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Production of Used Engine Oils with Defined Degree of Degradation in a Large-scale Device: Correlation of Artificially Altered Oils with Field Samples

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Abstract: Lubricant degradation by additive depletion, contamination and build-up of degradation products deteriorates lubrication performance. It can be expected that engine oil degradation also has an impact on performance parameters. Engine development tends to higher power density, lower engine oil viscosity to optimize fuel efficiency, which heavily stress engine oils and thus demand increased oil performance. Correlations between engine oil degradation, friction and wear during an engine oil's lifetime are a valuable asset in engine development towards performance, longevity and cost reduction. Here, physical-chemical and tribological properties of engine oils collected from passenger cars were determined. The results revealed the deterioration of friction (loss of fuel efficiency) and wear (increasing loss of material) depending on mileage. Defined used engine oils were produced by artificial alteration in a large-scale device and showed a very good correlation with the field samples.

Keywords: Internal combustion engine oils; Oil condition monitoring; Wear; Friction; Artificial oil aging

1. Introduction

Internal combustion engines (ICEs) are predominant in road traffic vehicles, such as commercial vehicles and passenger cars. The global vehicle fleet is continuously growing, estimations for 2015 showed 947 million passenger cars and 335 million commercial vehicles worldwide [1]. In 2016, a total of 257 million passenger cars and 38.7 million commercial vehicles were in use in the European Union (EU) [2]. 54 % petrol, 42 % diesel, 0.5 % hybrid, and 0.2 % electric vehicles were reported [2] regarding the fuel type of the passenger cars in the EU, emphasizing the dominant market share of ICEs. 70.5 million passenger cars and 24.4 million commercial vehicles were produced worldwide in 2018 alone [3]. Hence, the entry of numerous ICEs in service is expected in the next years. Simultaneously, customer and legislative demand regarding improving performance, fuel economy as well as stricter regulations regarding emissions and more realistic emission tests are expected [4]. Accordingly, continuous development of ICEs is an absolute necessity [5], which means increasing requirements on the performance of all machine components, including engine oils. To ensure reliable, environmentally friendly and economic deployment of modern and future ICEs in both private and industrial use, components working at peak performance are required. For example, the average expected lifespan of a passenger car is around 8-10 years correlating with 150.000-200.000 miles [6]. Therefore, it is expected that engine components, but especially hard-to-replace engine parts, such as piston rings, (camshaft) bearings, and valves perform comparably regarding lifetime in service. A premature failure of an ICE has to be prevented to avoid considerable financial consequences in private use and the industry, which harm customer trust and might pose a potential hazard.

Tribometers are modelling friction and wear behaviour [7] in a well-defined, controlled manner, and therefore are an essential tool to characterize materials, lubricants and engine components. Bench tests performed with single engine components as well as testing via engine test rigs are commonly used by development engineers to assess for the present and future requirements. However, the simulation of the entire lifecycle of engine components and complete ICEs remains challenging in the development phase due to long test periods and high expenses. Engine test rigs and test benches usually require substantial amounts of lubricants, usually in the range of 10-60 liters [8]. For this reason, such experiments are usually performed with fresh oils [9], since used lubricants are not available in a defined and reproducible condition as well as required quantity. The disregard of oil damage, i.e. oil degradation or oil aging in use bears the danger to false conclusions regarding lifetime and performance of engine components and ICEs. This is enhanced by the fact that fresh oils are practically not available in service, since oils are usually contaminated with remains of used oil aliquots in the engine after oil changes [10], which can account for up to 20 % directly after an oil change [11].

Moreover, oil degradation occurs in the course of the utilization of the engine oil, resulting in:

- additive depletion, especially antioxidants and anti-wear additives [12],
- formation of degradation products, especially oxidation products [13] and
- accumulation of contaminants, e.g., wear, dust, water or soot [14].

