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Effects of sulphur on the storage stability of the bio and fossil fuel blends

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Abstract. In this study, the aim was to find out if mixing two common fuels together could be beneficial for both the environment and storage stability of fuel. It is obvious, that adding biodiesel to fossil fuel will decrease its sulphur content and reduce its carbon monoxide and hydrocarbon, sulphur dioxide and soot emissions. But will the high sulphur content enhance the storage stability of the biodiesel? Four B20 samples were produced, consisting of 20 vol% biodiesel and 80 vol% fossil diesel. The samples were prepared from rapeseed methyl ester (RME), low sulphuric fossil diesel fuel and high sulphuric diesel solvent. The blends had different sulphur contents of 6, 76, 149 and 226 mg kg^{-1}. For these B20 fuel samples, the parameters were compared that correlate with the storage stability of the fuel blends. The studied parameters were the oxidation stability (OSI, according to EN 15751:2015), acid number (AN, according to EN 14104:2003) and kinematic viscosity (KV, by Stabinger SVM 3000 rotational viscometer). The measurements were carried out straight after mixing the blends, and again after 4, 8 and 12 weeks. According to the results, the fuel containing less sulphur slightly lost its oxidation stability within three months. Instead, the oxidation stability of high sulphuric samples improved within the same time frame. As a conclusion, the study gave a reason to assume that – in spite of its known drawbacks – the sulphur may be favourable to fuel blends' storage stability but the phenomenon and chemistry should be studied in more detail.

Key words: Biofuel, blending, FAME, diesel fuel, B20, stability, sulphur.

INTRODUCTION

During the recent decades, the sulphur content of liquid engine fuels has drastically decreased to reduce harmful effects of exhaust sulphur compounds. The reasons for the fuel sulphur limitations are both technical and environmental; sulphur pollutes the air and poisons the catalysts in emission control systems. Fuel sulphur also increases soot and particulate matter emissions in the exhaust of all kind of internal combustion engines, causes corrosion and also has a deleterious effect on advanced after treatment systems used for CO, HC, NOx and particulate reduction (Kalghatgi, 2014; Zhao et al., 2016). Moreover, the fuel sulphur causes corrosion inside the engine cylinder. In combustion processes, sulphur oxidizes to sulphur dioxide SO_2. It will be further oxidized to SO_3 and then again react with water. Formed sulphuric acid, H_2SO_4, will condensate on the metal surfaces and cause corrosion (Heywood, 1988). This aspect makes the de-sulphurization important but refining processes which remove the sulphur from the fuel simultaneously reduce fuels lubricity.

Sulphur emissions, both gaseous and particles, are also harmful for human health. An acute exposure can cause trouble in breathing and a long-time exposure for those emissions can cause heart disease, pulmonary illness or even untimely death. In the environment, sulphur oxides are reactive and form H_2SO_4 which comes down with the rain and the acid rain again depletes the nature in many ways. Moreover, buildings disintegrate because of the acid rain (Pan, 2011).

In parallel with the fuel de-sulphurization development, the need to increase the use of renewable fuels continues to grow to also reduce greenhouse gas (GHG) emissions. In countryside, there may also be possibilities to utilize own yield of rapeseed or other oils as biodiesel fuel through transesterification and this could be financially beneficial for the farmers. In rural areas, there is an increasing need for cheap, both fossil and renewable, fuels in agricultural engine applications. Due to the environmental aspects, the fuel sulphur content is widely regulated but the content still remains quite high in the developing countries in Asia and Southern America. The cheapest fuels are not the cleanest, i.e., the sulphur contents in non-road fuel may in those regions be as high as 500 mg kg^{-1} while in Europe, the automotive diesel fuel standard, EN 590:2013 allows the fuel to contain a maximum of 10 mg kg^{-1} (SFS-EN 590:2013, 2013).

Due to the environmental reasons on one hand and to the agricultural needs, on the other hand, different blends of bio- and fossil fuels are becoming more popular. High sulphuric fossil fuels and the effect, they have on blends' properties, need to be studied further to find an economic, environmental and technical balance between de-sulphurization, fuel blending, and the usage of renewable components. It is also noteworthy that after de-sulphurization processing, the fuel lubricity must usually be returned by adding lubricity additives into the fuel. However, biodiesels or fatty acid methyl esters (FAMEs) have also been found to enhance the lubricity when they are mixed with diesel fuel oil (Guibet, 1999; Kalghatgi, 2014). Blending of FAME with low sulphur fossil diesel thus also improves the fuel lubricity.

