrer at 200 rpm. The resulting product was put into a separating funnel containing 200 mL of water and 20 mL of diethyl ether, then shaken and allowed to stand until two phases were formed. The upper phase (MEN) and lower phase (water and acid residue) are separated to obtain the MEN whose yield will be calculated (Abdullah et al., 2017). The test for the presence of a nitro group in a sample of MEN was carried out by reacting $FeSO_4$ in a tilted test tube and then dripping with H_2SO_4 slowly (Ratna & Andriyatie, 2017).

Gas Chromatogaphy and Mass Spectrometry (GC-MS) Analysis

Identification of compounds contained in ME using GC-MS HP-5MS UI with specifications of equipment conditions, namely column Rt 2 30 metres long with a diameter of 0.25 mm, film thickness 0.25 µm. The carrier gas used is Helium, the type of Electron Impact (EI) 70 ev, column temperature 325/350^{0}C, injection temperature 260^{0}C, pressure 13 kPa, column flow 0.55 mL/min. Data interpretation was then performed (Diningrat et al., 2018).

Fourier Transform Infrared (FTIR) Spectrophotometer Analysis

The composition of the constituent compounds of ME and the synthesis of MEN from ketapang seed oil was identified using an FTIR spectrophotometer with a wavelength of 4000-400 cm^{-1} with the specifications of the equipment conditions, namely Scan: 32 sec/scan, resolution: 4 and pressure: 80 Torr. Furthermore, the interpretation of IR spectrum data

obtained by comparing the absorption of functional groups in the IR spectrum of the sample with the wave number of the literature or library (Musta et al., 2021).

RESULTS AND DISCUSSION

Preparation of ketapang seed oil

The initial preparation includes several stages, namely the separation of seeds from the shell, then the seed washing process which aims to clean the seeds from impurities. The next stage was the drying process which was carried out by air-drying air at outdoor temperature for several days until ketapang seeds were obtained which have a constant weight when weighed several times. The drying process aims to reduce the water content in ketapang seeds. Furthermore, the ketapang seed was smoothed which aims to reduce the particle size of the sample. The finer the sample, the more extraction results will be obtained (Ardyanti et al., 2020).

Ketapang oil in this study was obtained by soxhlet extraction method followed by separation process using evaporation method. The advantages of the soxhletation method include being able to extract more oil, less solvent used and shorter extraction time. The effectiveness of extraction of a compound by a solvent is highly dependent on the solubility of the compound in the solvent, according to the principle of a compound will be dissolved in a solvent with the same polarity. In this study, the solvent used to extract ketapang oil was n- hexane (Ariyani et al., 2020). The volume ratio between the crushed ketapang seed and the solvent was 1:4 with an extraction time of 8 hours with temperature conditions of 65℃-70℃ (Maslukhah et al., 2016). Ketapang seed oil after evaporation was yellow as shown in Figure 1.

The following stage was the determination of ketapang seed oil density and determination of FFA content and the resulting density was 0.881 g/mL. According to research by Warsito et al. (2013) high quality ketapang oil is indicated by a high density value which is above 0.8 g/mL. While the FFA content of ketapang oil is 47%, when compared with the results of FFA levels from the research of Marjenah & Putri (2017) which was 58%, there is a slight difference. The difference was caused by several factors including extraction time, particle size and extraction temperature (Tambun et al., 2016). In addition, it can also be caused by differences in varieties, age of harvest mass, soil pH and environmental conditions of ketapang plant growth (Kartika et al., 2017). The determination of FFA levels will be a parameter for the next stage of ME synthesis. Based on the Indonesian National Standard (SNI) regulations, FFA levels should not be more than 5% (Melwita et al., 2014). FFA levels of more than 5% will affect the transesterification process. If ketapang oil has high FFA levels (>5%) directly transesterified with a base catalyst, the FFA will react with the catalyst to form soap. The formation of soap in large enough quantities can inhibit the separation of glycerol from ME and result in the formation of emulsions during the washing process.

