Automotive Experiences
Vol. 4 No.1 (2021) pp. 44-50
p-ISSN: 2615-6202 e-ISSN: 2615-6636

Research Paper

Synthesis of Bio-additive for Low Sulphur Diesel: Transesterification of Soybean Oil and Ethylene Glycol using K_2CO_3 Catalyst

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https://doi.org/10.31603/ae.4694

Published by Automotive Laboratory of Universitas Muhammadiyah Magelang collaboration with Association of Indonesian Vocational Educators (AIVE)

Article Info
Submitted:
15/02/2021
Revised:
09/03/2021
Accepted:
10/03/2021
Online first:
13/03/2021

Abstract

The desulphurization process of diesel fuel is carried out to reduce the amount of SO_2 emissions that can cause acid rain. However, the desulphurization process in diesel fuel not only removes the sulfur compounds but polyaromatic and polar compounds are also eliminated during this process. The loss of these two compounds can reduce the lubricity properties of diesel fuel. Therefore, it is necessary to add an additive compound that can increase the lubricity properties. In this research, 2-hydroxyethyl ester (HEE) was synthesized as an additive to increase the lubricity of diesel fuel. This compound was synthesized through the transesterification reaction of soybean oil and ethylene glycol with K_2CO_3 as the base catalyst. The composition of the synthesized additives was analyzed using the Gas Chromatography-Mass Spectrometry (GC-MS). Based on the results of GC-MS spectrum analysis, it is known that the 2-hydroxyethyl ester compound has been formed with a yield of 66.5% (relative to the area of the chromatogram peak). The HEE compound obtained is a mixture of 2 hydroxyethyl palmitate, 2 hydroxyethyl linoleate, 2 hydroxyethyl stearate, 2 hydroxyethyl arachidonate, 2 hydroxyethyl nervate, and 2 hydroxyethyl behenate.

Keywords: Low Sulphur diesel; Lubricity improver; Bio-additive; Transesterification

Abstrak

Proses desulfurisasi bahan bakar diesel dilakukan untuk mengurangi jumlah emisi SO_2 yang dapat menyebabkan hujan asam. Namun proses desulfurisasi pada minyak diesel tidak hanya menghilangkan senyawa sulfur tetapi senyawa polyaromatik dan polar juga tereliminasi selama proses ini. Hilangnya kedua senyawa ini dapat mengurangi sifat pelumasan bahan bakar. Oleh karena itu, perlu dilakukan penambahan senyawa aditif yang dapat meningkatkan sifat pelumasan. Pada penelitian ini disintesis 2-hidroksietil ester (HEE) sebagai aditif untuk meningkatkan pelumasan minyak diesel. Senyawa ini disintesis melalui reaksi transesterifikasi minyak kedelai dan etilen glikol dengan K_2CO_3 sebagai katalis basa. Komposisi zat aditif hasil sintesis dianalisis menggunakan Gas Chromatography-Mass Spectrometry (GC-MS). Berdasarkan hasil analisis spektrum GC-MS diketahui telah terbentuk senyawa 2-hidroksietil ester dengan rendemen sebesar 66.5% (relatif terhadap luas puncak kromatogram). Senyawa HEE yang diperoleh merupakan campuran 2 hidroksietil palmitat, 2 hidroksietil linoleat, 2 hidroksietil stearat, 2 hidroksietil arakidonat, 2 hidroksietil saraf, dan 2 hidroksietil behenat.

Kata-kata kunci: Minyak diesel sulfur rendah; Perbaikan pelumasan; Bio-aditif; Transesterifikasi

1. Introduction

The development of the transportation industry was followed by an increase in diesel consumption and it can result more pollutants such as SO_2 gas, which is the main cause of acid rain [1]. Due to the seriousness of air pollution, regulations on limiting the amount of emission of fuel are applied. In Indonesia, the amount of sulphur content in diesel fuel is limited to only 50 ppm [2]. However, the process of reducing sulphur content in diesel fuel can reduce its lubrication properties due to the loss of polyaromatic and polar compounds [1]. Diesel fuel must have good lubrication properties to reduce and prevent wear of components [3]. The addition of an additive to increase lubricity properties in ultra-low sulphur diesel is an alternative solution that can be applied [4].

