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Characterization of Palm Biodiesel Deposit on The Locomotive Engine Fuel Filters: Composition, Physical, Chemical Properties

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Abstract. High ratio biodiesel blend fuel has become mandatory for all sectors including road and non-road vehicles since 2016. All stakeholders of biofuel in Indonesia had conducted a specified investigation through road tests 40,000 km to evaluate the high biodiesel effect on performance, emissions, and engine components. An improvement in fuel properties of biodiesel FAME has also been stated in the new fuel standard. Moreover, the recommendation for proper high biodiesel ratio handling and storage also socialized to all stakeholders. However, shorter fuel filter mileage and fuel degradation for long storing still remain issues for the implementation of biodiesel in Indonesia. An assessment of biodiesel in a locomotive in Indonesia was conducted through a rail test conducted for 6 months with a coal train in Sumatera Island with a blend ratio of 20% by volume (B20) and pure diesel fuel (B0) as a reference. The purpose of this rail test was to investigate the effect of B20 on various factors at railway application. This paper is presented a detailed analysis of the deposits formed in the used main and used twin filters of the locomotive for one maintenance period. An analysis of morphology, elemental analysis, and its physical and chemical properties was done by using a digital microscope, thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR), and gas chromatography-mass spectrometry (GC-MS), respectively. An analysis showed that B20 had more deposits on the main filter compared with B0, while it had fewer deposits on the twin filter. Deposit in both of main and twin filters for B20 was identified mostly from hydrocarbons and fatty acid methyl esters. Analysis results also recommended that the fuel filter mileage of B20 was still possible to be prolonged with the current fuel filter standard by considering to deposit formation for both B0 and B20 still under tolerance level.

Keywords: B20; FAME; Fuel filter; Locomotive; Palm biodiesel

1. Introduction

Biodiesel is a promising alternative fuel to support the implementation of sustainable fuels and reduction of exhaust gas emissions from transportation sector in Indonesia (Wirawan *et al.*, 2024; Tjahjono *et al.*, 2021). Several improvements have been applied to the automotive sector, including the specified development of a fuel system for biodiesel, appropriate handling and storage of biodiesel, and limitations on the amount of water and

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mono-glycerides permitted biodiesel. Those improvements are required to meet specified engine performance and emission from various biodiesel production processes and feedstock resources (Zhang *et al.,* 2022; Said *et al.,* 2018; Susila, Rachimoellah, and Sutantra, 2011). Many studies have reported the effects of using biodiesel fuel in automotive applications with various engine technologies and biodiesel resources (Lee and Cho, 2024; Reksowardojo *et al*., 2023; Mokhtar *et al*., 2023; Pandey, Mishra, and Verma, 2022; Kumbhar *et al*., 2021). Nevertheless, a few studies have reported the utilization of high-ratio biodiesel with a blending volume above 10% in the locomotive applications. An implementation of biodiesel on a locomotive had similar results to automotive, which could raise an issue in the intake system and fuel filter clogging (Stead *et al.,* 2019). Skinner *et al.* (2007) reported that the utilization of biodiesel could decrease fuel filter mileage and reduce fuel economy. At the same time, it has an advantage on low exhaust gas emission except for NOx without any significant reduction in engine power. Utilization of high-ratio biodiesel fuels could also affect to accelerate the replacement time of fuel filters due to their faster deposit formation (Heck, Mohammad, and Abi-Akar, n.d.; Komariah *et al.,* 2018; Graver, Frey, and Hu, 2016; Ge *et al.,* 2009).

