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Limitary State of Heavy-Duty Engine Oils and Their Evaluation According to the Change of Tribological Properties during Operation

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Abstract: The tribological properties of engine oils for heavy-duty trucks are evaluated, taking into consideration their variation during operation. After testing new oils or oils after a 2500 km and 5000 km run, there were no essential differences in their tribological properties at lower loads, but at higher loads and longer durations of operation, significant differences were found, including increased friction losses and the reduced surface wear protection ability of the oils. There are two main reasons for this reduced ability of the tested oils to form a boundary lubrication layer: the consuming of the functional additives and the aging of the oil, i.e., oxidation and an increase in acidity. Research data show a close relationship between the increasing acidity and surface wear.

Keywords: limitary state of engine oil; heavy-duty; friction torque; wear; oil acidity

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1. Introduction

The degradation of the lubricant which takes place during operation is an important factor in the longevity and performance of lubricated machinery. This is influenced by different parameters such as the operation temperature and contamination. The applied formulated oils consist of a base oil and additives that improve the oils' operating properties, including a reduction in the oxidation rate. However, once the additives are consumed, the oil performance rapidly decreases, influencing the life cycle of a machine [1–3].

The basic physical and chemical characteristics of the oil quality do not always reach the limit values at the oil drain interval, and in such cases, the engine oil is still suitable for future use. By knowing the chemical characteristics and intensity of the wear products on engine oil, it is possible to evaluate the engine performance parameters to determine the optimized oil change intervals and detect when the mechanical wear increases in order to ascertain the oil's lost lubricating properties and assess the technical condition of the engine systems [4].

However, the values of the tribological properties (i.e., friction losses and surface wear) deteriorate as the oil ages, which is indicated by changes in viscosity, oxidation and acidity and alkalinity and increased wear debris and soot formation. This leads to changes in the oil's parameters and structure. One of most important factors regarding the degradation is the oxidation of mineral engine oils, which takes place through the formation of oxidized aliphatics [5]. The increase in acidity changes the structure and lubricating properties of the oil. This chemical reaction is time-dependent and neutralization due to commercial additives cannot prevent the lubricant degradation due to the formation of sulfuric acid [6].

The indicator for oil degradation and deterioration of operational characteristics could be the increase in kinematic viscosity at 40 and 100 °C, which can explain the oxidation process occurring in the engine oil. However, to gain a more comprehensive understanding of the changes taking place in the oil, the dynamic viscosity, oxidation and acidity number should be monitored in the oil [7].

The viscosity measurements at 100 °C using on-board sensors show that the viscosity increase over 73 h testing cannot be related to soot loading and fuel contamination and is instead dependent on the depletion of the additives and incipient accumulation of oxidation by-products [8].

The operating oil is a bearer of information about the chemical, tribological, thermodynamic and other changes taking place both on the friction surface and in the lubrication system itself. During the operation of the engine, the wear particles of organic (hydrocarbon) and inorganic (wear debris) products represent one of the main types of oil contamination [9]. Besides the wear products, there are several indicators (i.e., oxidation, nitration, sulfonation, acidity number, total base number, soot content, etc.) for the physical and chemical monitoring of the lubricating oils, using such methods as multivariate techniques of infrared spectroscopy [10], Fourier transform infrared spectroscopy [11], dielectric spectroscopy [12], laser-induced breakdown spectroscopy [13] and others. Engine oil monitoring methods can be used not only for the maintenance of the lubrication system, but also for the qualitative evaluation of different oils according to the longevity of their efficiency.

The investigations of the internal combustion engines' lubrication show that low-viscosity engine oils could be used for application in heavy-duty transport machinery, increasing the oil drain interval and reducing the maintenance cost. However, in some cases, a higher oil degradation in terms of oxidation and nitration could take place [14]. Therefore, is important to use the chemically active oil additives which interact with and neutralize the engine oil's contaminants and oxidative by-products of oil degradation (i.e, ketones, high aldehydes and carboxylic acids). Such additives include dispersants, detergents (surfactants), oxidation inhibitors and anti-wear additives, and their performance mechanism is quite complex [15]. The most important of the functional additives is the tribotechnical anti-wear additives, which can improve the tribological characteristics of the lubricants. The efficiency of specific functional additives, such as nano-particles [16], metal-cladding [17], surface layer activation [18], commercial anti-wear [19] and many other types of additives, have been investigated in several studies.

Previous investigations show that the acidity of the lubricating oil increases significantly during the lubricant operation; it can rise by up to 4 times after 12 months of operation compared to fresh oil [20]. However, such investigations do not include the correlation with the tribological properties of oil.

The aim of this study is to investigate how to evaluate the tribological properties of engine oils for heavy-duty trucks while taking into consideration their variation during operation and a determination of their efficiency-limiting factors.

2. Materials and Methods

2.1. Materials

The evaluation of the engine oils' tribological properties and their variation was performed at operating conditions for three multipurpose armored heavy-duty trucks designed for various supply tasks in extreme driving conditions. Diesel trucks of same model with high terrain passability and high carrying capacity manufactured in 2010 and in approximately the same technical condition (mileage until the start of the tests: 28,923 km, 29,857 km and 27,782 km) were tested. Before the start of the tests, all trucks underwent technical maintenance. All three trucks were operated under almost identical operating conditions while transporting cargo, without normal driving conditions and under off-road driving conditions on the training ground. The oil samples for the tribological evaluation were taken after 2500 km of work and after the next stage at the same operating conditions when the total service was 5000 km.

The types of engine oils which were investigated in this research are presented in Table 1.

Table 1. Marking and description of investigated engine oil.