Esterification

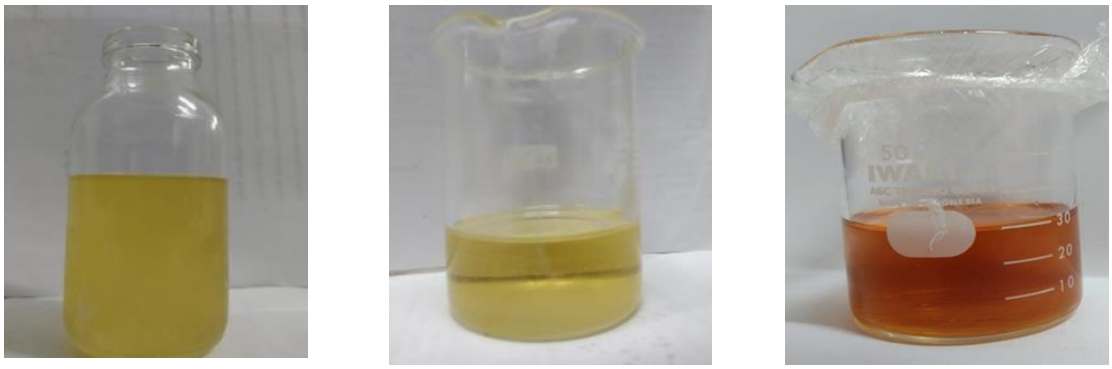
Esterification was a process of converting FFA into ME. The esterification process was a chemical reaction that is reversible so that in this process excess methanol must be used in order to shift the reaction equilibrium towards the product to get maximum results. In this study, 1% H_2SO_4 was used as a catalyst. Acid catalysts were needed to accelerate the reaction in oils that have FFA The temperature used in this process is 50℃-65℃. The higher the temperature used, it will cause the molecular movement to accelerate or the kinetic energy possessed by the reagent molecules is greater so that the collision between the reagent

molecules also increases (Trisunaryanti & Haryadi, 2017). The reaction lasted for a hour while stirring using a magnetic strirer at 400 rpm.

The following stage was the esterification product left in a separating funnel for 24 hours. This stage aims to separate the ME (lower phase) from the remaining methanol and catalyst (upper phase). Furthermore, the lower phase was neutralised by dilution using warm distilled water. The addition of warm distilled water in the neutralisation process will dissolve the remaining solvent and catalyst. The neutralisation process was stopped when the pH neutral, which was indicated by the colour of the distilled water in the oil turning clear. The neutralisation results were then heated at 105℃ to evaporate the remaining water. The top phase (ME) resulting from esterification had a clear yellow colour (Figure 1).

Transesterification

Transesterification is the reaction of forming triglycerides, diglycerides, and monoglycerides modified into glycerol. The transesterification reaction is a reversible reaction that can take place in two directions, so that the provision of excess methanol can increase the yield of ME (Trisunaryanti & Haryadi, 2017). The results obtained from the transesterification process are then allowed to stand for 24 hours so that two phases are formed where the lower phase is the remaining catalyst and glycerol while the upper phase is the ME obtained. The upper phase or ME obtained is continued with the washing process. Washing aims to remove impurities that were still present in the methyl ester. The impurities in question are the remaining catalyst and glycerol as well as the remaining unreacted alcohol. Washing was done by adding warm water about 50% of the total volume of oil to be washed. The resulting ME are clear yellow in colour, clearer than the esterification results and smell good as shown in Figure 1.