Deposit in the engine could be in various forms, including carbonaceous, polymeric, carboxylate salts, and inorganic salts. Further, a typical deposit in an engine fuelled with biodiesel was a sticky deposit due to its saturated fatty acid contents. High temperature and pressure of the advanced injection system would lead to building deposits at the injector due to cavitation in the injector and fuel thermal stress. While storage sediment, biodiesel, lubricant adulteration, metal catalyst, water as well as bio-contaminant were also the sources of deposit for both in injector and fuel filter (Barker *et al.,* 2020). Deposit formation in the injector formed due to hydrocarbon decomposition and condensation as a result of the unburn product of low volatility, high viscosity, and density of biodiesel. Carbonaceous deposits could occur in injectors at a temperature higher than 350°C (Hoang, Le, and Pham, 2020; Hoang and Le, 2019). Deposit formation in biodiesel fuel is also affected by its long chains, low ignition point, and high boiling point, resulting in a higher maximum evaporation rate point (MEP) compared to diesel fuel (Suryantoro *et al.,* 2016). Moreover, Hoang also reported that the degradation of lubricating oil would also influence deposit formation in the combustion chamber (Hoang and Pham, 2018). While the deposit in the fuel filter was in a sticky form which was mainly influenced by biodiesel contaminants such as monoglyceride, water, and metal content which occur at a temperature below its pour point. These types of deposits would be easily removed as the temperature increase. However, if there is some solid deposit from diesel fuel accumulated in the void space of the fuel filter, then it is mixed with a sticky deposit of biodiesel then, which would result in fuel filter clogging (Paryanto *et al.,* 2022). Csontos *et al.* (2020; 2019) evaluated deposit characteristics by using Gas Chromatography-Mass Spectrometry (GC-MS), *X-ray Fluorescence* Spectroscopy-Energy Dispersive X-ray Spectrometry (XRF-EDX), Fourier-Transform Infrared Spectroscopy (FTIR), and Thermogravimetric (TGA). They identified that carboxylic acid (CA) and unreacted CA, oxidized polymer compounds, glycerol, sterol, other impurities, and contaminants could be from biodiesel degradation and its production process (Csontos *et al.,* 2020; 2019). Meanwhile, Barker *et al.* (2009) have reported that carbon with C_{16}-C_{18} was the main component of the injector deposit (Barker *et al.,* 2009). However, some studies also have reported that the utilization of biodiesel fuel would not have a significant difference in fuel filter mileage compared with pure fossil diesel fuel (Petiteaux and Monsallier, 2019; Nelson, 2012). Lammert, Barnitt, and McCormick (2009) have recognized unsuitable cold flow properties of biodiesel for implementation in low-temperature conditions during the winter season.

In a previous study, a used fuel filter of B20 from one period of fuel filter changing was analyzed with uncontrolled properties of B20, locomotives, and trips. It found that the B20 fuel filter had shorter mileage (Ma'ruf and Haryono, 2020). Furthermore, an evaluation with-rail test B20 was conducted on a coal train in Sumatera Island to evaluate the effect of B20 on the locomotive in the more controllable testing environment, including fuel, locomotive, and trip. Specified locomotives with controlled B20 properties were prepared for this evaluation during its six months rail test. This paper reports specifically on the characteristic of a used fuel filter diesel locomotive for 3 months fuelled with B20 and pure diesel fuel. This rail test has been used as a reference to improve the biodiesel production process, the application of proper handling and storing of B20, and the development of engine components, especially fuel filters. Thus, the target for a longer lifetime of biodiesel fuel filters with high concentrations can be achieved with the use of diesel oil (B0).

2. Methods

Deposit formation in fuel filters is influenced by fuel impurities and contaminants and fuel flow rate. In this rail test, fuel properties were controlled with specified fuel sources, handling, and storing. Periodic sampling and analyzing of tested fuel were conducted during the rail test to ensure the fuel properties meet the requirements stated by the Directorate General of Oil and Gas of Indonesia. However, a contaminant from the surrounding environment during the rail test, such as coal dust, could not be controlled. Here, the period analysis of tested fuels showed a deviation of less than 5%**.** The fuel flow rate, which is indicated by the mode of operation of the locomotive, was controlled by a constant trip with the same locomotive. Here, the trainload represented by carried coal could not also be set in the same amount for each trip. Nevertheless, the trainload for every trip was recorded to analyze the deviation of load. Furthermore, a source of error could also be possible during the sampling process, preparation, and analysis of the samples.

2.1. Materials

A rail test was conducted using two types of fuels: a biodiesel blend with a ratio of 20% by volume (B20) and industrial diesel fuel available in the Indonesian market (B0). Table 1 shows the fuel properties for both B0 and B20. In this study, the fuel properties shown in Table 1 were tested using the specified ASTM methods, except for oxidation stability, which was measured according to EN 15751 standard. B20 exhibited better properties in terms of cetane number, density, flash point, sulfur content, distillation, and lubricity compared to B0, as shown in Table 1. However, B20 had some disadvantages, such as higher water content and monoglyceride levels, which could lead to deposit formation in the fuel filter.

Table 1 Detailed Properties of B0 and B20 Fuel Sample

No	Parameter	Unit	Test Result		Methods
			B-0	B-20	
1	Cetane Number		49.8	53.4	D 613
2	Density at 15 ºC	kg/m³	842.2	848.1	D 4052
3	Kinematic Viscosity at 40 ºC	mm² /s	2.43	2.8	D 445
4	Sulfur	%m/m	0.135	0.091	D 4294
5	Distillation (T90)	ºC	344	342.5	D 86
6	Flash Point	ºC	58	63	D 93
7	Pour Point	ºC	12	9	D 97
8	Water Content	mg/kg	115.63	232.11	D 6304
9	Monoglyceride Content	%v/v	0	0.8	D 6584
10	Total Acid Number	mg KOH/g	0.14	0.16	D 664
11	Lubricity HFRR	micron	398	218	D 6079
12	Oxidation Stability	Hours	>48	>48	EN 15751