Marking of Engine Oil	Description of Engine Oil
MINERAL1	Mineral engine oil 15W40 OE/HDO for heavy-duty diesel engines, including turbo-charged engines, operating continuously long time under hard conditions.
MINERAL2	Mineral engine oil 15W40 API CI-4 for advanced high-speed powerful turbo diesel engines
SEMISYNTH	Semi-synthetic engine oil 10W40 API CI-4 for advanced high-speed powerful turbo diesel engines

2.2. Tribological Testing and Friction Surface Characterization

Tribological tests of friction torque and surface wear according to the wear scar diameter (WSD) were performed using the four-balls testing machine at room temperature. The testing methodology used was in accordance with the standard DIN 51 350 [21]. The diameter of the steel balls used in the test was 12.7 mm. According to the standard, the tests were carried out at 150 and 300 N of loading, choosing 300 N as the main loading for heavy-duty trucks' engine oil and 150 N for the comparative evaluation of their tribological properties. The tests were performed with oils which were new and with oils which tested after being operated for 2500 and 5000 km runs of the trucks. The wear resistance of the different oils was evaluated according the measured WSD and calculated wear volume of the balls.

The investigation of the different physical and chemical properties of the lubricants was performed according to standard methodologies (Table 2).

Table 2. Methodology standards for tested engine oil properties.

Parameter	Measurement Method
Kinematic viscosity, at 100 °C, cSt	ASTM D445
Kinematic viscosity, at 40 °C, cSt	ASTM D445
Viscosity index	ASTM D2270
Acidity value, mg KOH/g	ASTM D664
Total Base Number, mg KOH/g	ASTM D2896
Pour point, °C	ASTM D97

The surface morphology of the friction surfaces was characterized by scanning electron microscopy (SEM, Hitachi S-3400N). The chemical composition of the samples was determined by energy dispersive X-ray spectroscopy (EDX, Bruker Quad 5040).

3. Results

3.1. Tribological Testing

The friction torque was measured during the tests at two different loading versions for each tested oil and is presented in the graphs in Figure 1.

The analysis of the graphs shows that for the testing of new oils at 150 N of load there are no significant differences in the friction torque for all three tested oils (Figure 1a,c,e); it was stable (45–55 mNm) during the entire test. When testing at a load of 300 N, important differences emerged (Figure 1b,d,f). Only the MINERAL1 oil ensured a stable friction torque of 110–115 mNm during the entire test period. The friction torque increased significantly to 150–160 mNm for the MINERAL2 (after 20–30 min) and SEMISYNTH oils (after 5–10 min). This demonstrates the lower performance of the lubricating properties of these oils (especially the semi-synthetic oil SEMISYNTH) at higher loads compared to MINERAL1.