The lubricity enhancer additive derived from vegetable oil was reported to increase the lubricity properties of low sulphur diesel fuel [5]. However, the use of vegetable oil as an additive for lubricity has immediate drawbacks due to its high viscosity, low oxidative stability, and instability at low temperatures [6, 7]. Modification of vegetable oil triglycerides to form their alkyl esters can solve that problems [5]. These alkyl esters can be synthesized through transesterification reactions of vegetable oils and alcohol compounds, where the catalysts commonly used are acid, base, and enzyme catalysts. The use of alkaline catalysts in this reaction was reported to have a faster reaction time compared to acid catalysts and enzyme catalysts. Two alkaline catalysts that are widely used in synthesizing alkyl esters from vegetable oils are sodium hydroxide (NaOH) and potassium hydroxide (KOH). Although the reaction is faster, there is a problem due to the soap formation during the process of transesterification of vegetable oils [8]. This is because when the hydroxide ions from this catalyst react with alcohol, it can form water in the reaction. This formed water can hydrolyze triglycerides, forming diglycerides and free fatty acids. These free fatty acids can react with potassium or sodium ions to form soap through a saponification reaction [9]. The soap formation is undesirable because it makes difficult the separation and purification steps [8]. An alternative base catalyst that can be used for the transesterification of vegetable oils is potassium carbonate (K_2CO_3). The use of a potassium carbonate catalyst results in a high yield of fatty acid ester and less soap formation than potassium hydroxide and sodium hydroxide [10].

The alkyl ester compounds from vegetable oils qualify as lubricity improver additive because they have a polar group on the head, so they can interact with the metal surface being lubricated and have long-chain carbon in the tail (usually C12-C18) to provide their solubility in diesel fuel [11]. The lubrication ability of this compound depends on the type of raw material, such as the type of vegetable oil and alcohol used. Several researchers previously reported that the fatty acid composition of vegetable oils contributed to the lubricity ability of an alkyl ester. An increase in chain length of fatty acid alkyl ester leads to improvement of its lubricity properties [12]. Besides, the increase in the number of double bonds in the fatty acid chain slightly increases the lubrication ability [13]. Therefore, vegetable oil which is dominant with unsaturated fatty acid content like soybean oil is good for use as raw material for making lubricity-enhancing additives [14, 15]. Methyl esters from soybean oil showed better lubricity ability compared to methyl esters from coconut oil, palm oil, olive oil, and canola oil [15].

Most of the lubrication enhancing additives are fatty acid methyl ester (FAME) and fatty acid ethyl ester (FAEE), which are made by transesterification of vegetable oils with simple monohydroxy alcohols, namely methanol and ethanol [5], [16-18]. Alkyl ester compounds can also be made with polyhydroxy alcohols, such as ethylene glycol to produce 2-hydroxyethyl ester compounds [10]. The presence of this hydroxy group is believed to be able to provide better lubrication capabilities than FAME and FAEE. This is based on the results of research conducted by Sukjit et al. [12], where the more polar the functional groups in lubricant additives can form stronger intermolecular bonds with metal substrates which can improve lubrication properties. However, there is still not much information regarding these hydroxyalkyl esters as lubricating additives in low sulphur diesel fuel.

This study aims to synthesize 2-hydroxyethyl ester by transesterification of soybean oil and ethylene glycol using potassium carbonate as a catalyst. Based on the structural review, this compound has the potential to be used as a lubricity-enhancing additive, because it has a long unsaturated carbon chain and the presence of a hydroxy-functional group from the alcohol chain. The synthesized bio-additive is characterized using Gas Chromatography coupled with a Mass Spectrometer detector (GC-MS).

2. Materials and Methods

2.1. Materials

This research consists of two-stage, such as fatty acid composition analysis of soybean oil and synthesis of 2-hydroxyethyl ester as lubricity additive compound. The soybean oil used in this study has a low acid value of less than 1 mg/KOH and was obtained from the local market. In the analysis of the fatty acid composition of the soybean oil stage, the materials used such as methanol, potassium hydroxide, sodium sulfate anhydrous and n-hexane were pro analysis grade and purchased from Merck. In the synthesis of 2-hydroxyethyl ester compounds, all materials used, such as ethylene glycol, potassium carbonate, ethyl acetate, hydrochloric acid (37%), and anhydrous sodium sulfate are pro-grade analysis and purchased from Merck. The characterization of the 2-hydroxyethyl ester compound was carried out by the GC-MS method, where this compound was dissolved with n-hexane. The n-hexane used was also a pro-grade analysis and was purchased from Meck.