2.2. Testing Methods and Analysis

An analysis of the deposit and its contents of the fuel filter was done for the used main and twin filters of the coal train fuelled with B20 and B0. Two trains were run at the same time for 6 months, and periodic maintenance was at 3 months. Here, the sampling of used fuel filterss was at fuel filter changing on 3 months based on a recommendation from the locomotive manufacturer. In this study, used main and twin filter deposits were identified for their morphology, elemental analysis, and chemical and physical analysis by using a Hirox microscope with type KH-8700, thermogravimetric (TGA) with differential thermogravimetric (DTG), gas chromatography-mass spectrometry (GC-MS) type 6890 and Fourier-transform infrared spectroscopy (FTIR) Shimadzu–IR Prestige 21, respectively. Here, a filter size of 1 cm^2 was soaked in 10ml of acetone, chloroform dichloromethane (DCM), and n-hexane as organic solvents before GC-MS analysis. The purposes of using various solvents were to evaluate and determine the effectivity of solvent on dissolving precipitate. A Whatman microfiber filter paper with a diameter of 47 mm and pore of 0.7 µm was utilized to filter precipitate matter in solvent to avoid small solid particles entering the GC-MS column.

2.3. Diesel Locomotive Coal Train

A rail test was conducted with a locomotive from Electro-Motive Diesel (EMD) Model GT 38 AC. The locomotive has three stages of fuel filtration, including a strainer, main filter, and twin filter. A strainer is installed to filter contaminants with sizes >10 microns and water. While the main filter and twin filter was intended to filter particle with a size of 5-10 micron and 3-4 microns, respectively. Figure 1 shows the fuel filtration system for the EMD locomotive for both fuelled with B20 and B0.

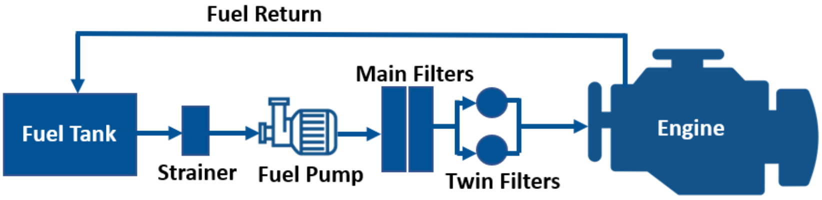


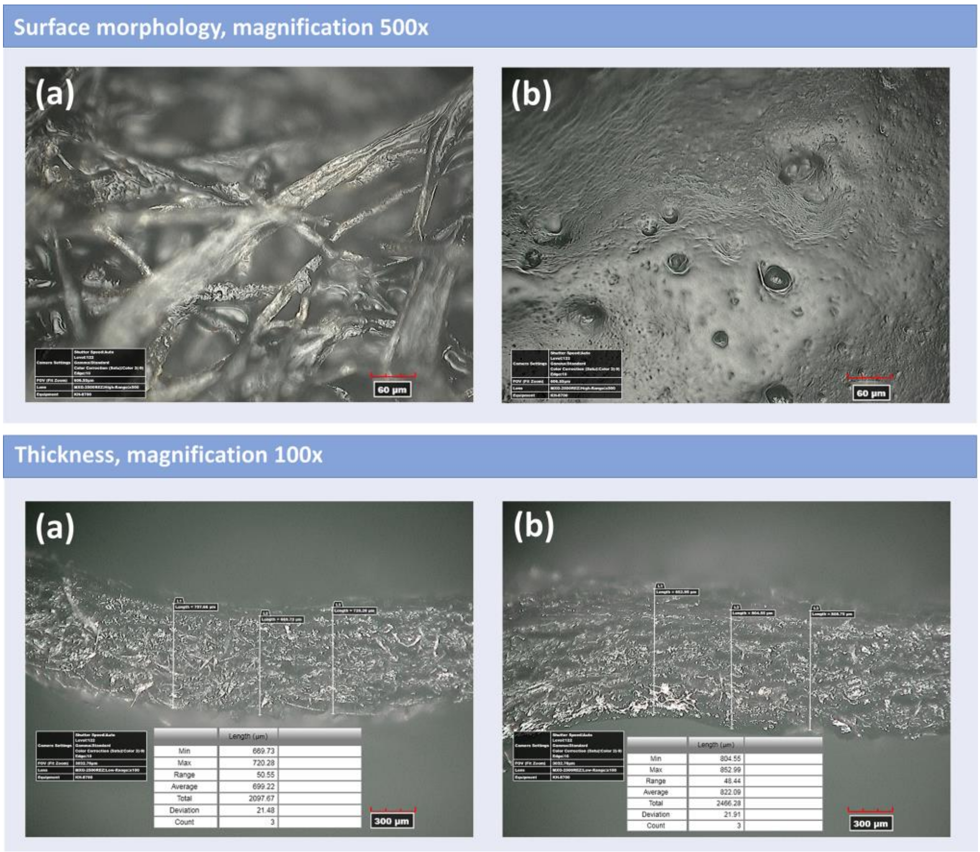
Figure 1 Schematic diagram of fuel system in EMD locomotive