a b c

Figure 1. The product of a) Ketapang Seed Oil, b) ME, and c) MEN

Nitration

Nitration is a process of including nitro groups in ME compounds. The nitro group in this study was obtained from nitric acid. The nitration process includes two processes, namely the preparation of nitro compounds and the preparation of nitrate esters. Nitro ions can be generated from the interaction of nitric acid with sulfuric acid as a catalyst. Through nitration of ME, the amount of molecular oxygen of ME components increases so that ME have more oxygen which is needed in the perfection of the combustion process (Abdullah et al., 2017). The nitration process is carried out by reflux method for 4 hours at a temperature of 15℃-20℃. The nitration reaction is carried out at low temperatures because at high temperatures there may be compounds that are oxidised by nitric acid (Nasikin & Ade, 2003). Nitric acid and sulfuric acid were first put in a two neck flask and then added ME drop by drop while stirring using a magnetic stirrer at 200 rpm. The reaction temperature was kept stable by using ice cubes around the round bottom flask. The product resulting from the nitration reaction was dark brown in colour as shown in Figure 1.

The next process is neutralisation of MEN, because the resulting MEN still has an acidic pH level at pH 4-5, it is neutralised by adding diethyl ether and warm distilled water and then adding anhydrous CuSO4 to dry the remaining distilled water. MEN produced after neutralisation is light yellow and clear. The average percentage of MEN yield obtained was 86%.

Identification of ME using GC-MS

Based on the interpretation of the fragmentation pattern of the mass spectrum of the standard compound, it is concluded as shown in Table 4.2 where the highest abundance is found in peak 3, namely stearic acid. In ketapang seed oil, the compound that has the highest abundance is oleic acid but after becoming a ME the abundance shifts to stearic acid. Methyl stearate appeared at a retention time of 36.142 with an abundance of 46.09%. This is because the conversion of oil into ME occurs maximally in stearic acid. The chromatogram of ketapang seed ME can be shown in Figure 2. as follows:

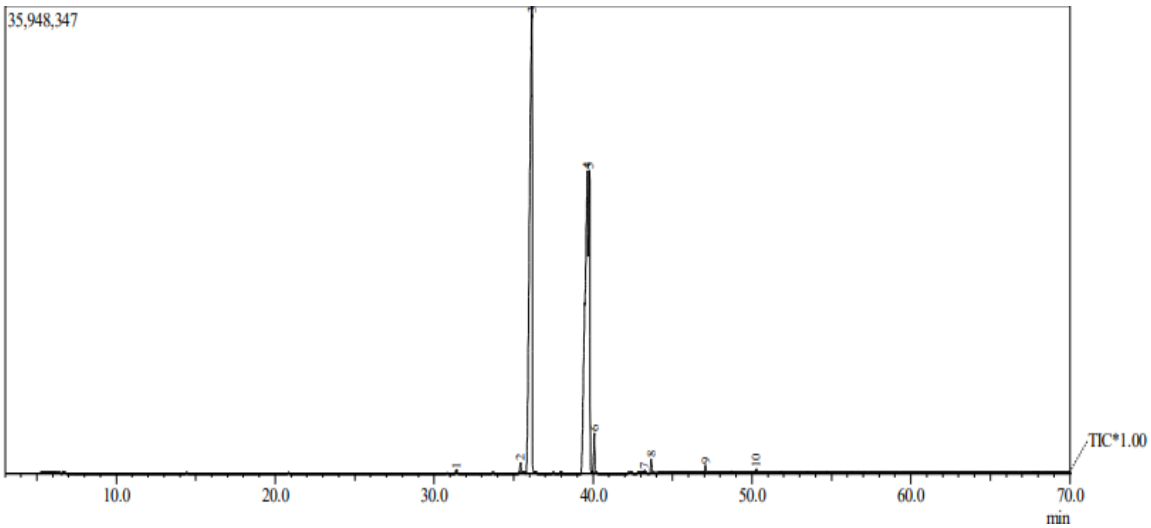


Figure 2. Chromatogram of ME

Table 1. Compounds of ME characterisation results

Peak	Compounds	Molecule formula	% Area	Retention time (minutes)
2	Methyl palmitoleate	$C_{17}H_{32}O_2$	0.47	35.433
3	Methyl palmitate	$C_{19}H_{34}O_2$	46.09	36.144
4	Methyl oleate	$C_{19}H_{36}O_2$	33.95	39.645
5	Methyl 13-oktadecanoate	$C_{19}H_{36}O_2$	16.65	39.779
6	Methyl stearate	$C_{19}H_{38}O_2$	1.65	40.081
8	Methyl 18-methylnonadecanoate	$C_{21}H_{42}O_2$	0.52	43.660