Thus, lubricating properties of the oil are depending on the oil condition and deteriorate with the utilization of the lubricants [15]. In addition, significant differences regarding the oil condition can be found when comparing diesel and gasoline fuelled passenger cars [16].

Accordingly, tribometrical experiments and bench tests applying lubricants with defined degree of degradation, e.g., in critical condition, can significantly contribute to validated knowledge on the overall performance of lubricated systems. Generally, defined oil condition can be achieved by various artificial alteration methods, i.e., simulation of oil degradation in laboratory-based devices, combined with adequate oil condition monitoring. Several standardized artificial oil alteration methods are available to determine the conformity of oils with respective specifications. A typical approach is a thermo-oxidative alteration at ambient pressure, e.g., CEC L-48-A00 [17]. Most standardized artificial alterations enable a rapid execution in the course of a few days to weeks, but the obtained oil quantity is strongly limited, reaching a maximum of 1 liter, which makes such approaches unsuitable for the application of defined used oils as test oils in bench tests or engine test rigs [17].

Besser et al. proposed an artificial alteration method in a large-scale device for the time- and cost-efficient production of test oils with defined and reproducible degree of degradation [18]. This study focuses on the validation of the large-scale device, comparing it to the comprehensive physical-chemical data regarding engine oil degradation derived from a field test as reported by *Dörr et al.* [11]. In this publication, friction and wear behaviour of field samples and artificially altered oils are compared with the fresh engine oil as reference. Tribometrical properties are further correlated with the physical-chemical characteristics of the investigated engines oils, thus allow the correlation between tribological performance and oil degradation.

2. Materials and methods

The authors previously discussed engine oil degradation during a field test and by artificial alteration in a large-scale device, see [18] and [11]. Since this work is intended as a continuation of the two publication, only an overview of the most important methodological aspects are presented.

2.1. Engine oil

The study was performed utilizing a commercially available SAE 5W-30 engine oil suitable for both diesel and gasoline engines approved for various specifications. The approved specifications and the key properties of the selected engine oil are listed in table 1.

Table 1. Approved specifications and key physical-chemical properties of the engine oil

Engine oil specifications	
Engine oil type	SAE 5W-30
Approvals	ACEA C3 API SN BMW longlife-04 MB 229.51 / 229.52 VW 502.00 / 505.00 / 505.01 GM dexos2™
Acidification, base reserve	
Total base number (mg KOH/g)	6.5
Neutralization number (mg KOH/g)	1.6
Elemental composition	
Ca content (mg/kg)	1910
P content (mg/kg)	720
S content (mg/kg)	2350
Zn content (mg/kg)	880

This SAE 5W-30 engine oil displays typical properties amongst currently available engine oils. Calcium, phosphorus, sulfur and zinc were identified as additive elements, where the additive package contains calcium carbonate suspended by detergents as base reserve and zinc dialkyl dithiophosphate (ZDDP) for wear protection. The state-of-the-art approvals ensure that the obtained results are valid for and transferable to modern internal combustion engines.

2.2. Field test

The evaluated field test experiment is identical to the one presented in [11]. The selected engine oil was applied in a conventional passenger vehicle equipped with a modern internal combustion engine for the length of a typical oil change interval. The main technical parameters of the vehicle and the typical mode of operation during the field test are presented in table 2.

Table 2. Key technical parameters of the applied passenger vehicle

Engine parameters	
Engine alignment	Inline, 4 cylinder
Charging	Turbocharged
Displacement volume (L)	1.4
Power at 4200 min^{-1} (kW)	88
Compression ratio (-)	9.5:1
Fuel type	Gasoline RON 95 (see EN 228 [19])
Fuel system	Sequential multi-port fuel injectors
Vehicle	
Year of manufacture	2013
Engine mileage at start of test (km)	63,391
Deadweight (kg)	1396
Operating conditions	
Typical daily operating distance (km)	~ 110
Freeway (%)	~ 70
City (%)	~ 20
Thoroughfare (%)	~ 10

Before the field test, an oil change was performed twice to reduce residual used oil amounts in the engine, which can account for up to 20 % right after an oil change [11]. After the first oil change a short driving period for approx. 10 km was performed to ensure that the removal of the used engine oil is almost complete, then this “rinse-oil” was drained and a further oil change was performed including the replacement of the oil filter.