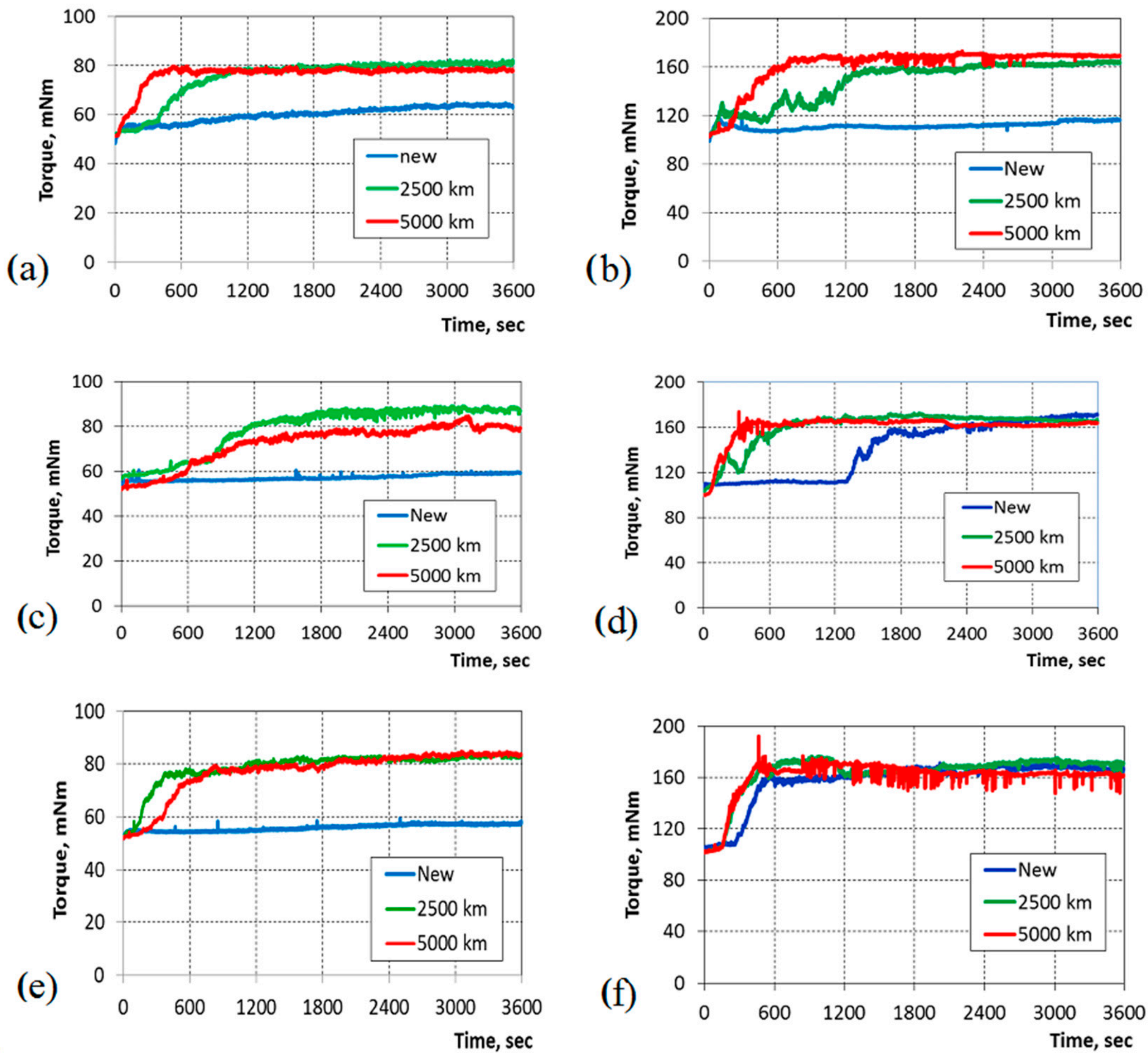


Figure 1. Changes in friction torque during tests for different engine oils, loads and run distances: oil MINERAL1 at 150 N (**a**) and 300 N (**b**) loads; oil MINERAL2 at 150 (**c**) and 300 N (**d**) loads; and oil SEMISYNTH at 150 N (**e**) and 300 N (**f**) loads.

The variations in the friction torque of the tested oils after a 2500 km and a 5000 km run constitute extremely informative tribological testing results. The value of the friction torque started to differ and change from 7 min onwards (especially at 5000 km) and fluctuated until the end of the test. The variation range for the oils in the 5000 km run was about 10–15 mNm. This testifies to the unstable friction conditions in the contact and possible fitful breaking of the boundary lubrication layer. The changes in friction torque found were also remarkable in tests of the oils that were ran for 2500 km, but further investigations of the surface wear show that the advantages and disadvantages of the tested oils, according to the wear decrease abilities, were apparent only after the 5000 km run, especially under higher load (300 N) conditions.

When analyzing the results of the surface wear of different oil samples (Figure 2), we found that the loading of 150 N does not reveal which oil is better, taking into consideration both the new oil and the oil after a 2500 km and 5000 km run. Moreover, when evaluating the wear spot of the new oil, the worst results were found with MINERAL1 (WSD was 0.33 ± 0.01 mm), while the other two oils showed lower wear (WSD of 0.25 ± 0.008 mm).

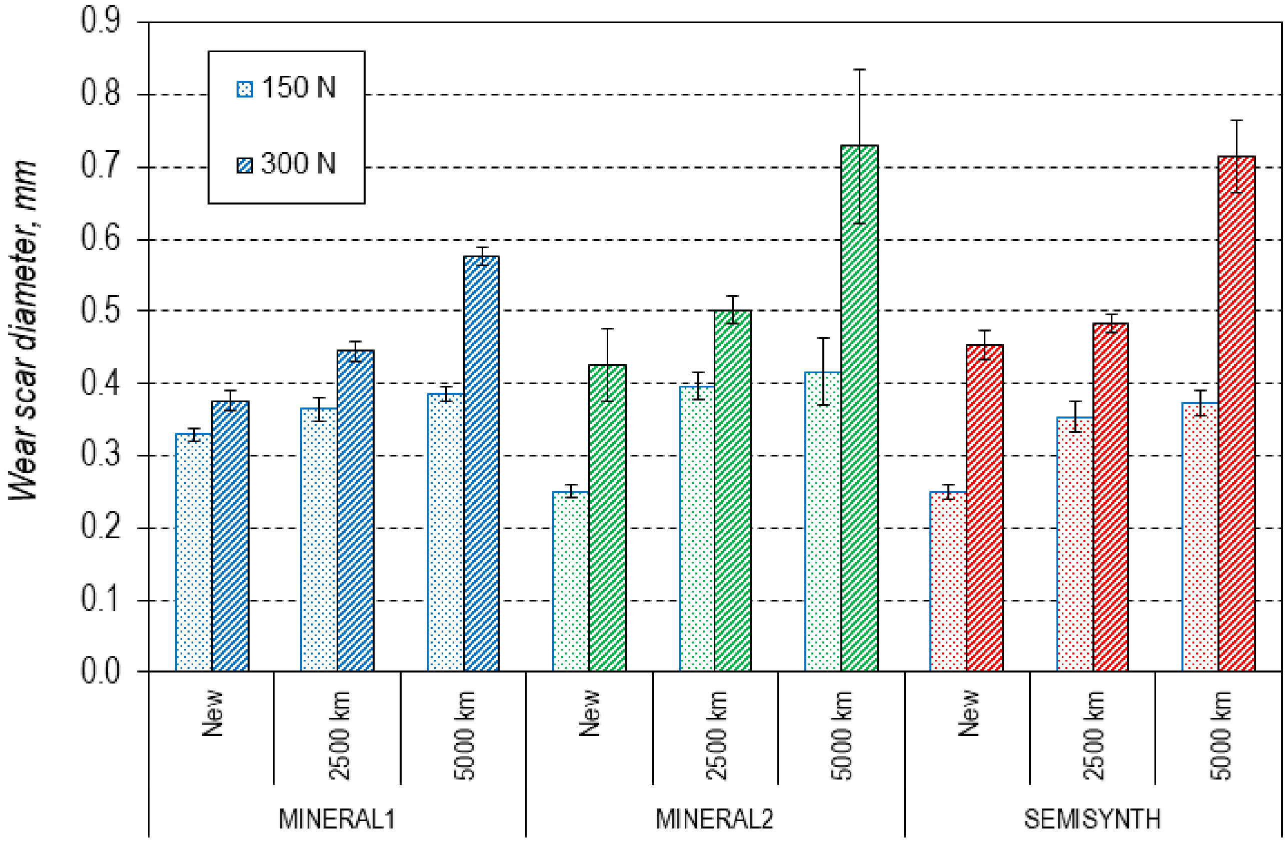


Figure 2. Results of wear scar diameter for different engine oils, loads and run distances.