Initial Identification of Nitro Groups

The test for the presence of nitro groups in the MEN sample was carried out by reacting $FeSO_4$ and MEN in a tilted test tube and then slowly dropping H_2SO_4. The reaction equation is as follows:

$Fe^{2+} \longrightarrow Fe^{3+}$ Fe oxidation

$NO_2 \longrightarrow NH_3$ N reduction

$Fe^{2+} \longrightarrow Fe^{3+} + e$

$NO_2 + 7H^+ \ 7e \longrightarrow NH_3 + 2H_2O$

+

$7Fe^{2+} + No_2 + 7e \longrightarrow 7Fe^{3+} + NH_3 + 2H_2O$ x2

$14\ Fe^{2+} + 2NO_2 + 14H^+ \longrightarrow 7Fe^{3+} + 2NH_3 + 4H_2O$

The overall reaction equation can be written as follows:

$14FeSO_4 + 2NO_2 + 7H_2SO_4 \rightarrow 7Fe_2(SO_4)_3 + 2NH_3 + 4H_2O$

The formation of a reddish-brown ring at the surface boundary of the two liquids indicates the presence of NO^{2+} ions. This reaction is an oxidation-reduction reaction in which the compound $7Fe_2(SO)_3$ is formed which has a reddish brown colour (Ratna & Andriyatie, 2017). The results of the nitro group identification test are shown in Figure 3.

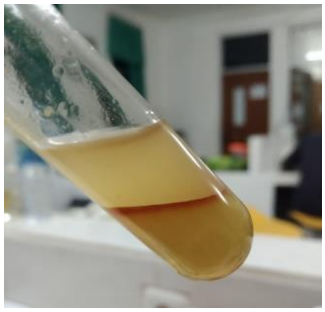


Figure 3. Identification of the nitro group

Identification Using FTIR Spectrophotometer

Analysis using an FTIR spectrophotometer aims to determine the presence of groups in various synthesis results. The FTIR spectrum of ketapang oil shows that there are functional group absorptions including C-CH_3 group absorption at wave numbers 2924.09 cm-1 and 2854.65 cm-1, C=O group absorption at wave number 1743.65 cm-1, CH_2 group absorption at wave number 1435.04 cm-1, at wave number 1373.32 cm-1 showing the vibration of O-C O bond and O-H group absorption. at wave number 3471.87 cm-1. The interpretation results show that there are functional group absorptions that are characteristic of fatty acid compounds that make up ketapang seed oil. The cluster absorption found in ketapang seed oil is in accordance with the functional group absorption in the research of Ravensca et al. (2017) and Sumartono et al. (2018).

The FTIR spectrum of ME shows that there are functional group absorptions including C-CH_3 group absorption at wave numbers 2924.09 cm^{-1} and 2854.65 cm^{-1}, C=O group absorption at wave number 1743.65 cm^{-1}, CH_2 group absorption at wave number 1458.18 cm^{-1}, O-C-O bond at wave number 1365.60 cm^{-1}. The interpretation results show that there are functional group absorptions that are characteristic of ME compounds. The cluster absorption found in ME is in accordance with the absorption of functional groups in the research of Ravensca et al. (2017) and Sumartono et al. (2018).