The field test was conducted under real conditions, which was primarily commuting for approx. 110 km per day. As presented in table 2, the typical daily driving cycle consisted of approx. 70 % freeway, 20 % city and 10 % thoroughfare, respectively. This resulted in a total of 19,800 km in 8 months.

The engine oil was sampled in regular intervals, where, approx. 10 mL of engine oil were removed in 1,000 km intervals during the first 10,000 km of utilization. This sampling interval was later extended to 3,000 km. Before every sampling, the engine was running in idle mode for a few minutes to ensure the homogeneity of the engine oil. The engine oil was not refilled during the field test; hence the field test was terminated once the oil level reached the OEM-specified minimum.

2.3. Artificial alteration in large-scale device

Artificial alteration of the engine oil was performed in a large-scale device as described in [18]. 100 L of engine oil were placed in a 250 L stainless-steel jacketed reactor, tempered and subjected to an air flow via a stainless-steel air-inlet pipe under continuous stirring. The engine oil sample was kept at a constant regulated temperature throughout the whole alteration process under constant stirring. Compressed air was used for oil oxidation at a constant air flow throughout the whole alteration process. The exhaust gases were cooled by a primary condenser and a secondary condenser. In this two-step system, the primary condenser operated under reflux condition, while the secondary condenser acted as an oil trap. The engine oil was sampled in intervals of 24 h.

There are significant differences in the timescale and the volume between a field test and the large-scale alteration. Performing oil degradation in a vehicle is a very resource-intensive process, which requires more than 8 months for a typical oil change interval to produce some liters of used engine oil. An engine test rig can provide such an oil in a week, however, the achievable quantity remains low and the efforts needed are tremendous. Artificial alteration in a large-scale device as described in this publication is capable to provide up to 200 L of defined “used” oil in one batch within only a few days, which presents a great cost-reduction potential. Figure 1 summarizes the time requirements and lubricant quantities for the discussed possibilities to produce used engine oils.

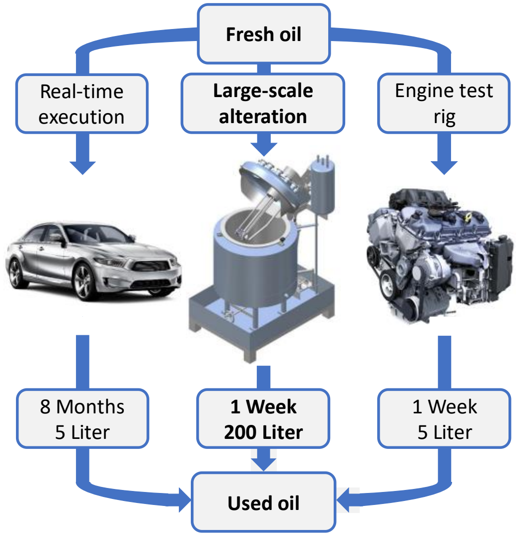

Figure 1. Summary of the discussed oil alteration methods regarding time requirement and lubricant amount

2.4. Conventional oil analysis

The oil samples from the field test and by artificial alteration were analysed by conventional analytical techniques to describe the oil condition over time. Of particular interest were the changes in the chemical composition of the engine oils, i.e., oil additive depletion and the formation of degradation products. Conventional oil parameters were determined similarly to the works presented in [18] and [11], where the detailed description of the applied in-house methods are available. The performed analyses of all oil samples consisted of:

- oxidation via Fourier transform infrared spectrometry (FTIR) utilizing an in-house method based on the determination of the absorption peak heights at 1720 cm^{-1},
- residual phenolic and aminic antioxidant content (AO) as well as ZDDP, based on the initial amounts in the fresh oil (initial absorption peak height equal to 100 %) also monitored by FTIR according to in-house methods, absorption peak heights were determined at 3650 cm^{-1} for phenolic AO, at 1515 cm^{-1} for aminic AO, and the highest intensity in the range from 1020 to 920 cm^{-1} for ZDDP, see [11],
- neutralization number (NN) according to DIN 51558 [21] by color-indicator titration,

- total base number (TBN) according to DIN ISO 3771 [22] by potentiometric titration with perchloric acid and
- elemental composition via inductively coupled plasma optical emission spectrometry (ICP-OES) after microwave-assisted digestion with nitric acid, according to an in-house method [11].

In addition, the gravimetrical quantification of the *n*-heptane insoluble fraction of the oil samples that derived from the field test was also performed to account for soot as main contributor to *n*-heptane insolubles. Focus was laid on the influence on the tribological properties caused by soot in engine oil, since the presence of soot might have an impact on the tribological performance [23]. Soot was gained via centrifugation and microfiltration with three independent aliquots as described in [11]. In detail, the final used engine oil was mixed with *n*-heptane, then centrifuged at 12,000 rpm and 40°C for 60 minutes. The supernatant solution was carefully removed after centrifugation. This process was repeated with fresh solvent four times in total. To gain soot not removed by centrifugation, the collected supernatant *n*-heptane phases were filtered through a cellulose nitrate membrane filter with a pore size of 0.1 µm. The reported total soot content refers to the sum of sediment after centrifugation and residues after microfiltration.

2.5. Tribometrical characterization

Selected used and artificially altered oil samples were subjected to an SRV® 5 model tribometer (Optimol Instruments Prüftechnik, Munich, Germany) for the determination of friction and wear properties. Table 3 shows an overview of the contact configuration and the experimental parameters.

The average surface roughness of the test specimens was adjusted to approx. 0.1 µm via polishing, to ensure that the surfaces involved are uniform and defined, and to reduce related interferences in the friction and wear measurements.

The coefficient of friction (CoF) was monitored continuously during the duration of the SRV® tests. Afterwards, the balls were cleaned, amongst others, in a 0.05 M disodium ethylenediaminetetraacetate (EDTA) solution at room temperature for 2 minutes to remove chemical species, i.e., the tribofilm, from the ball surface. Wear on the utilized balls was then determined by means of wear scar diameter and wear volume by confocal microscopy, see [24] and [25]. For this purpose, 3D topography data obtained from the utilized balls with an approx. 150 nm lateral and 2 nm vertical resolution was compared to the reference surface (sphere) before the SRV® experiment. The reported values of CoF, wear scar diameter and wear volume calculations are based on one experiment per oil.

Table 3. Configuration and operating conditions of the tribometrical experiments

Tribometer configuration	
Contact	Ball on disc
Mode	Oscillation
Ball diameter (mm)	10
Disc dimensions (mm)	10 x 7.9
Material (ball and disc)	100Cr6
Hardness (ball and disc)	HRc 62
Average roughness (µm)	0.01
Operating conditions	
Oil quantity (mL)	~ 0.1
Temperature (°C)	100
Load (N)	50
Stroke (mm)	1
Frequency (Hz)	30
Mean contact pressure (GPa)	1.2
Maximal contact pressure (GPa)	1.7

3. Results and discussion

3.1. Conventional oil analysis

Figure 2 shows the progress of oxidation and the depletion of the antioxidant additives as determined by FTIR. This specific engine oil formulation contains alkylated diphenyl amines (aminic AO) and sterically hindered phenols (phenolic AO). The synergic effect between those two compounds is well known and described in [26], [27] and [28]. Figure 2 b) displays the average of the two compounds as "total antioxidant content". It is noted that "utilization time" is different in the two methods, being kilometers (km) for the field test and alteration time (h) for the artificial alteration. As the final oxidation and final AO content are comparable, a direct comparison is somewhat possible.