3. Results and Discussion

Morphology and deposit content was evaluated on the main and twin fuel filter of the locomotive fueled with B20 after 3 months of use. Further, an evaluation of the fuel filter with B0 fuel had also been done with B0 as a reference condition. Here, deposit evaluation on the strainer was not conducted due to fewer deposits found as it just filtered coarse bulk particles such as rocks or gavel for both locomotives with B20 and B0.

3.1. Morphology and Thickness of Deposit

Figure 2 shows an evaluation of morphology and thickness of deposit on used main filters with B20 and B0 fuel after 3 months by using a 3D digital microscope at a magnification of 500 times and 100 times, respectively. The used main filter for B20 was fully covered by deposit so that the fuel fiber could not be recognized, while the fiber and pores of the used main filter with B0 were still clearly observable. An evaluation of deposit thickness on the used main filter showed that B20 was higher compared with B0, with an average of 822 µm and 699 µm, respectively. Moreover, it is found that the deposit characteristic of B20 was soft so that B20 was still possible to flow through the used main filter with less resistance. A measurement of differential pressure also showed that the pressure difference between the upstream and downstream of the filter did not change significantly. Therefore, it could be the reason for similar power of locomotive after 3 months with B20 (EBTKE, 2018).



(a)

	Length (µm)	
Min	669.73	
Max	720.28	
Range	50.55	
Average	699.22	
Total	2097.67	
Deviation	21.48	
Count	3	

(b)

	Length (µm)	
Min	804.55	
Max	852.99	
Range	48.44	
Average	822.09	
Total	2466.28	
Deviation	21.91	
Count	3	

Figure 2 Main filter condition after 3 months of usage: (a) B0; and (b) B20

Visual inspection of deposit on the used twin filter showed that fibers were still clearly visible for both B20 and B0 as shown in Figure 3. However, the granule deposit size of B20 was bigger compared with B0, which covers the used twin filter surface uniformly. Figure 3 also shows the thickness of the deposit at the used twin filter fueled with B20 and B0. Measurement results showed that B20 and B0 had an average thickness of 601 µm and 621 µm, respectively, as shown in Figure 3. The lower deposit thickness of B20 compared with B0 could be considered due to most of the contaminants B20 was filtered on the main filter resulted in less filtered deposit in twin filters. Blend fuel of FAME biodiesel is characterized by solid-formed deposit and the wet flexible waxy precipitate, which resulted from impurities of diesel fuel and FAME. Moreover, contaminants of blend fuel could also be from thermal oxidation and metal-catalyzed oxidation of high-temperature recirculation fuel. In our previous investigation on used locomotive fuel filters, we identified some elements contained in the deposit of the used filters, either organics or un-organics. An analysis of three samples of a used locomotive, water separator, and generator fuel filter has found metal elements such as Al, Si, S, and Ca. An accumulated combination of those solid and wet waxy deposits would result in suddenly blocked fuel filter (Paryanto *et al.*, 2022; Ma'ruf and Haryono, 2020; Wadumesthrige *et al.*, 2010).

Morphology and thickness measurement on the same pores at the main and twin filter result showed that the filtering efficiency of B20 is better than B0. The difference in result with the previous study could be considered due to its better properties, handling, and storing of B20 during rail test. Here, a suppression of deposit in fuel filter could be achieved in a more significant result by controlling an amount of monoglyceride content at a maximum of 0.4%, as an amount above this number is still expected to form deposit (Cardeno *et al.*, 2020). Therefore, an evaluation of the proper pore size diameter of high-ratio biodiesel would be required to achieve effective filtration. Fuel filter pore diameter also can determine fuel filter mileage based on targeted contaminants of high biodiesel blends.