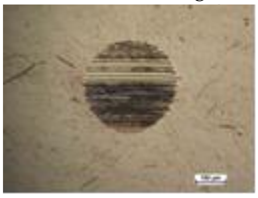
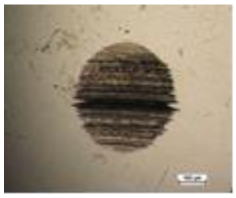
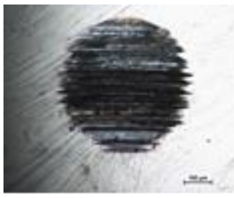
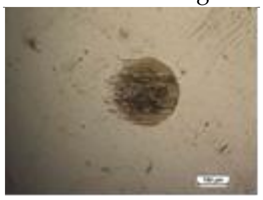
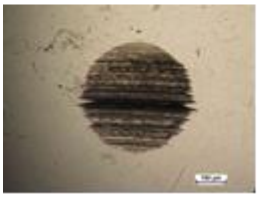
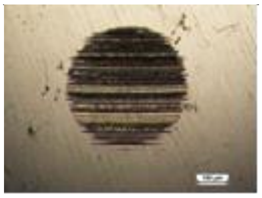
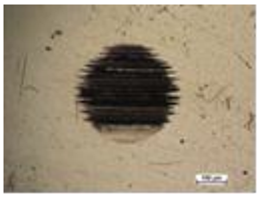
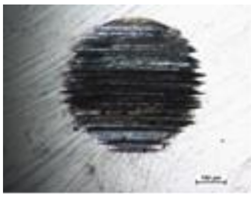
The actual lubrication efficiency of the oils according to the surface abrasion became evident in the case of higher load at 300 N, especially after a longer run. Even the new oil MINERAL1 displayed the best wear protection at 300 N of load (0.38 ± 0.01 mm WSD) compared to the MINERAL2 (0.43 ± 0.03 mm) and SEMISYNTH (0.45 ± 0.02 mm) oils. The difference is more pronounced after 5000 km: the MINERAL1 oil had an average wear scar diameter of 0.58 ± 0.01 mm, while that of MINERAL2 was 0.73 ± 0.08 mm and that of SEMISYNTH was 0.71 ± 0.05 mm.

3.2. Friction Surface Investigation

Lighter and heavier working conditions of friction contact were clearly shown for different oils when exploring the surface morphology in the pictures of wear spots (see Table 3).

If there is no significant difference in the wear spots after the 150 N loading tests, then at 300 N of load, the friction surfaces in the MINERAL2 and SEMISYNTH oils are characterized by increased scratches and deeper scars, and when testing the oils after being run for 5000 km, there is an obvious, significant change in the operating conditions in the contact: the short- or long-term damage of the boundary layer that causes more intense surface abrasion.

Table 3. Wear spots in the samples after the tests for different engine oils, loads and run distances.

Test Options	New	2500 km Run	5000 km Run
	Engine oil MINERAL1		
150 N loading			
300 N loading			
	Engine oil MINERAL2		
150 N loading			
300 N loading			
	Engine oil SEMISYNTH		
150 N loading			
300 N loading			

3.3. *Chemical Analysis of Friction Surface*

In order to find the regularity of the changes in the properties of oils, we conducted an SEM analysis of the damaged surfaces and evaluated the physical mechanical properties of the oils.

Table 4 provides data on the amounts of some of the more important elements found in the wear scars (X-ray spectroscopy, %) of the tested samples, and Table 5 displays the elements' maps in the wear scars after being run for 2500 km.

Table 4. Chemical composition of most important elements at the wear scar of the samples.

Lubricants		Elements, Atom. C [at. %]			
		Phosphorus (P)	Zinc (Zn)	Calcium (Ca)	Sulphur (S)
MINERAL1	New	2.61	3.32	1.72	1.13
	2500 km	1.74	3.71	0.95	3.13
	5000 km	0.32	2.92	1.19	1.22
MINERAL2	New	2.36	4.20	1.16	2.56
	2500 km	1.56	3.96	1.04	2.53
	5000 km	0.38	2.64	0.51	2.14
SEMISYNTH	New	2.08	4.06	1.72	4.00
	2500 km	1.79	3.18	1.12	2.21
	5000 km	0.51	3.87	0.74	2.45

Table 5. Elements' maps at the tested samples after the tests with the tested oils.