The storage stability is one of the main quality parameters related to FAMEs. The deterioration of the properties of FAMEs during storage is more serious than for fossil fuels. Storage conditions, like ambient temperature, have of course an effect on storage time. Improving oxidation and storage stability is an important issue for commercializing the biodiesel (Bouaid et al., 2009). Some studies indicate that higher sulphur contents may beneficially affect the oxidation stability of the fuel blends (McCormick & Westbrook, 2007; Sirviö et al., 2016). Sulphur is an essential compound for living organisms. For a long time, organic sulphuric compounds, like glutathione, have been well known of their action to protect cells from free radicals and reactive oxygen compounds (Meister & Anderson, 1983). It has also been shown that glutathione can reduce the lipid peroxidation caused by reactive oxygen radicals (Roxas et al., 1997). In other words, an example of the anti-oxidative effect of sulphur compounds can be found from biochemistry. Biodiesel oxidation is a complex process of radical chain reactions. Hydroperoxides are formed first during degradation and they react further to form secondary oxidation products (Fattah et al., 2014). In living organisms and fuel blends, the reaction environments and reaction chains differ and it cannot be stated that the phenomenon is the same. Still, there is an interesting similarity and it needs to be studied further.

To investigate how the sulphur of fossil diesel may affect the OSI and other properties of biodiesel-fossil fuel blends, fuel blends were prepared having different sulphur contents. In Europe, the maximum FAME content in diesel fuel is 7 vol% according to the EN 590:2013 (SFS-EN 590:2013, 2013) but higher percentages are also available and targeted around the world. For example in the United States, the 20% blend fraction is becoming more common. B20 and B30 fuels do even have their own Standard, EN 16709, which specifies the quality of those fuels with a high biodiesel content (SFS-EN 16709:2015, 2015). That is why B20 fuels were chosen to be investigated in this study. Fuel blending may, however, cause operating risks. The fuels need to be stable and compatible with engine and other blended fuels. Several studies have been made to figure out how biodiesels and their blends affect the lifetime of the engines. A review of short run tests reports that biofuels can replace conventional diesel fuel but a long run analysis is needed for assessment of the engine life. Problems that may occur are carbon deposition, lubricating oil dilution, piston ring sticking and injector nozzle choking (Patel et al., 2016).

In this study, the aim was to find out how sulphur affects the storage stability of the prepared fuel blends. It was interesting to see if mixing two common non-road fuels could be beneficial for both the environment and storage stability of fuel in developing countries. It is clear, that adding non-sulphuric biodiesel to fossil fuel will decrease their blends' sulphur content and reduce emissions and other adverse effects. But will the high sulphur content enhance the storage stability of the biodiesel?

MATERIALS AND METHODS

In the current study, the parameters were investigated that correlate with the storage stability of the fuel blends. Four B20 fuel samples were compared consisting of 20 vol% biodiesel and 80 vol% fossil diesel. All of the samples were prepared by mixing rapeseed methyl ester (RME), diesel fuel oil (DFO) containing 6.6 mg kg^{-1} sulphur, and high sulphuric diesel solvent. Different amounts of sulphuric diesel solvent were added into the B20 samples. The achieved sulphur concentrations were 6 mg kg^{-1} (B20 1), 76 mg kg^{-1} (B20 2), 149 mg kg^{-1} (B20 3), and 226 mg kg^{-1} (B20 4). The aim was to figure out how the storage stability of the fuel blends change as a function of time when the samples were stored in stable conditions (in dark, at 20 °C) for 12 weeks.

The fuel samples were made by mixing three different compounds; RME, DFO and ICP diesel solvent, which contained 1,000 mg kg^{-1}, sulphur. The total volumes of the samples were 500 ml and the dilution was carried out as presented in Table 1.