2.2. Experimental

2.2.1. Analysis of soybean oil

The fatty acid composition of soybean oil was analyzed using gas chromatography with a mass spectrometric detector (GCMS-QP2010). The soybean oil sample was converted into its methyl ester before it was analyzed. Synthesis of FAME using a method adapted from Rashid et al., [19]. First, preheated soybean oil and methanol with a molar ratio (1:10) were reacted into a 100 mL three-neck flask. After that, 1% KOH in methanol solution (% mass of oil) was added to the reflux flask. The reaction temperature was set at 60 °C. After two hours, the product formed was transferred into a separating funnel and cooled to room temperature resulting in two separate layers. After the separation of the two-phase, the organic layer (upper layer) was evaporated to eliminate the remaining methanol. The evaporated mixture was washed with distilled water followed by treatment with Na_2SO_4, and filtration.

Qualitative analysis of transesterified soybean oil was carried out using a gas chromatography instrument with a mass spectrometric detector (GCMS-QP2010 SE) and equipped with a capillary column (Rtx-5MS) with a size (30 m/ 0.25 mmID/ 0.25 μm df). The carrier gas was helium with a flow rate of 1.16 mL/min. The temperature program was set at 100 °C (for 4 min) and then was increased to 240 °C at a rate of 3 °C/min and hold for 15 min. finally, the temperature was increased to 250 °C at a rate of 10 °C/min with a final isothermal period of 4.33 min. The injector and interface temperatures were held at 250 °C, respectively, and the run time was 71 min. Soybean FAME sample preparation was carried out by diluting 0.1 mL soybean FAME with 1 mL n-hexane solvent. As much as 1.0 μL of the sample was injected into the instrument using the split mode injection with a ratio of 1:138.7. Mass scanning was carried out in the range 45 - 501 m/z.

2.2.2. Synthesis of 2-Hydroxy Ethyl Ester

The synthesis of 2-hydroxyethyl ester additives adapted the method from Rezende et al., [10] and Costa et al., [20]. First, preheated soybean oil and ethylene glycol (1:10 molar ratio) were reacted into a 100 mL three-neck flask. After that, 7% potassium carbonate (% mole to oil) was added to the reflux. The reaction temperature was set at 150 °C. After 5 hours, the product formed was neutralized with 10% HCl solution followed by extraction with ethyl acetate and transferred to a separating funnel. The organic layer formed (upper layer) was washed with warm water and then dried with anhydrous sodium sulfate. The solvent in the filtrate was evaporated with a rotary evaporator.

Qualitative analysis of HEE product was carried out using a gas chromatography instrument with a mass spectrometric detector (GC-MS QP-2020 NX) and equipped with a capillary column (Rtx-5MS) with a size (30 m/ 0.25 mmID/ 0.25 μm df). The carrier gas was helium with a flow rate of 2.38 mL/min. The temperature program was 70–200 °C (hold for 5 min) at a rate of 8 °C/min and finally increased to 305 °C at a rate of 4 °C/min with a final isothermal period of 20 min. The injector and interface temperatures were held at 290 and 300 °C, respectively, and the run time was 64.25 min. Soybean HEE sample preparation was carried out by diluting 0.1 mL HEE with 1 mL n-hexane solvent. As much as 1.0 μL was injected using split mode injection with a ratio of 1:10. The sample was analyzed using SCAN mode in the mass range of 28 – 1000 m/z.

3. Result and Discussion

3.1. *Results*

3.1.1. *Analysis of soybean oil*

The results of the Gas Chromatography-Mass Spectrometer (GC-MS) analysis showed that there were nine peaks indicating the occurrence of nine fatty acid components in the soybean oil as shown in Figure 1. Identification of those nine peaks using mass spectrum data and mass fragmentation studies. The type of fatty acid and its relative concentration in soybean oil can be seen in Table 1.

3.1.2. *Synthesis of Hydroxy Ethyl Ester (HEE)*

The composition of the synthesized additives was investigated using the GC-MS instrument. The identification of the GC-MS spectrum was carried out referring to mass spectrum data and fragmentation studies, as presented in Table 2.