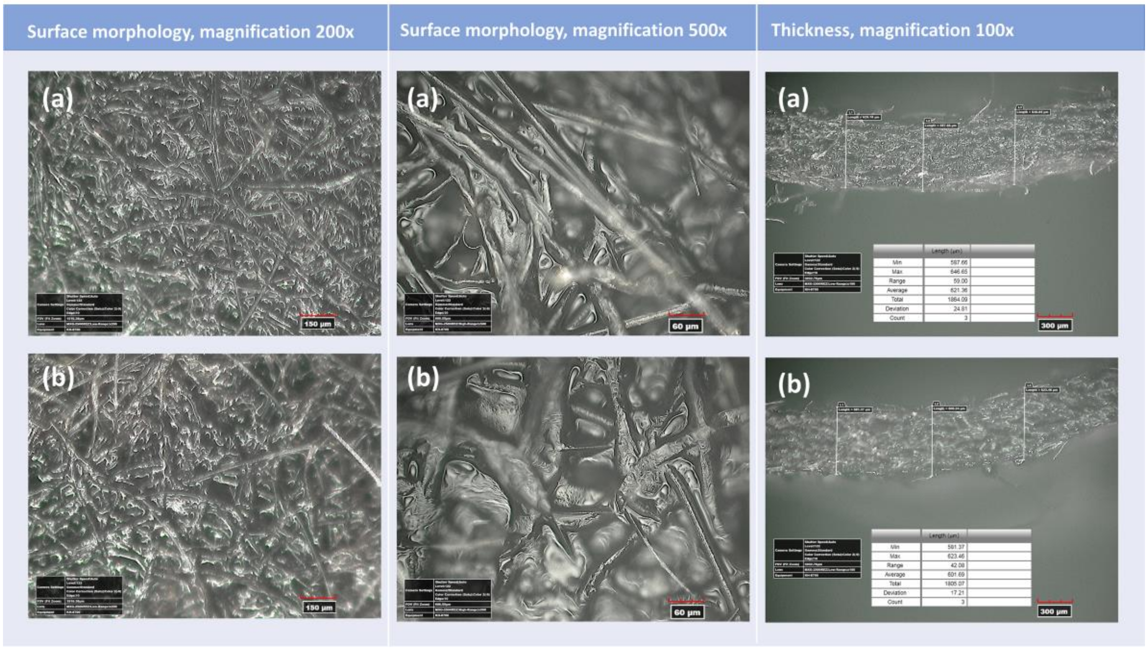


Figure 3 Twin filter condition after used for 3 months: (a) B0; and (b) B20

3.2. *Physical and chemical properties of the filter*

To identify the chemical functional groups captured on the filter, an analysis of the filter used with B20 fuel was performed using FTIR. Csontos *et al.* (2019) also utilized this method in their work. However, since FTIR can also detect the functional groups of the filter material itself, a new filter was used as a comparison. The FTIR analysis spectrum is presented in Figure 4, and Table 2 shows the predicted functional groups for each peak.

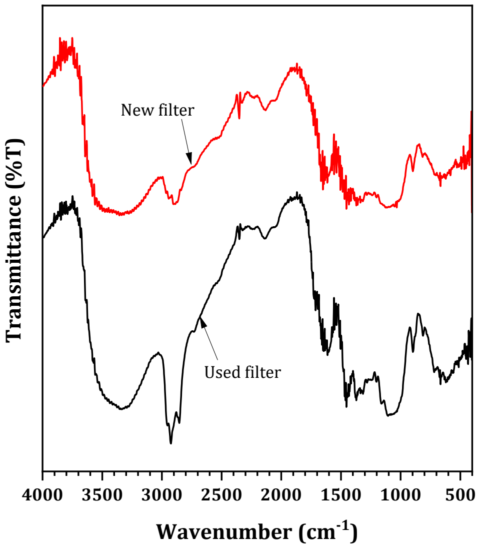
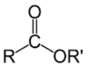
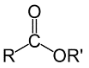


Figure 4 Spectrum FTIR of B20 fuel

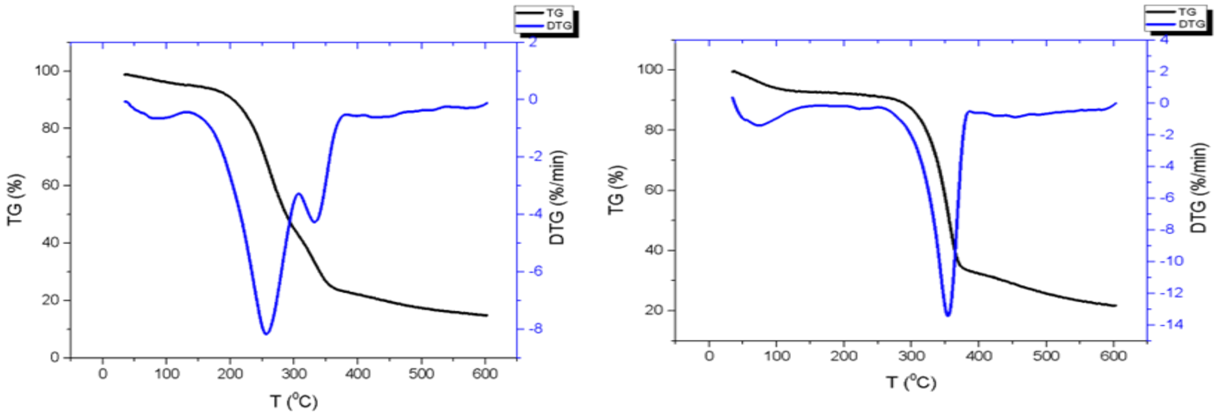
Based on the spectrum of FTIR, as shown in Figure 4, the peaks on a new filter generally also appeared on the used filter. However, several new peaks appeared on the used filter, such as at wave numbers 1259 and 1232 cm^{-1}, which were the C-O-C group of ester, probably derived from biodiesel. Furthermore, wave numbers of 626 and 468 cm^1 were the naphthalene group, and 609 cm^{-1} was SO_2 group derived from petrodiesel fuel.