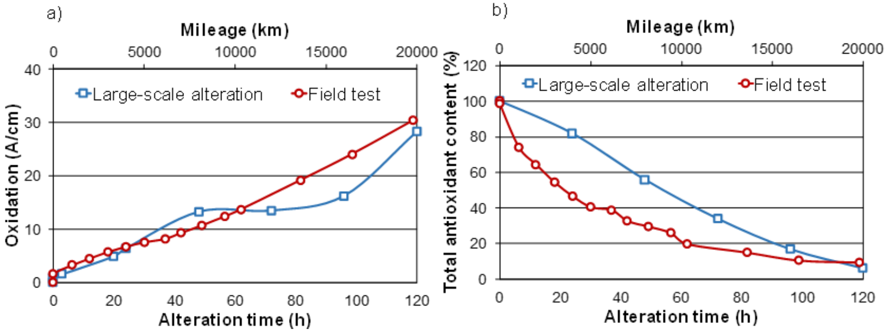

Figure 2. Progress of oxidation and depletion of the antioxidants during large-scale alteration and field test

As illustrated in Figure 2 a), oxidation increases over time both in the passenger car and in the large-scale device. Generally, oxidation in case of the artificial alteration follows the trends postulated by Besser et al. in [18], where an initial increase in the oxidation is observable (induction phase), which is followed by a close-to constant phase, the AO consumption phase. After the AO consumption phase is completed and the residual AO content is negligible, the oxidation phase takes place (after approx. 72 h). In case of the field test, the three phases of the oxidation are not as pronounced as in case of the large-scale alteration. Regardless, a significant increase in the rate of the oxidation is observable around 6,000 km, where the total AO content reaches about 40 %. The trend of AO depletion seems to be faster in case of the field test. Nevertheless, the final oxidation and final total AO contents for both large-scale alteration and field test are very well comparable to each other, being about 10 % of total AO content at an oxidation of 28.1 A/cm for the large-scale alteration and 30.2 A/cm for the field test. This suggests good overall comparability between the final oil condition achieved in the large-scale device and that obtained in the field test.

The oxidation of the engine oil results in the formation of acids, which is characterized by NN increase. Furthermore, increased NN causes the consumption of the base reserve, which results in TBN decrease. Figure 3 displays the progress of NN and TBN.

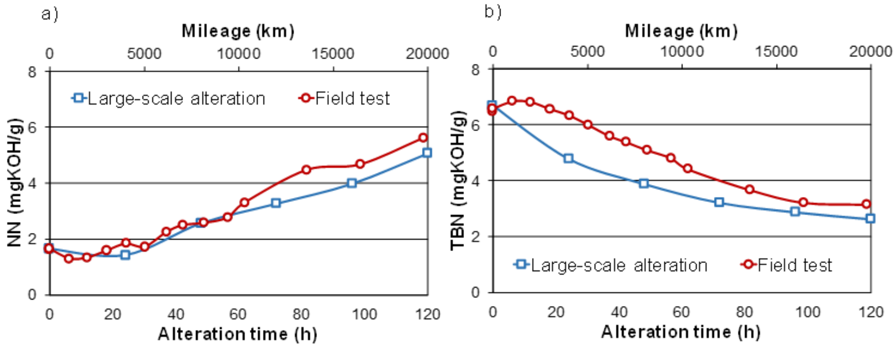

Figure 3. Progress of NN and TBN during large-scale alteration and field test

As shown in Figure 3 a), the initial NN of the fresh engine oil amounts to 1.6 mg KOH/g and is attributed to additives with acidic characteristics, e.g., ZDDP as antiwear additive. At low initial oxidation levels, the NN shows some minor decrease, possibly linked to the degradation of the mentioned engine oil additives. Afterwards, a dynamic increase in NN takes place. This is attributed to the already mentioned formation of acids through oxidation. The NN trends in the large-scale alteration and the field test are almost identical, which suggest a good comparability of both degradation processes. The final oil conditions are also very similar regarding NN, being 5.1 mg KOH/g for the large-scale alteration and 5.6 mg KOH/g for the field test.