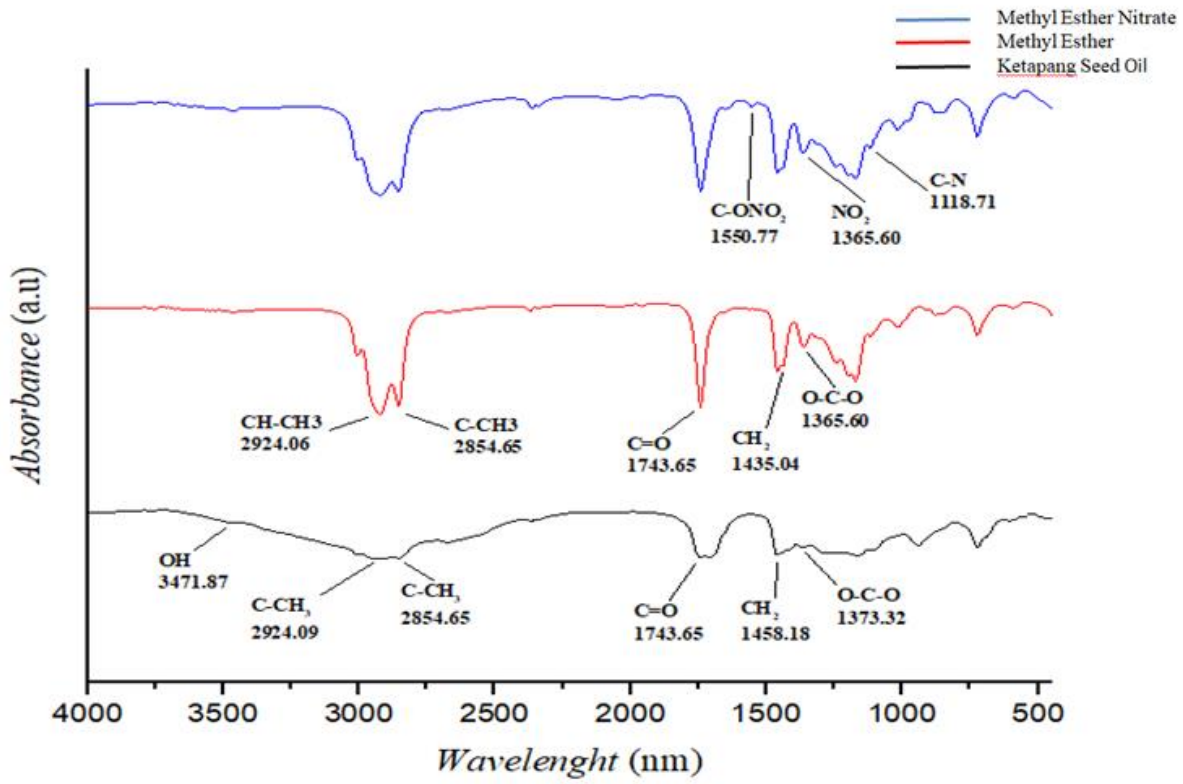


Figure 4. FTIR spectrum

Table 2. FTIR Spectrum Interpretation Result Data

No.	Wave number (cm-1)						Vibration Type
	Ketapang Seed Oil	Reference of Ketapang Seed Oil	ME	Reference of ME	MEN	Reference of MEN	
1.	3471.87	3550-3200	-	-	-	-	O-H
2.	2924.04 & 2854.65	3000-2800	2924.04 & 2854.65	3000-2800	-	-	C-CH3
3.	1743.65	1740-1720	1743.65	1740-1720	-	-	C=O
5.	-	-	-	-	1550	1545-1660	C-ONO2
6.	-	-	-	-	1365	1300-1500	NO2
7.	-	-	-	-	1118	1180-1360	C-N
8.	1435.04	1470-1450	1458.18	1470-1458	-	-	CH2
9.	1373.32	1320-1210	1365.60	1320-1210	-	-	O-CO
10.	1033.85	-	1018.41	-	-	-	C-OH

Figure interpretation of the FTIR spectrum of MEN, showing that there are several shifts in the peak spectrum between the FTIR results of MEN in contrast to the FTIR spectrum of methyl ester, including at wave number 1550 cm^{-1} which is predicted as the presence of C-ONO_2 bonds and C=C absorption in the ME compound, as well as at wave number 1365 cm^{-1} which is predicted as NO_2 bonds with C-N at wave number 1118 cm^{-1}. The wave number is in accordance with the ONO_2 wave number formed in the research conducted by Abdullah et al. (2017).