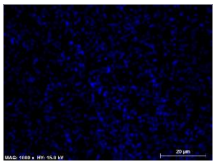
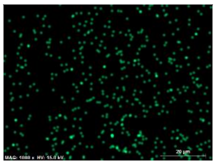
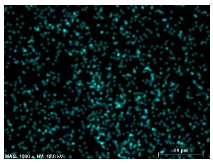
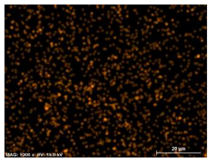
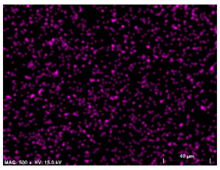
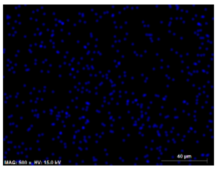
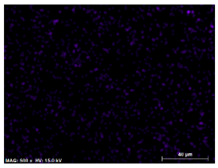
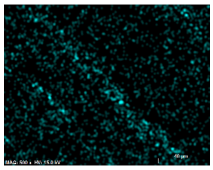
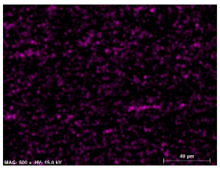
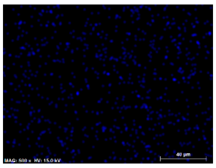
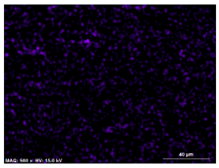
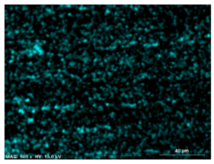
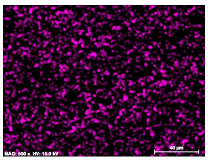
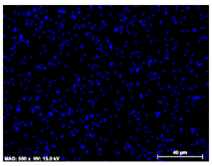
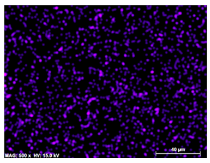
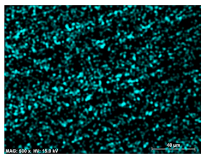
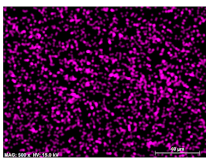
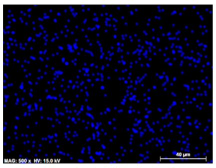
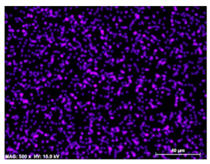
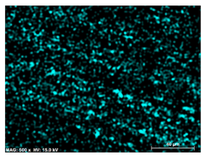
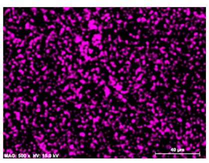
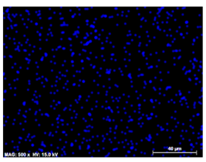
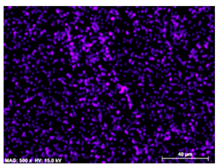
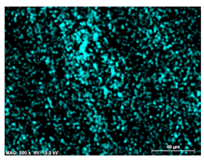
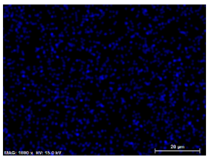
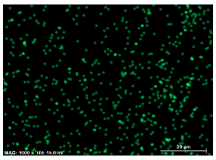
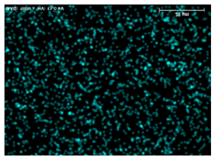
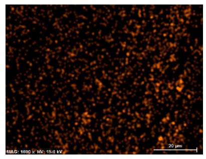
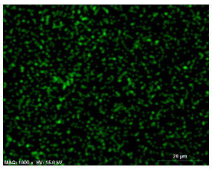
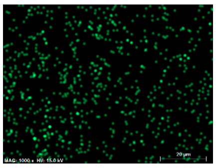
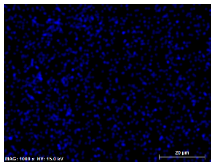
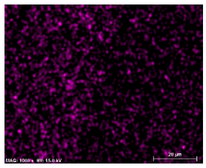
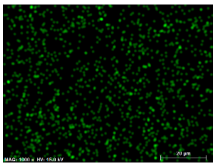
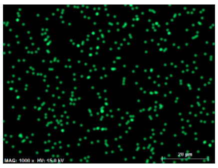
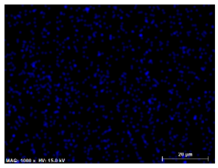
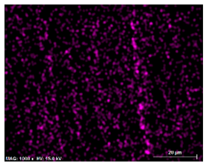
Lubricants	Elements, Atom. C [at. %]			
	Phosphorus (P)	Zinc (Zn)	Calcium (Ca)	Sulphur (S)
		New		
MINERAL1				
MINERAL2				
SEMISYNTH				
		2500 km		
MINERAL1				
MINERAL2				
SEMISYNTH				

Table 5. *Cont.*

Lubricants	**Elements, Atom. C [at. %]**			
	Phosphorus (P)	**Zinc (Zn)**	**Calcium (Ca)**	**Sulphur (S)**
		5000 km		
MINERAL1				
MINERAL2				
SEMISYNTH				

Chemical analysis of the elements on the wear spots shows that P and Ca decreased with operation time, while there was no clearly expressed tendency for a content change in Zn or S. According to the mapping of the elements, we can see that P and S were distributed in the wear scar according to the direction of the trace of wear, which cannot be said about the Zn and Ca distributions. This could be related to the chemical and physical properties of these elements, which are related to the thermal and elastic conditions in the friction contact.

3.4. Physical and Chemical Properties of the Oils

Our analysis of the physical and chemical properties of the tested oils helps to explain the changes in the operational properties of the oils during the longer working period. Therefore, the most important oil properties were measured, as presented in Table 6.

Most of the physical properties of all of the investigated oils showed a similar trend, for example, kinematic viscosity decreased in the beginning of the oil's operation and was later more or less stable, and the change in the viscosity index and pour point was not significant. However, the change in the acidity number could influence the operating properties of the oils.

The oils MINERAL2 and SEMISYNTH, which showed worse tribological properties, had a clearly increasing trend in acidity. The increasing alkalinity (total base number) of these oils also had an influence, increasing wear after 5000 km of operation. At the same time, the acidity and alkalinity changes of the engine oil MINERAL1 were not very drastic.