Table 1. Dilution ratios of the B20 samples

Sample	RME (ml)	DFO (ml)	Diesel solvent (ml)
B20 1	100	400	0
B20 2	100	360	40
B20 3	100	320	80
B20 4	100	280	120

The analyses carried out for these four samples were the oxidation stability (OSI), acid number (AN) and kinematic viscosity (KV). The samples were analysed straight after mixing and after 4, 8 and 12 weeks of storage. The storage conditions were a dark cupboard at room temperature (around 20 °C). The OSI, AN, and KV of both RME and DFO were determined before mixing them together and adding high sulphuric diesel solvent. For the prepared sulphur containing B20 samples, the sulphur contents were measured straight after mixing. After the whole storage time, the elemental analysis (CHNO) was carried out for the samples.

RME used as the bio component in the fuel blends was a product of ecoMotion GmbH, Germany. RME is ISCC EU certified and meets Standard EN14214:2012 (SFS-EN 14214:2012, 2012). Fuel had been produced three years before this study and delivered to the University of Vaasa in 2012. Still, RME fulfilled the requirements of Standard EN 14214:2012 in terms of the properties measured in this study. The ester content of RME was not measured. Any information about the most likely added antioxidant of RME was not available.

DFO was a product of Neste and it had been produced in Finland. It did not contain any bio component before mixing it with RME. DFO contained 6.6 mg kg^{-1} sulphur.

CONOSTAN Premisolv™ ICP Solvent containing 1,000 mg kg^{-1} sulphur was a product of Conostan®, Canada. Conostan® is a division of SCP SCIENCE. According to the safety data sheet of this solvent, it contained sulphur as n-dibutyl sulphide in diesel fuel.

Acid number

The acid number of the blends was analysed by a titrator Metrohm Titrando 888. The method is a potentiometric titration method. The sample is diluted with iso-propanol and titrated by potassium hydroxide. The measurement was produced according to Standard EN 14104:2003 (SFS-EN 14104, 2003).

Oxidation stability

The oxidation stability was measured by a Biodiesel Rancimat 873 instrument. The method describes the accelerated oxidation stability of biodiesel. The sample is heated and air flow is conducted through it. The vaporizing compounds of the sample drift with air into water and the conductivity of the water is measured. The end point is achieved when the conductivity increase is at its highest. The method is described in Standard EN 15751:2014 and according this Standard the maximum induction period is 48 hours (SFS-EN 15751, 2014).

Viscosity

The viscosities were measured by a Stabinger SVM 3000 rotational viscometer. The measurement is based on torque and speed measurements. The device calculates the dynamic viscosity from the rotor speed. The device also has a density measuring cell that employs an oscillating U-tube principle. The kinematic viscosity is calculated automatically based on these measurements (Anton Paar, 2012).

Elemental analysis

The elemental analysis was measured by a Thermo Scientific Flash 2000 instrument. It determines the mass fractions of carbon, hydrogen, nitrogen and oxygen. Sulphur may also be analyzed in case its amount is high enough. The analysis was produced according to Standard ASTM D5291 (ASTM D5291-16, 2016).

Sulphur content

The sulphur contents were measured by a Thermo Scientific, iCAP 6500 duo instrument. In this method, the samples are decomposed by an ultrasonic oven and transformed from organic to water-soluble form. The solution is injected to spectrometers plasma. Calibration was made by the known concentrations of the standards. The defined standards (emission intensities in known concentrations) and the presence and concentration of the element are evaluated by comparing the intensity of the light to these standards. The method was an in-house method produced based on Standard ISO 10478 (ISO 10478, 1994).

RESULTS AND DISCUSSION

The analyses results related to storage stability are presented in Table 2 and elemental analyses results in Table 3. All the obtained results are arithmetic means of at least two replicate measurements. The OSI, AN and KV results are also depicted in Figs 1, 2 and 3, respectively, and the elemental analyses in Fig. 4.

Table 2. The storage stability analyses results for DFO, RME and B20 fuels

Property	DFO	RME	B20 1	B20 2	B20 3	B20 4
Oxidation stability (h)						
Fresh	72*	11	45	47	47	51
4 weeks			44	47	51	54
8 weeks			42	48	52	55
12 weeks		10	42	47	53	56
Acid number (mgKOH g^{-1})						
Fresh		0.19	0.07	0.06	0.06	0.07
4 weeks			0.06	0.06	0.06	0.06
8 weeks			0.07	0.06	0.08	0.07
12 weeks			0.07	0.07	0.07	0.07
Kinematic viscosity, 40 °C (mm^2 s^{-1})						
Fresh	3.5	4.5	3.6	3.4	3.2	3.1
4 weeks			3.6	3.4	3.2	3.1
8 weeks			3.6	3.4	3.2	3.1
12 weeks			3.7	3.4	3.2	3.1

The relative standard deviations are: KV 1%, AN 7.9%, and OSI 4.5%. These had been determined for the analysis methods earlier. For the OSI results above 48 hours, the reproducibility is approximately 9.5 hours. The OSI results above 48 hours should be announced >48 hours according to Standard EN 15751.
*The measurement was stopped after 72 hours.