Table 2 Chemical functional groups in the filter based on FTIR analysis

Chemical functional group	Wavenumber for used filter (cm^{-1})	Wavenumber for the new filter (cm^{-1})
$-NH_2$ aromatic amines, primary amines, and amides		3485
$-OH$ alcohol or phenol	3342	3344
$-OH$ alcohol or phenol	3329	3269
$-CH_3$ and $-CH_2-$ aliphatic compounds	2929	2887
$-CH_3$ and $-CH_2-$ aliphatic compounds	2873	
C≡C	2139	2135
C=C aromatic	1600	1600
$-CH_3$ or $-CH_2-$	1448	1448
H-C=O	1357	1357
H-C=O		1340
C-O-C from Ester 	1259	
C-O-C from Ester 	1232	
C-OH from secondary alcohol or C-O-C in aliphatic ethers		1109
C-OH from secondary alcohol or C-O-C in aliphatic ethers	1093	1070
C-C-O in alcohol	894	898
C-C-CHO in aldehydes		665
Naphthalene	626	
O-C-O in esters		621
$-SO_2-$ in sulfones	609	513
Naphthalene	468	

The filtered impurities after using B20 fuel originated from biodiesel and petrodiesel. One of the efforts to improve the quality of biodiesel blends can be done by reducing the content of impurities in B0, which implies minimizing the interaction in the biodiesel blend. The used filter was also analyzed by Thermogravimetric (TG) analysis to understand its thermal properties compared with the new filter. TG analysis was conducted at N_2 conditions with a heating rate of 10°C /min from room temperature to 600°C. Figure 5 shows TG or weight loss and derivative thermogravimetry (DTG) curves of the filters.

Figure 5 showed that thermal degradation of the used filter containing deposits vaporized at three different regions of 35 - 150°C, 150 - 320°C, and 320 - 400°C with peak temperatures around 80°C, 250°C, and 330°C. The peak temperature at 250°C could correspond with TGA results conducted by ELTahry *et al.* (2022) that peak temperatures of vaporized deposits in the form of glycerol, sterol glucosides (SGs), and metal carboxylates at around 250°C, 350°C, and 480°C, respectively. At region 35 – 150°C was attributed to water evaporation and volatile compounds in the filter material. Besides glycerol (ELTahry *et al.*, 2022), at region 150 – 320°C hydrocarbons with carbon atoms [C] ranging from C13 - C18, such as Tridecane, Tetradecane, Pentadecane, Hexadecane, Tridecane, and Octadecane were decomposed because the used filter was analyzed as received. Moreover, in the region at 320 – 400°C, there was decomposition or evaporation of larger carbon atoms [C] ranging from C19 - C27, such as Nonadecane, Eicosane, Heneicosane, Docosane, Tricosane, Heptacosane, Tetracosane, Pentacosane, and Eicosane based on GCMS analysis as shown in Figs. 6-7. While the new filter only consisted of two regions of 35 – 150°C and 250 – 400°C. At 250 - 400°C, decomposition of the larger molecular weight material, such as polymer or composite, occurred in the new filter. Decomposition of the filter material also occurred in the used filter that was incorporated with the deposited material.



(a) (b)

Figure 5 TG and DTG curves of the used filter (a) and new filter (b)

3.3. Composition of deposit on the filters

The filter used was analyzed with an elemental analyzer to determine the percentage of carbon, hydrogen, and oxygen in the deposited materials. As well as FTIR analysis, a new filter was also used for comparison because the elements in the filter material were detected. Table 3 shows the elemental analysis results. Carbon [C] and hydrogen [H] contents in the used filter increased compared to the new one by 20.4% and 3%, respectively, caused by fuel or engine crust passing through the filter. The nitrogen [N] content was minimal and can be neglected as a trace element. Meanwhile, the oxygen [O] content in the used filter was lesser than in the new one because of the high added carbon from trapped fuel, while the O value was calculated based on the difference between the percentages of C, H, and N. The value of detected O in the used filter was derived from the filter material, fuels, or sulfur oxide. The existence of sulfur compounds on the used filter was confirmed by FTIR analysis at peak 609 cm^{-1} (Table 1) because the elemental analyzer could only detect C, H, and N.