Figure 3 b) reveals a dynamic trend for the TBN decrease. This highlights the function of the base reserve to counteract the acidification of the engine oil by neutralizing the formed acids, hence, depletes during utilization. The TBN seems to level out at a residual value of approx. 3 mg KOH/g, which can be caused by the limitations of the titration method with strong perchloric acid as described by DIN ISO 3771 [22]. This is attributed to the very strong acidic nature of perchloric acid, which protonates and hence detects not only the base reserve, but other lightly basic engine oil additives [11], such as dispersant bases and even diphenylamine antioxidants [29]. Consequently, it is assumed that the base reserve completely depletes after approx. 16,500 km in the field test and after 72 h in the large-scale alteration. The final TBN is 2.6 mg KOH/g for the large-scale alteration and 3.1 mg KOH/g for the field test, which are very well comparable values. These results further point to a very good correlation between both final oils and the progress of the acid-base reactions in both systems.

Table 4 provides a comparison of the measured conventional oil parameters for the fresh engine oil and the respective final oil condition obtained by the large-scale alteration and the field test.

Table 4. Comparison of the fresh oil and the final oils of the large-scale alteration and the field test

Oil property	Fresh oil	Final condition large-scale alteration	Final condition field-testing
Oxidation (A/cm)	0	28	30
Phenolic AO (%)	100	2	11
Aminic AO (%)	100	10	7
Total AO (%)	100	6	9
ZDDP (%)	100	0	0
TBN (mg KOH/g)	6.5	2.6	3.1
NN (mg KOH/g)	1.6	5.1	5.6

The determination of the gravimetric soot content was performed as presented in [11]. The analysis of the final used oil derived from the field test resulted in a total soot content of 1.5 ± 0.1 wt%. This result cannot be directly compared to reference values, since although publications are available on gasoline soot, e.g., [30] and [31], the studies primarily focus on the morphology of the soot particles and absolute quantities are not available. Up to 10 wt% of soot are reported for diesel engine oils [32], which supports the measured 1.5 wt% since gasoline engines are less prone to soot formation compared with the diesel counterparts.

3.2. Tribometrical characterization

Tribometrical experiments were applied to characterize the changes in friction and wear properties with propagating oil degradation. Figure 4 a) shows the CoF of the fresh engine oil, the final oil and the samples obtained from the field test and the large-scale alteration. Furthermore, the final oil sample from the large-scale alteration with 1.5 wt% added soot is also displayed. As shown, the CoF stays close to constant during all SRV® experiments and shows only a minor, but similar increase in the used or altered samples. This most probably due to the degradation of friction modifier additives but the exact mechanism has yet to be confirmed. Furthermore, the results show that friction measurements alone are not sufficient to determine oil condition, since as discussed in 3.1, the engine oil deteriorated to a great extent during both the large-scale alteration and the utilization in the field. Figure 4 b) displays the wear volume measured on the ball after the SRV® tests for the field samples (disc wear is minor compared to ball wear, hence was not further considered). As clearly illustrated, the wear properties of the oil are deteriorating with utilization in an ICE, as the wear volume of the final oil sample is over a magnitude of power higher compared to the fresh oil. Some decrease of the wear volume is experienced when comparing the used oil samples after 13.500 and 20.000 km. The exact reason for this observation is not yet known, but it is assumed that it is connected with the composition of the tribofilm.