Table 3. Elemental analyses result for B20 fuels

Property	B20 1	B20 2	B20 3	B20 4
C (m-%)	83.7	83.5	84	83
H (m-%)	13.2	13.2	13.8	13.7
N (m-%)	0.2	0.2	0.2	0.2
O (m-%)	2.2	2.2	2.2	2.2
S (mg kg^{-1})	6	76	149	226

The relative standard deviations for these methods were not determined.

Fig. 1 shows how the oxidation stability of the samples changed during 12 weeks:

- B20 1, decreased 5% (from 46 to 42 h)
- B20 2, remained almost constant (varied between 47 and 48 h)
- B20 3, increased 12% (from 47 to 53 h)
- B20 4, increased 10% (from 51 to 56 hours).

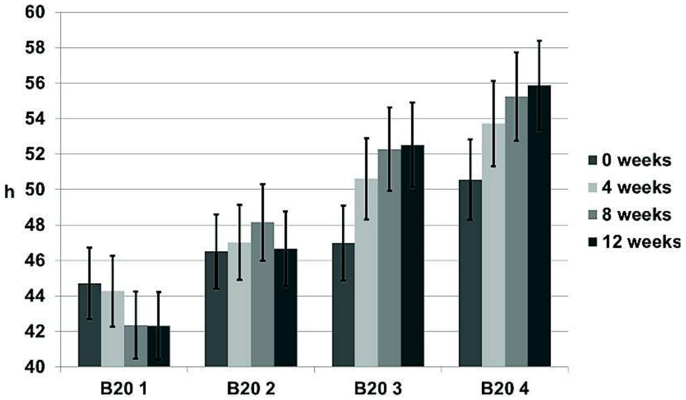

Figure 1. Oxidation stability results for B20 fuels. The sulphur concentrations were 6 mg kg^{-1} (B20 1), 76 mg kg^{-1} (B20 2), 149 mg kg^{-1} (B20 3), and 226 mg kg^{-1} (B20 4).

For the OSI results of above 48 hours, the reproducibility is approximately 9.5 hours. According to Standard EN 15751:2014, the OSI results of above 48 hours should therefore be announced as > 48 hours. In this study, the trend in the results was, however, so clear that the actually measured values were chosen to announce.

It should also be noted that, as fresh, the OSI was the longer, the higher was the sulphur content.

Fig. 2 shows that the acid numbers of all the samples remained within 0.06–0.08 mgKOH g^{-1} during the whole study. There were no significant differences between the samples and the results did not change during the storage. Thus, the sulphur content did not either seem to affect the AN.

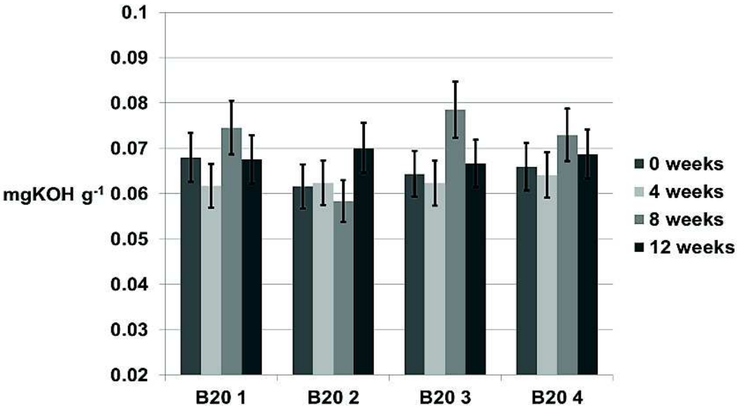

Figure 2. Acid number results for B20 fuels. The sulphur concentrations were 6 mg kg^{-1} (B20 1), 76 mg kg^{-1} (B20 2), 149 mg kg^{-1} (B20 3), and 226 mg kg^{-1} (B20 4).