Table 3 Analysis of used and new filters

Element [wt. %]	Used filter	New filter
Carbon	66.0	45.6
Hydrogen	9.3	6.4
Nitrogen	0.5	0.8
Oxygen*	24.2	47.2

*) *calculated by 100% - (C+H+N)*

The deposited substance on the main and twin filters with B20 or B0 fuels was identified by GCMS. Among solvents used for diluted chemicals on the B0 fuel filter, chloroform appears to have a better solvent than the others (acetone, dichloromethane, and n-hexane) due to more chemicals identified, as shown in Figure 6. Based on the identified compounds, the chemicals trapped on the used filter of B0 were hydrocarbons ranging from C12 - C27, which were dominated by pentadecane, 2,6,10,14-tetramethyl- ($C_{19}H_{40}$). Alkane hydrocarbon compounds with carbon number ≥C17 are in a solid form at 20°C and were naturally trapped on the filter, leading to a blockage. The presence of C12-C16 compounds in Figure 6 can be attributed to the lack of pretreatment of the filter before soaking it in the organic solvent. As a results, the C12-C16 compounds remained on the filter even at 20°C. Additionally, naphthalene compounds caused blockage due to their high melting point and solid form at room temperature. These compounds may have been formed through the aromatization of straight-chain alkanes (Venkataraman and Eser, 2008).

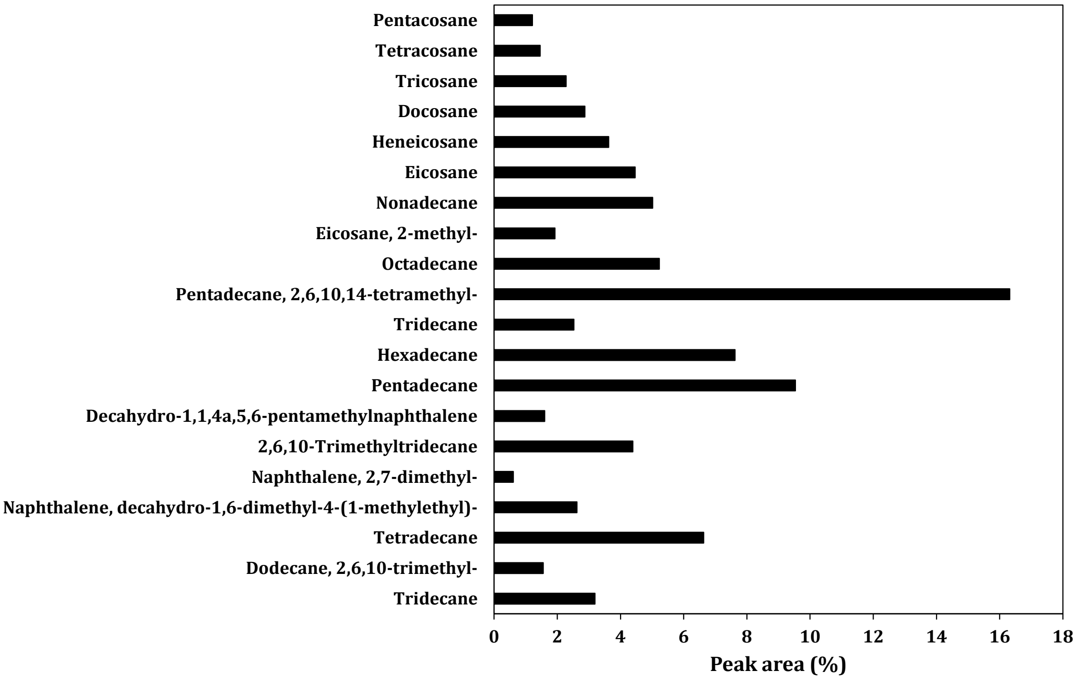


Figure 6 Identified chemicals collected by chloroform solvent from the used filter after passing B0 fuel

Chloroform was a more effective solvent in the used filter that passed through B0. Similarly, in the used filter that passed through B20, many chemical compounds were also dissolved using chloroform as the solvent. The identified chemicals on the used filter with B20 fuels using chloroform solvent can be seen in Figure 7. The identified chemicals were alkane hydrocarbons and fatty acid methyl ester, mainly Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$), and 9-Octadecenoic acid, methyl ester, (E) - ($C_{19}H_{36}O_2$).