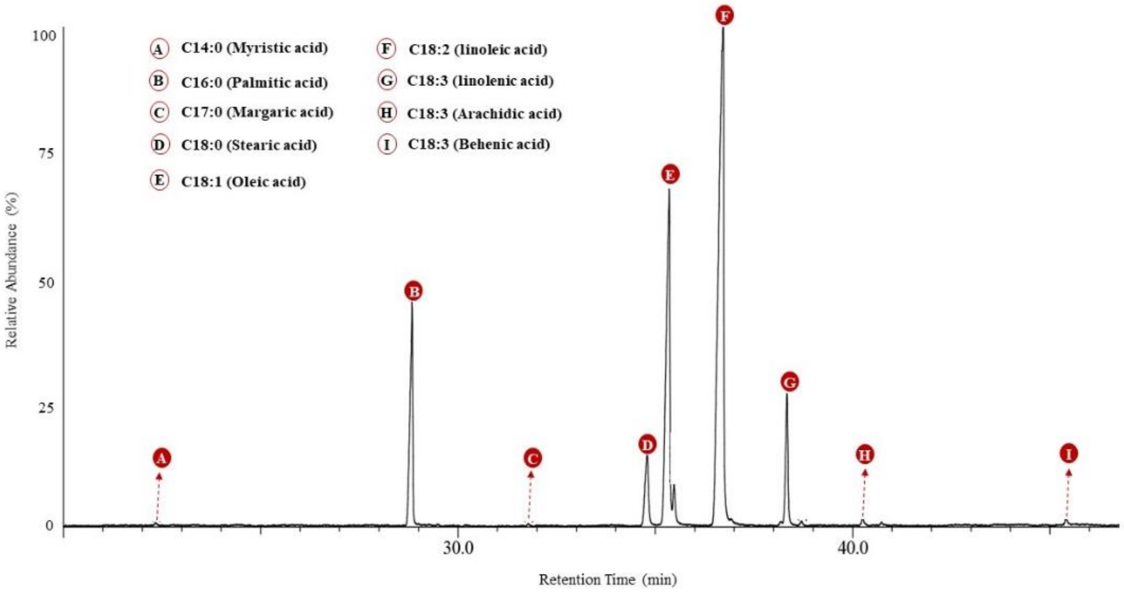

Figure 1. Chromatogram of soybean oil methyl

Table 1. Fatty acid composition of soybean oil

Fatty Acid	Relative Concentration (%)
C14:0 (Myristic acid)	0.09
C16:0 (Palmitic acid)	11.26
C17:0 (Margaric acid)	0.1
C18:0 (Stearic acid)	4.83
C18:1 (Oleic acid)	25.73
C18:2 (Linoleic acid)	50.39
C18:3 (Linolenic acid)	6.85
C20:0 (Arachidic acid)	0.41
C22:0 (Behenic acid)	0.34

Table 2. Chemical composition of synthesized soybean bio-additive

Peak	Relative Concentration (%)	Boiling Point (°C)	Chemical Composition
A	0.19	200	Palmitic acid
B	1.20	200	Linoleic acid
C	6.14	210	2-hydroxyethyl ester palmitate
D	53.02	227	2-hydroxyethyl ester linoleate
E	4.08	229	2-hydroxyethyl ester stearate
F	0.34	244	2-hydroxyethyl ester arachidate
G	2.60	248	2-hydroxyethyl ester nervonate
H	0.32	260	2-hydroxyethyl ester behenate
I	0.62	305	(12E,15E)-2-(heptadecanoyloxy)ethyl octadeca-12,15-dienoate
J	6.32	305	(12E, 15E)-2-(stearoyloxy)ethyl octadeca-12,15-dienoate
K	0.94	305	9E,12E,15E-2-(palmitoyloxy)ethyl octadeca-9,12,15-trienoate
L	24.23	305	(9E,12E,15E)-2-((E)-octadec-9-enoyloxy)ethyl octadeca-9,12,15-trienoate

3.2. Discussion

3.2.1. Fatty acid composition of soybean oil

The three main fatty acids found in soybean oil are linoleic acid (C18:2), oleic acid (C18:1), and palmitic acid (C16: 0) which are the same as those studied by [8-10]. Previous researchers reported a correlation between the length of the fatty acid chain (C12-C18) and the number of double bonds of an FAEE and its lubricity ability. The result is that the ethyl ester with the longest fatty acid chain (C18) has the best lubricity. Furthermore, the lubricity ability of FAEE (C18) was observed with the different number of double bonds. As a result, ethyl linolenic acid (C18:2) has a better lubricity ability than ethyl oleate (C18:1) and ethyl stearate (C18:0) [13]. So, the abundant amount of unsaturated fatty acid from soybean oil makes it a good raw material to produce lubricity improver bio-additive.

3.2.2. Synthesis of Hydroxy Ethyl Ester (HEE)

Based on analysis of the GC-MS spectrum, the 2-hydroxyethyl ester compounds have been formed with a yield of 66.5% (relative to the area of the chromatogram peak). HEE compound is a mixture of numerous compounds, such as 2 hydroxyethyl palmitate, 2 hydroxyethyl linoleate, 2 hydroxyethyl stearate, 2 hydroxyethyl arachidonate, 2 hydroxyethyl nervate, and 2 hydroxyethyl behenate. Among the five HEE compounds identified, 2-hydroxyethyl linoleate (18: 2) was the HEE compound with the most relative abundance, which was 53.02%.