Fig. 3 shows that the kinematic viscosities remained quite constant during the storage time of 12 weeks for all the samples. The differences between the samples were due to different mixing ratios. For the samples B20 2, 3 and 4, the diesel solvent containing sulphur lowered the KV in relation to its mixing ratio.

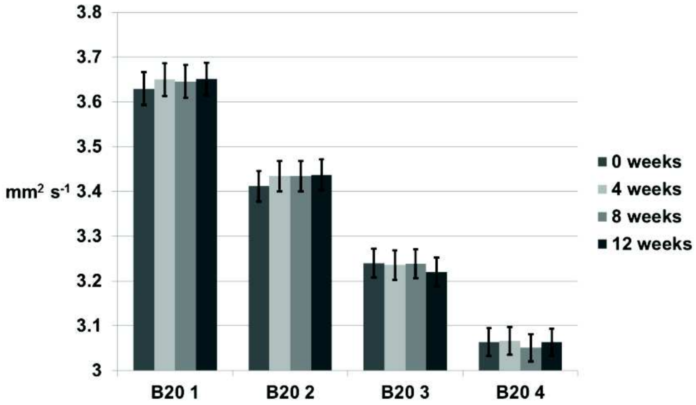

Figure 3. Kinematic viscosity results for B20 fuels. The sulphur concentrations were 6 mg kg^{-1} (B20 1), 76 mg kg^{-1} (B20 2), 149 mg kg^{-1} (B20 3), and 226 mg kg^{-1} (B20 4).

Pölczmann et al. (2016) studied the storage stability of diesel fuels containing biodiesel and made a mathematical correlation between a) the viscosity increase and oxidation stability decrease and b) between the acid number increase and oxidation stability decrease. As a result of their study, the authors conclude that the found correlation could reduce the number of needed analyses in biodiesel quality follow-up. According to them, the kinematic viscosity and acid number could be estimated based on the oxidation stability result (Pölczmann et al., 2016).

However, this was not clearly seen in the present study because no correlation was observed between the oxidation stability and acid number nor kinematic viscosity. In the current study, the amount of samples was, though, small and the timescale was shorter (12 weeks against 150 weeks in Pölczmann et al., 2016).

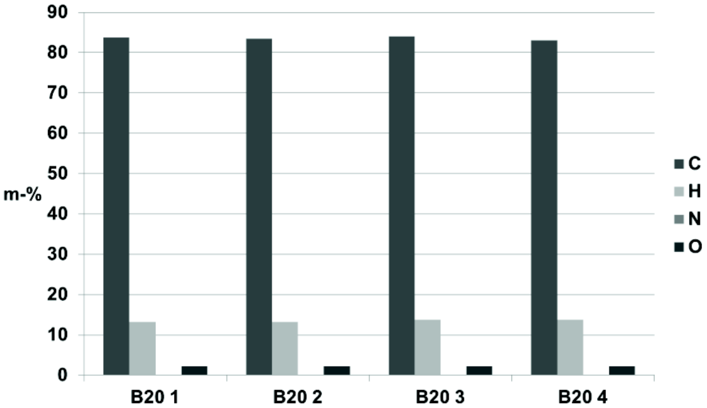

Figure 4. Elemental analyses result for B20 fuels. The sulphur concentrations were 6 mg kg^{-1} (B20 1), 76 mg kg^{-1} (B20 2), 149 mg kg^{-1} (B20 3), and 226 mg kg^{-1} (B20 4).

Fuels mainly consist of carbon and hydrogen and some small quantities of sulphur, nitrogen, and oxygen (Riazi, 2005). Even at its highest in the present study, the amount of added sulphur was not high enough to cause differences in elemental contents of the fuels, Table 2, Fig. 4. It can, thus, be assumed that adding diesel solvent did not affect the elemental composition of the samples. It needs, however, to be kept in mind, that the elemental contents are approximate. The measured sulphur values of 6, 76, 149 and 226 mg kg^{-1} were very close to the calculated values of 5, 78, 151 and 226 mg kg^{-1}.