Based on research conducted by Canoira et al. (2007) and Diop et al. (2019) revealed that MEN is formed from the binding of nitro group and nitrate group on the double bond of methyl ester. Therefore, in this study, it can be predicted that the reaction mechanism that occurs can be seen in Figure 6. as follows:

Reaction of electrophilic (+NO_2) subsitution

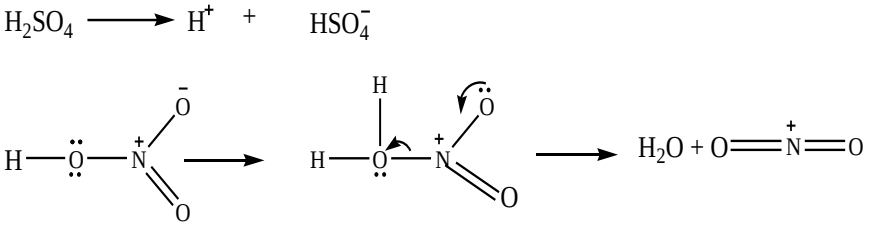
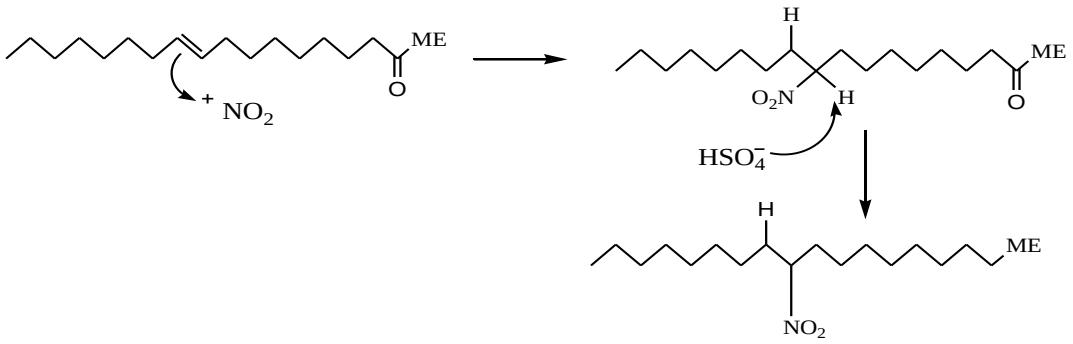
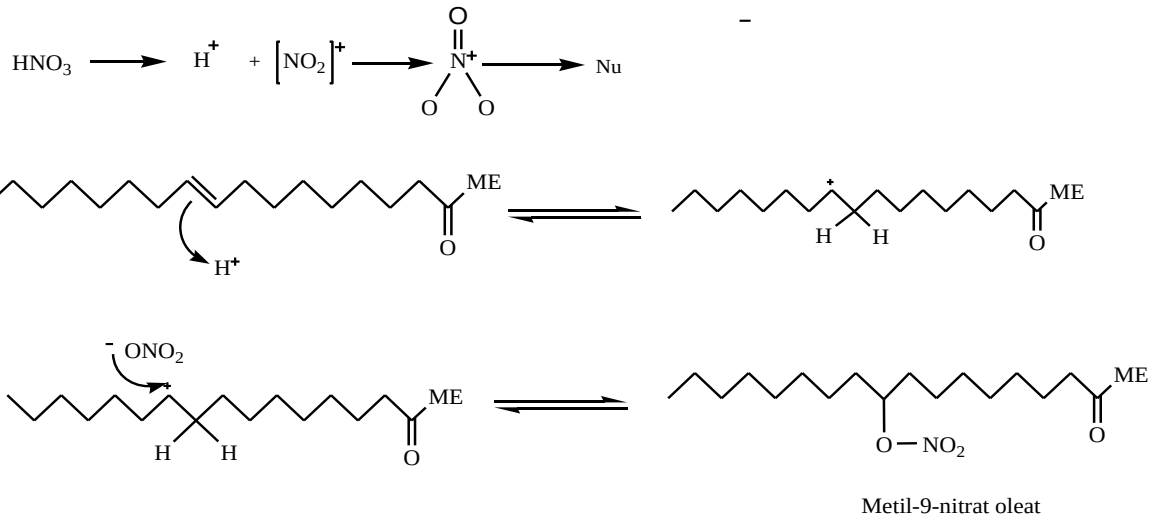