The identified hydrocarbon and fatty acid methyl ester compounds were constituent chemicals of petrodiesel oil and palm biodiesel, respectively. Chemical compounds such as Methyl tetradecanoate and Tridecanoic acid 12-methyl-, methyl esters were derived from fatty acid C14 (myristic acid) that solid phase at room temperature. Likewise, Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$), and Methyl stearate ($C_{19}H_{38}O_2$) were derived from C16:0 (palmitic acid) and C18:0 (stearic acid) fatty acids, respectively, also in solid form at room temperature. The presence of these chemicals in a fuel caused filter blockage.

According to Figure 7, peak areas of Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$), and 9-Octadecenoic acid methyl ester, (E) - ($C_{19}H_{36}O_2$) were higher than other chemicals due to the higher concentration of palmitic and oleic acid in the palm oil (Mancini *et al.,* 2015). The methyl esters derived from oleic acids, such as 8,11-Octadecadienoic acid, methyl ester, and 9-Octadecenoic acid, methyl ester, (E)- do not cause filter blocking because of the liquid phase at room temperature. As shown in Figure 7, their presence was due to the fact that no pretreatment was performed on the used filter before soaking.

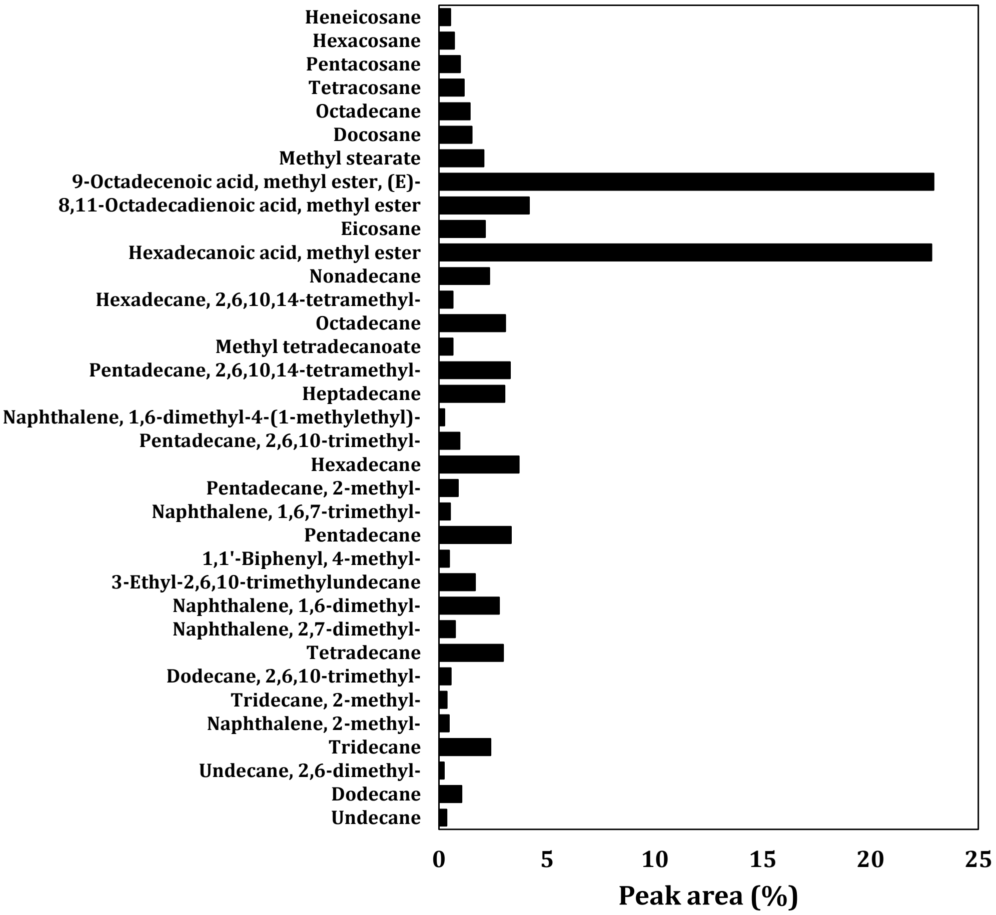


Figure 7 GC-MS analysis of compounds collected by chloroform solvent from the used filter after passing B20 fuel

4. Conclusions

The study on deposit analysis of used fuel filters showed that B20 had higher deposit content in the main filter while less deposit was formed in the twin filter compared with B0. An evaluation of 3 months of usage showed deposit filled almost all surfaces of the main filter, while the coverage of the deposit in the twin filter was not as much as in the main filter with visible porosity and filter fibers. Furthermore, the used filter of rail locomotive fuelled by B20 identified various deposit compounds which contained alkane hydrocarbon compounds with carbon number ≥C17 and fatty acid methyl esters C15 - C19. However, deposit formation on both B0 and B20 was at a tolerance level so that it did not require faster changing. This rail test also showed that controlled handling, storing and quality of B20 would result in less formed deposits in fuel filters.

Acknowledgments

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