Table 6. Physical and chemical properties of the tested oils.

Lubricants	Tested Oil			
	Run, km	**MINERAL1**	**MINERAL2**	**SEMISYNTH**
Kinematic viscosity, at 100 °C, cSt	New	15.47	13.85	14.19
	2500 km	13.46	13.27	13.06
	5000 km	13.38	13.28	12.91
Kinematic viscosity, at 40 °C, cSt	New	113.45	102.85	97.34
	2500 km	96.49	95.17	88.64
	5000 km	99.40	92.85	84.15
Viscosity index	New	146.6	135.6	149.7
	2500 km	139.6	138.7	146.8
	5000 km	133.6	142.8	153.0
Acidity value, mg KOH/g	New	1.68	1.60	1.66
	2500 km	1.76	1.72	1.78
	5000 km	1.83	2.20	1.96
Total Base Number, mg KOH/g	New	11.49	11.02	11.53
	2500 km	11.41	12.84	11.42
	5000 km	11.76	13.17	12.68
Pour point, °C	New	−38	−45	−46
	2500 km	−41	−42	−43
	5000 km	−43	−44	−42

4. Data Analysis and Discussion

The data on the wear resistance (Figure 2) and character of the surface wear (Table 3) of different lubricants show that the oil behaviors of separate oil types could differ for various loads. This means that the properties of engine oils can be formulated for different load operation conditions. Regarding the difference in wear resistance, it can be argued that, in the range of lower loads, all of the tested oils had similar tribological characteristics. In view of the fact that the oils are used in heavy-duty trucks in extreme conditions, i.e., often close to the limits of their possibilities, tests were carried out at a load of 300 N. At this load, after 2500 km of operation, significant differences were found in the tribological characteristics of the lubricants. The main difference is the reduced ability of the oil to protect the friction surface from abrasion.

The behavior of the oils' tribological properties is load- and time-dependent. The oil can show comparably good wear resistance at lower loads at the beginning of its services (Figure 2), but due to oil aging, the tribological properties can sharply worsen. This is clearly evident for higher loads, which is characteristic for heavy-duty trucks.

However, the wear evaluation according to the wear scar diameter on the ball has the disadvantage if the wear is evaluated during the time period, because the increase in the wear spot diameter is not linearly dependent on the wear volume of the ball. Therefore, when evaluating the changes in the wear during the operation, knowledge of the specimen wear volume would be particularly informative. This geometrically recalculated dependence is presented in Figure 3. Decreased wear resistance is clear at 150 N of load, but it is especially obvious if we analyze the abrasion data at the load of 300 N. A rapid increase in the wear volumes between 2500 km and 5000 km of oil operation also takes place for the oil MINERAL1, which produced the best wear resistance results, and this increase reached 2.9 times. However, we found an evident increase in the oils SEMISYNTH and MINERAL1, where the wear increased by 4.2 and 4.9 times, respectively. These results show that some important changes in the tribological properties of the oils take place between 2500 km and 5000 km of operation at higher loads.

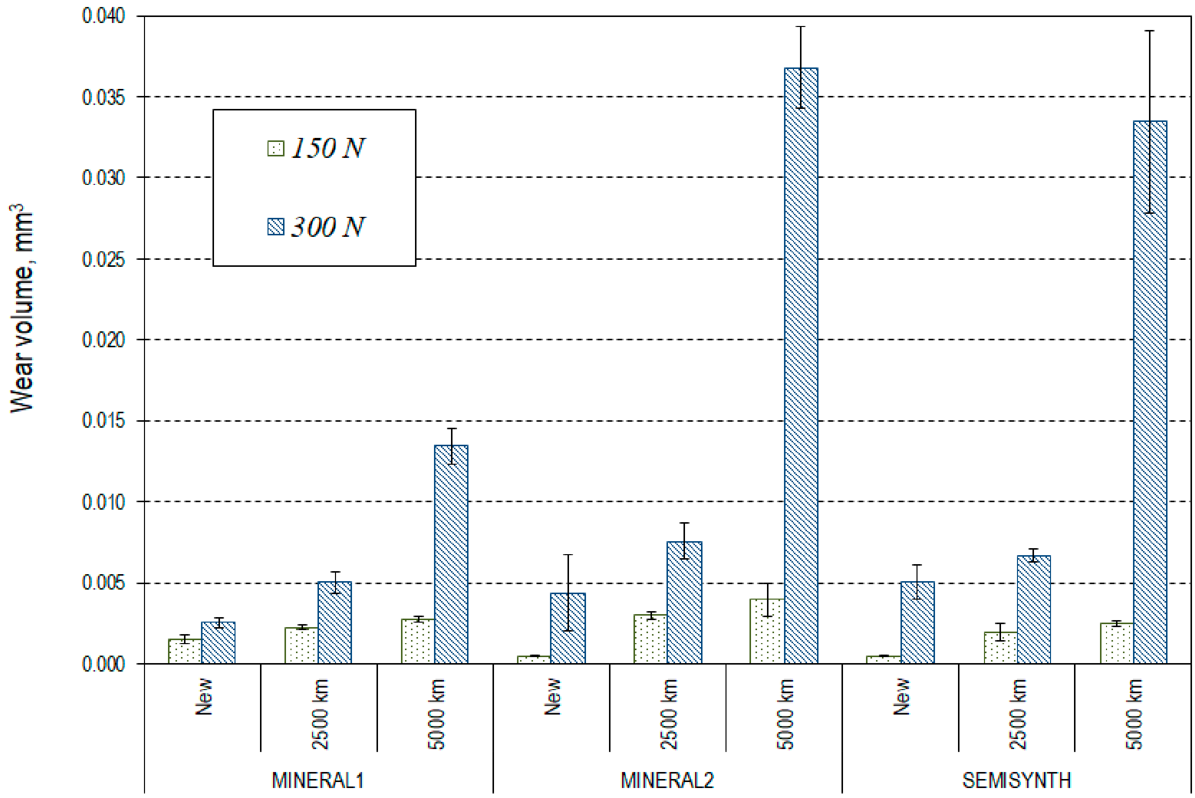


Figure 3. Results of the samples' wear volumes at 150 and 300 N loads for different types of engine oil.