Figure 5. Stage 1. Electrophilic Formation



Metil-9-nitro oleat

Figure 6. Stage 2. Electrophilic assault



Metil-9-nitrat oleat

Continued…

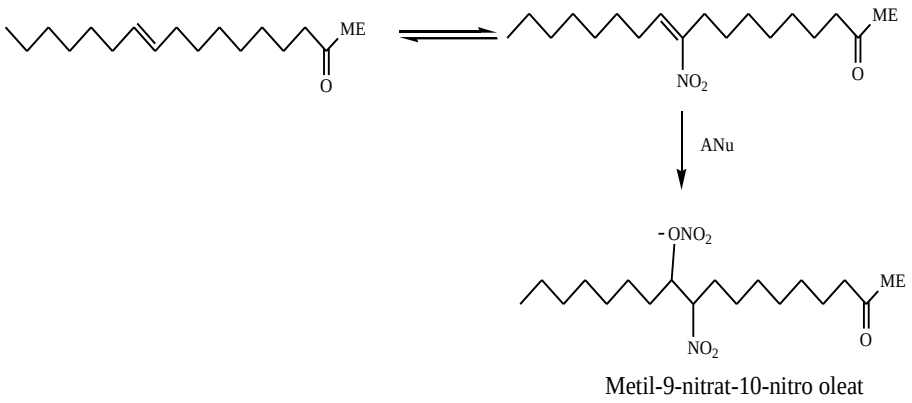


Figure 7. Reaction of nukleophilic (ONO_2-) addition

CONCLUSION

Based on the results of research and discussion in this study, it was concluded that MEN can be synthesized from ketapang seed oil with a nitration yield percentage of 86%. Characterization of ME using GC-MS characterization showed the presence of methyl palmitoleate ($C_{17}H_{32}O_2$), methyl palmitate ($C_{19}H_{34}O_2$), methyl oleate ($C_{19}H_{36}O_2$), methyl 13-octadecanoic ($C_{19}H_{36}O_2$), methyl stearate ($C_{19}H_{38}O_2$), and methyl 18-nonadecanoic ($C_{21}H_{42}O_2$). Characterisation of ME with FTIR showed C-CH_3, C=O, CH_2, O-C-O, C-OH, and C-H groups at 2924.04 cm^{-1}; 2854.65 cm^{-1}; 1743.65 cm^{-1}; 1458.18 cm^{-1}; 1365.60 cm^{-1}; 1018.4 cm-1 and 848.68 cm-1, while the MEN showed the presence of $CONO_2$ group at wave number 1550 cm^{-1}, NO_2 group at wave number 1365 cm^{-1} and C-N group at wave number 1118 cm^{-}1. This shows that ketapang oil is potential source for MEN producing.

RECOMMENDATIONS

Based on the results showed that the identification of the ME compound that forms MEN has not been identified in detail because the ME formed is a mixture of various kinds of ME, so our hope for further studies requires purification first on the ME. In addition, further identification of the MEN identification to determine the position of the nitrate group attachment to the ME compound, so that the predicted results of the reaction mechanism can be proven.

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