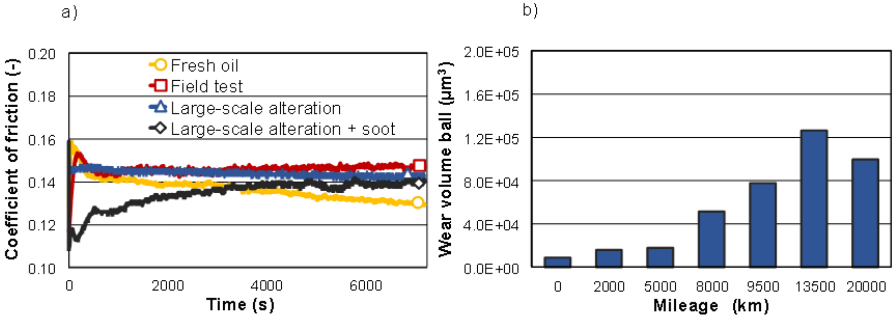

Figure 4. CoF of selected engine oil samples and wear volume of selected field samples

Figure 5 shows the iron and the ZDDP content of the field samples compared to the cumulative wear volume measured in the SRV® experiments. Dörr et al. attributed the increase of the iron content in the engine oil to wear and corrosion [11]. Since the wear particles are accumulating in the engine oil during its lifetime, the SRV® wear results were also summed up to enable a better comparison, hence cumulative wear is displayed. The good correlation between iron content and cumulative wear volume confirms the usability of tribometrical testing via an SRV® tribometer. Although direct monitoring of engine wear in the field, e.g., under real driving conditions is technically limited, indirect methods such as tribometrical testing and iron content analysis deliver meaningful information regarding the in-service tribological performance of lubricants relative to the fresh engine oil.

Figure 5 also shows the depletion of the ZDDP antiwear additive during the field test. Since ZDDP is one of the most extensively used antiwear additives, see [33], [34], and [35], the knowledge about its degradation processes is of great importance, primarily since the tribological properties of lubricants are substantially influenced by the amount of antiwear additives and the presence of its degradation products [36]. ZDDP depletes rapidly during the utilization of the engine oil, depleting completely between 8,000 and 9,500 km. As described in detail in [11], the residual ZDDP content is directly linked to the rate of iron accumulation in the engine oil during its life-cycle in a passenger car. The rate of iron accumulation increases by a factor of 4, once the ZDDP antiwear additive is close to its depletion. Comparatively, a significant increase in the wear volume by SRV® testing also correlates with the ZDDP depletion.

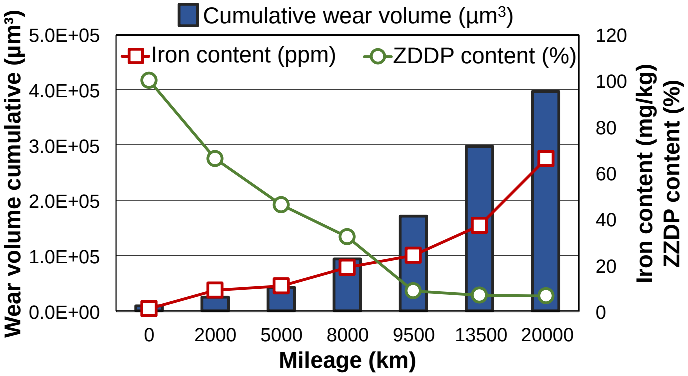

Figure 5. Iron and residual ZDDP content of the engine oils obtained from the field test compared to the cumulative wear volume during the SRV® experiments

Next, wear properties of the fresh engine oil and the final oils obtained from the field test and the large-scale alteration were compared, to determine whether the tribological properties are similar. Therefore, the soot sample isolated from the final oil from the field test was added to the final oil derived from the large-scale alteration in concentration of 1.5 wt% by ultrasonic dispersion to investigate the impact of soot on the wear properties.