Comparing the testing at different loads, it could be concluded that at a load of 150 N, no essential difference was determined, neither in terms of the friction losses nor in the surface wear when using new oils or oils after 2500 km and 5000 km of operation in trucks. However, when testing at higher loads (300 N), significant differences were found in the tribological characteristics of the lubricants, especially in terms of wear resistance ability during operation, according to the wear volume. No significant discrepancies in wear volume were observed among the oils while running until 2500 km, but at 5000 km of operation, there were substantial differences: for MINERAL2, the wear increased by 2.7 times, and for SEMISYNTH, the wear increased by 2.5 times, both compared to MINERAL1. This important loss in the oils' lubricating properties after the 2500 km run shows that the oil change interval for heavy-loaded engines should be shorter or those oils should be not recommended to be used in the engine systems of heavy-duty trucks.

During operation, the oil is impacted by the variable temperature, friction surface material (for example, metals that promote oxidation) and by-products of the operation process (combustion products at the operation in internal combustion engine). Oil-influencing environmental factors include moisture, air oxygen and sunlight. Temperature has the greatest impact on lubricating oil oxidation [22,23]. Higher loading during the operation of the heavy-duty engine oils together with increasing temperature can accelerate the formation and degradation of peroxides and hydro-peroxides in oil, and the increase in the catalytic effect of metals. That causes intense oxidation, aging and the loss of the tribological properties of the oil. Most oxidation products increase the acidity of the oil. The main signs of oxidizing oil are the increasing or decreasing viscosity and the increasing acidity. The investigation of the acidity could show the regularities of the oil aging and the loss of its lubricating properties in relation to the physical, chemical and mechanical properties of lubricant and surface. It should be noted that the acidity value can be influenced by the concentration of trace metals present in the additives of lubricants [24,25]. The impact of trace metal in additives used in tested commercial oils, e.g., ZDDP, could partially influence the acidity measurement results, especially for mineral oils, but such influence could be exactly measured only in the case of the availability of the clear content of additives, which is not the case for commercial additives. In addition, our results show that the changes in metals content in the friction surface is not considerable in most cases (Table 4).

The influence of aging on oils' tribological properties can be expressed in the changes in oil acidity. Figure 4 shows the rising trend in acidity after 5000 km of the trucks' operation.

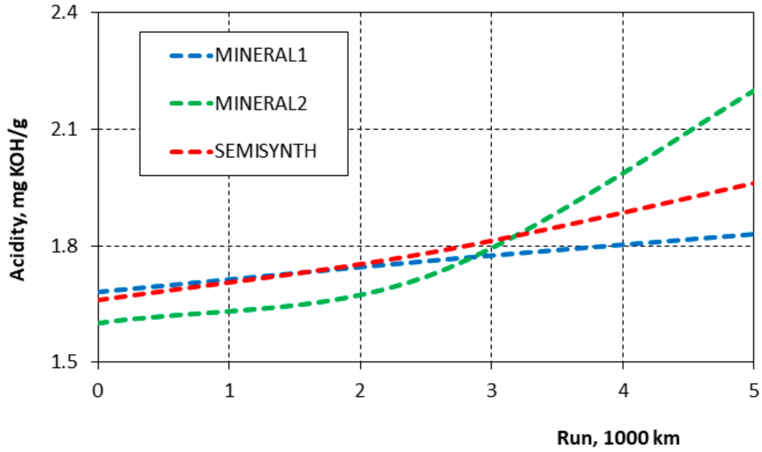


Figure 4. Change in acidity of different engine oils after 5000 km of operation.

It can be clearly seen that, starting from 3000 km of operation, the acidity of MINERAL2 and SEMISYNTH increased significantly. This correlates with the friction measurements and wear tests. The wear results after 2500 km of operation differed insignificantly for all tested oils, but after 5000 km, the wear was lowest for MINERAL1, which had the highest stability in the acidity value. The increasing acidity of the MINERAL2 and SEMISYNTH oils could have a direct influence on the oils' ability to form a lubricating film on the friction surface and their stability.

We found a remarkable correlation between the wear resistance of the friction pair and the acidity of the lubricant (Figure 5). Here, the loading plays an important role. When presenting the wear scar diameter results of the tested oils according to the acidity number, it can be clearly seen that a higher acidity has far more influence on the tribological efficiency at higher loading. If at 150 N of load the increasing acidity does not change the WSD significantly, then at the 300 N of load, the higher acidity almost guarantees a higher abrasion of the friction surface.