Additive compounds have better lubricating properties with higher levels of unsaturation, stronger polarity and contain more hydroxyl groups in their molecules [11]., while The presence of a hydroxy group on the head of the HEE compound makes it more polar than FAME or FAEE which has been widely used as lubrication enhancing additive in low sulphur diesel fuel. Therefore, the HEE compounds that have been successfully synthesized in this study have the potential to be a good lubricity-enhancing bio additive for diesel fuel with low sulphur content. The general mechanism by which HEE compounds can act as a lubricity improver on low sulphur diesel fuel is shown in Figure 2. when HEE is added to diesel fuel, polar heads of HEE compounds attract to the metal surface and upon interacting with a metal surface, the aliphatic chains can orient themselves to forms a surface boundary layer/film to prevent direct metal/metal contact [11, 21].

In addition to HEE compounds, there are 2 groups of compounds identified, the first is a group of free fatty acid compounds found at the retention times of 14.6 and 18 minutes which indicate palmitic acid and linoleic acid compounds. Another group of compounds is ethylene glycol diester with an amount of about 31.49% which is present at the retention time of 45.7; 49.3; 49.5 and 54.4 minutes, which indicate (12E, 15E) -2- (heptadecanoyloxy) ethyl octadeca-12,15-dienoate, (12E, 15E) -2- (stearoyloxy) ethyl octadeca-12,15-dienoate, 9E, 12E, 15E-2-(palmitoyloxy) ethyl octadeca-9,12,15-trienoate and (9E, 12E, 15E) -2 - ((E) -octadec-9-enoyloxy) ethyl octadeca-9,12,15-trienoate.

The ethylene glycol diester (EGDEs) group of compounds can be formed because the reaction between the two nucleophiles contained in ethylene glycol attacks the carbonyl carbon in the triglyceride molecule. Although it acts as a side compound of HEE synthesis, ethylene glycol diester compounds have the potential to be used as bioadditive lubricity improver [22, 23].

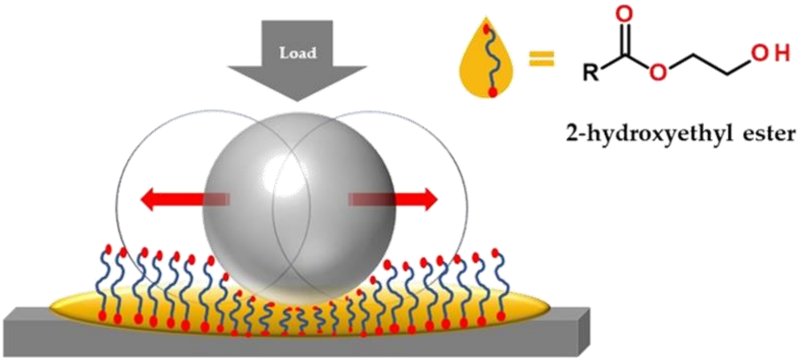

Figure 2. General mechanism HEE compound prevents metal/metal contact

4. Conclusion

Based on the results of the research conducted, we have successfully synthesized 2-hydroxyethyl ester (HEE) with a yield of 66.5%, through the transesterification reaction of soybean oil and ethylene glycol with potassium carbonate as a catalyst. HEE compound is a mixture of many compounds, where 2-hydroxyethyl linoleate (18:2) with the highest amount is 53.02%. The presence of a hydroxy group in this compound can give better lubricity properties compared to FAME and FAEE which are frequently used as lubricity-enhancing additives in low sulphur diesel fuel. However, there are products other than HEE that are formed in this reaction, namely a mixture of ethylene glycol diester compounds with a yield of 31.49%. Although it acts as a side compound for HEE synthesis, it is known that ethylene glycol diester can be used as a lubricant enhancer as well.

Acknowledgement

The authors fully acknowledged the Institut Teknologi Sepuluh Nopember, who has given financial support. PEM-Akamigas Cepu, Central of Java, has given the technical support to carry out this research.

Author's Declaration

Authors' contributions and responsibilities

The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

Funding

This research has been funded by Institut Teknologi Sepuluh Nopember, Indonesia.

Availability of data and materials

All data are available from the authors.

Competing interests

The authors declare no competing interest.

Additional information

Selected and revised paper from The 2nd Borobudur International Symposium 2020 (BIS 2020) after peer-reviewed by Scientific Committee of BIS 2020 and peer-reviewers of Automotive Experiences.

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