Figure 6 shows the wear scar area and wear volume of the SRV® balls of the four oils described above. As expected, the fresh oil performs well regarding both wear scar area and wear volume. The final field sample shows significant deterioration regarding the wear properties, where the wear scar increases with approx. 70% in area and the wear volume with more than a magnitude compared to the fresh oil. The final oil derived from the large-scale alteration shows a pronounced increase in the wear scar area and wear volume, approx. 5 % and 80 % compared to the fresh oil, respectively. Nevertheless, the tribological properties are not comparable to the final field sample. However, the addition of soot results in a significant further increase in both wear scar area and wear volume. Furthermore, the final oil from the large-scale alteration with added soot shows good comparability with the wear properties of the final field sample. This highlights that wear is a system characteristic and a variety of physical-chemical oil properties are contributing to tribological behaviour.

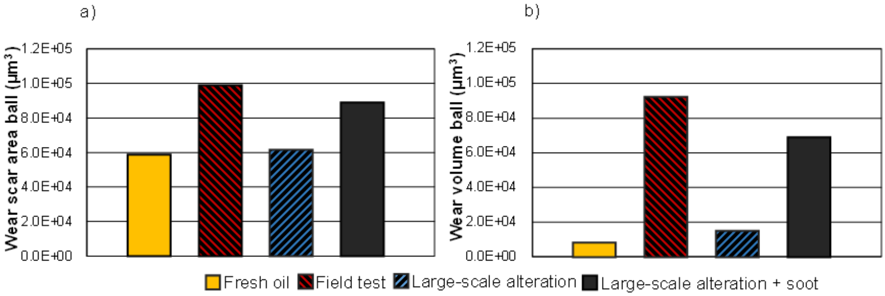

Figure 6. Wear scar area and wear volume of the fresh engine oil, the final oil obtained from the field test and the large-scale alteration and the final oil from the large-scale alteration with added soot.

To summarize, the wear results of the SRV® tests are showing a good correlation with the increase in iron content (engine wear) during the field test, which confirms that this tribometrical approach is suitable. Both wear scar area and wear volume increase significantly during the field test which highlights the tribological effects of oil degradation. There is also some increase in case of the final oil derived from the large-scale alteration, but comparable wear properties were reached when soot was added. Differences were also found in the friction properties (CoF), the findings suggest that higher friction levels of the degraded engine oils are due to friction modifier depletion.

4. Conclusions

The aim of the study was to reproduce a given used engine oil derived from a passenger car after 19,800 km in sufficient quantities for the execution of bench tests. Therefore, a field test and a large-scale alteration were performed and compared via conventional oil analytical methods and tribometrical testing using an SRV® tribometer. Conventional oil analyses showed very good comparability of the used and artificially altered oil samples regarding oxidation and NN as well as AO, ZDDP and TBN depletion.

Wear behavior in the SRV® tribometer correlated well with the engine wear in the field test expressed as iron content, which confirmed the applicability of the SRV® test method. Very similar friction and wear properties were found regarding CoF, wear scar area and wear volume, when comparing the final field sample with the final oil from large-scale alteration, where the latter contained similar amounts of soot as the final field sample. This also highlights the importance of soot regarding tribometrical properties and the necessity of further research regarding soot characterization. Further analysis of the surfaces is also needed, e.g. the utilization of X-Ray photoelectron spectroscopy (XPS), especially in regard of the tribofilm composition to uncover differences depending on the used oil condition.

Overall, very similar final oil conditions were achieved both regarding physical-chemical and tribological properties. The results also show that upon simulating oil degradation in the laboratory, utmost carefulness is to be taken, since degraded engine oils are very complex systems with various crucial properties, which all greatly impact lubrication performance in an ICE. Nevertheless, the artificial reproduction of used engine oils and reaching pre-defined, close-to-reality oil conditions regarding physical-chemical properties, friction and wear behaviour is possible, even in large scales.

In this regard, artificial alteration in the large-scale device can substantially contribute to component or system level testing of engine components by the provision of test oils with a defined degree of degradation via a close-to-reality, reproducible, rapid and cost-effective procedure. Bench and engine tests with such "used" engine oils provide substantial knowledge regarding component or engine performance over lifetime, which would remain hidden if fresh oils were used as the only test oils.

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