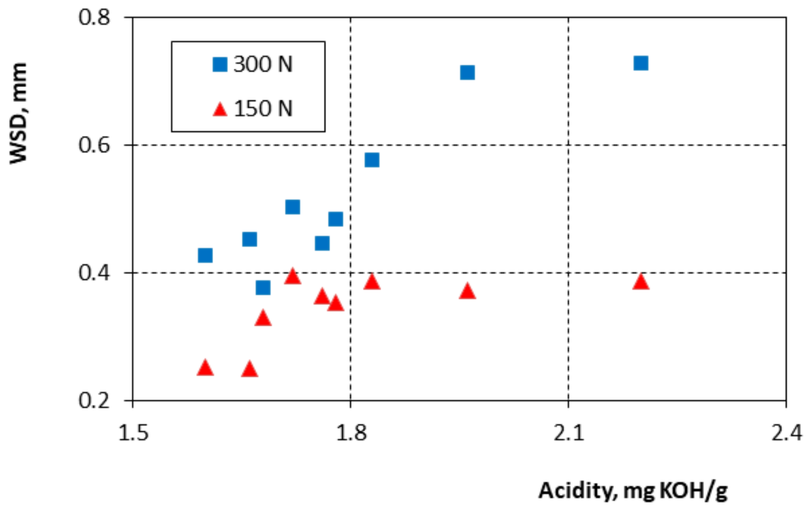


Figure 5. Distribution of wear results according to acidity in the 150 and 300 N loadings.

Analyzing the wear dependence regarding the acidity change according to the more informative wear measurement data, i.e., the wear volumes of the tested specimens (Figure 6), we can see that the trend of wear curves has very clear difference at the loads of 150 N and 300 N. At lower loading, the wear value did not increase significantly when the acidity rose and this is almost linearly dependent. However, this dependence drastically changes to the parabolic when the friction pair experiences higher loads. This dependence shows the importance of acidity changes for heavily loaded friction pairs.

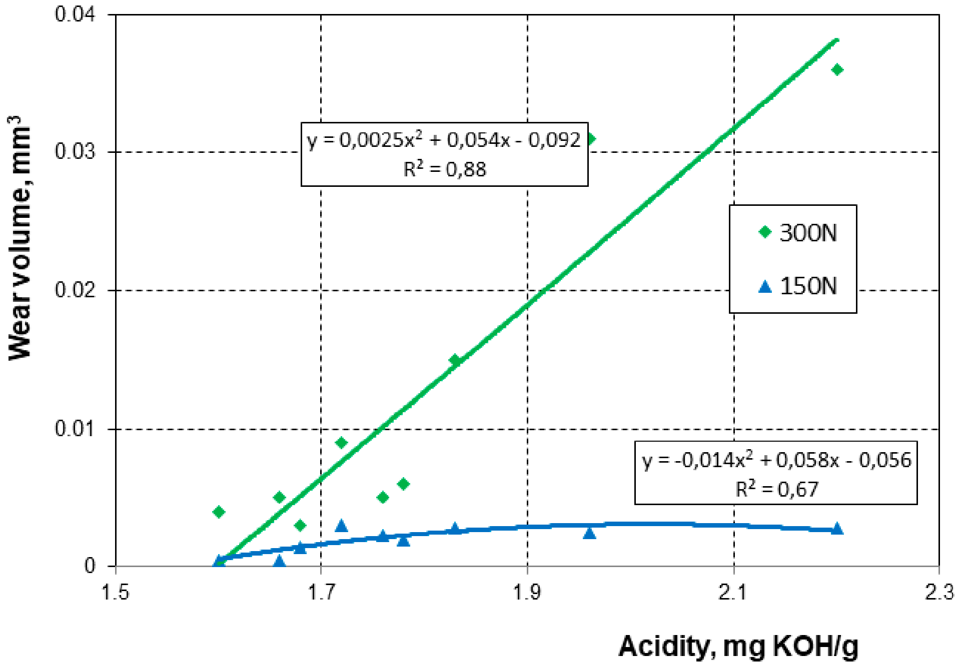


Figure 6. Dependence of wear volume on the acidity at the 150 N and 300 N loads.

A considerable number of studies shows the direct correlation between oil deterioration and the reducing tribological efficiency of lubricants [26–28]. The aging of lubricants causes the change in the lubricants' chemistry and the compounds formed at the friction surface [29]. That is the reason why higher acidity determines the lower lubricating properties and wear resistance ability of engine oils through the weaker boundary layer on the contact surface. This factor becomes especially important when the loading of the friction joints is higher, i.e., for heavy-loaded engines.

Therefore, it is very important to pay attention to oil acidity behavior during operation if oil producers want to assure the long-term efficient operation of their engine oils. The monitoring of acidity indicators and its control by neutralizing additives and oil filtering could be applied for this purpose [8,10–13,30].

5. Conclusions

1. The tribotechnical properties of the lubricants (friction losses and surface wear) are objectively estimated by determining these properties not only for the new lubricants but also for the variation in these properties during operation.
2. Tribotechnical tests for lubricants should be carried out on loads that are adequate for the operating loads in the investigated machines and equipment. The testing results in the surface wear at 150 N of load are obviously similar for all tested oils, but when higher loading (300 N) was applied, there were substantial differences in wear results after the 5000 km running of the trucks: the wear was higher by 2.7 times when applying the MINERAL2 oil and by 2.5 times when applying the SEMISYNTH oil, compared to the MINERAL1 oil. This shows that the oil change interval for heavy-loaded engines should be shorter or that those oils— MINERAL2

and SEMISYNTH—should be not recommended for use in the engine systems of heavy-duty trucks.

3. The main reason for the deterioration of the lubricating properties of the oils was the reduced ability to form a boundary lubrication layer. There are two main reasons for this loss in the oils we tested: the consumption of part of the functional additives that are responsible for this property and the aging of the oil, i.e., oxidation and the increase in acidity. Our research data show a close relationship between increased oil acidity and surface wear when testing at a higher load of 300 N. This correlation could be expressed more precisely if some additional factors, e.g., the content of the additives, could be found.

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