The form of the sulphur compound is determining. In fossil fuel, sulphur can be found in cyclic compounds such as thiophenes and benzothiophenes as well as in noncyclic compounds such as mercaptanes and sulphides (Ma, 2010). In this study, the added sulphur was n-dibutyl sulphide which is a simple hydrocarbon chain that contains sulphur in the middle, and it is a noncyclic compound, Fig. 5.

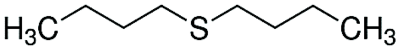

Figure 5. Structure of n-dibutyl sulphide. (Sigma Aldrich, 2016).

In the presence of water, sulphur may form sulphuric acid and the acids will promote oil thickening and oxidation, e.g., in lubricating oils (Folkson, 2014). This phenomenon may also occur within fuels. Biodiesel is hydrophilic and it is possible that its moisture content will increase during three years of storage. The water content of RME was not measured. Most likely, based on the other analysis results, it can be assumed that no water was present in the studied samples and the increasing amount of sulphur did not affect adversely the oxidation stability, quite the contrary.

McCormick & Westbrook (2007) mentioned that sulphur compounds can work as antioxidants and the same conclusion is also supported by the studies of Karavalakis et al. (2010) and Sirviö et al. (2016). Sirviö et al. (2016) was a prestudy in relation to this research theme. In that study, the B20 sample containing more sulphur loosed its oxidation stability less than the B20 sample containing less sulphur. Karavalakis et al.(2010) have found that the oxidation stability behaviour of biodiesel blends is a very complicated process. They say that the most important factors affecting the blends' oxidation are the biodiesel composition and used antioxidants. Additionally, according to McCormick & Westbrook (2007), the stability of the biodiesel is the most important factor from the point of view of the blends storage stability.

In this study, the biodiesel and fossil diesel were the same in all samples but the mixing ratios of the diesel solvent containing sulphur varied from 0 (B20 1) to 22% (B20 4). Most likely, the reason for the difference in the oxidation stability of the fresh blend samples was, thus, the different sulphur content. For the diesel solvent, the oxidation stability was not measured but the oxidation stability of the DFO was high (measurement was stopped at 72 hours). The DFO quality was, thus, not a threat to the stability of the blends.

As mentioned above, the decrease in OSI was 5% for the sample B20 1 while the oxidative stability increased by 12 and 10% for the samples B20 3 and 4, respectively. This may indicate the antioxidative effect of sulphur. To prove this thesis, again indicated by the current study, the chemistry of the fuel blends and organic sulphur should be studied more detailed.

Karavalakis et al. (2011) showed in another study, that even a 4–6 weeks storage time may cause OSI to decrease below the 20 hours' limit due to natural ageing. The 20 hours' limit is set in Standard EN 590:2013 for automotive diesel fuels. In this study, the OSI of all the samples remained clearly above the 20 hours' limit for 12 weeks. The other measured properties were also at a proper level though the adopted RME was produced 3 years before this study.

CONCLUSIONS

The aim of this study was to find out if increasing sulphur content is beneficial for storage stability of the fuel blend. Four B20 fuel samples were produced consisting of 20 vol% biodiesel and 80 vol% fossil diesel. All the samples were prepared from rapeseed methyl ester (RME), diesel fuel oil (DFO) containing 6.6 mg kg^{-1} sulphur, and a high sulphuric diesel solvent (n-dibutyl sulphide in diesel fuel). The measured sulphur contents of the samples were 6, 76, 149 and 226 mg kg^{-1}. The effects of the sulphur content on the oxidation stability, acid number (AN) and kinematic viscosity (KV) of the blends were investigated. Based on the study, the following conclusions could be drawn:

- There is a reason to assume that the sulphur may be favourable to fuel blends' storage stability. The decrease in oxidation stability was 5% for the sample B20 containing the lowest amount of sulphur while the other samples behaved differently. During the storage, the oxidation stability of the sample containing 76 mg kg^{-1} sulphur remained quite constant and for the samples containing 149 and 226 mg kg^{-1} sulphur, the OSI increased by 12 and 10%, respectively. This may indicate the antioxidative effect sulphur has. The phenomenon and chemistry should, however, be studied more thoroughly and for a longer period to confirm this statement.
- The other properties related to storage stability, i.e., AN and KV seemed not to vary much within the storage time of 12 weeks. Any clear correlations between the oxidation stability and the acid number and kinematic viscosity were not observed in the examined samples.

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