

FUEL LUBRICITY AND ITS LABORATORY EVALUATION

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Abstract – This literature review paper discusses the subject of lubricating properties of liquid hydrocarbon-based fuels and laboratory bench tests applied in lubricity evaluation. The analysis was made in order to highlight the importance of fuel lubricity evaluation, especially application of relatively rapid laboratory tests. Inadequate lubricity may lead to an excessive wear of fuel injection system components and in some cases – even to catastrophic failure what, in turn, manifests itself in higher replacement costs, shortened service life, inefficient engine performance and increased tailpipe emissions. Nowadays, when more and more rigorous emissions standards for transportation fuels are continuously established, the satisfactory fuel lubricity is of great importance. Lubricity determines the antiwear behaviour of the lubricant over the regime of boundary lubrication when the moving surfaces are separated only by a very thin fluid film adhering to them. The most important role in forming such films is played by polar compounds and aromatic hydrocarbons that are naturally present in crude oil derived fuels. However, the refinery processes applied in fuel production remove them, thus reducing the lubricity. Fuel lubricity problems were first defined in the mid-1960s and resulted from more severe refining and treatment processes applied in the production of aviation kerosene. In those days, injection equipment failures in aircraft turbine engines were reported. Then, in the late 1980s, similar problems were revealed after the implementation by US and NATO forces of “The Single Fuel Forward” policy which mandated that all military vehicles must be operable with kerosene-based fuel. Lubricity problems regarding diesel fuel emerged in the late 1990s when some countries set limits on the sulphur and aromatic hydrocarbon content in this fuel. The paraffinic diesel fuel produced by the Fischer-Tropsch synthesis or hydrotreatment process that is more commonly applied nowadays also possesses very low lubricating properties. Generally, to provide good fuel lubricity, various additives are applied and bench tests are mostly employed to estimate their effectiveness. Since 1960 many test rigs have been developed. Several inter-laboratory test programs were carried out to select the best bench tests that would show good correlation with field experience. Among them, only BOCLE, HFFR, and SLBOCLE test methods become industry standards.

Key words – boundary lubrication, fuel lubricity, BOCLE, HFFR, SLBOCLE

JEL Classification – L62

INTRODUCTION

After the Second World War, economic growth led to rapid development in the automotive industry and thus to the widespread use of motor vehicles. However, for many years, environmental problems associated with motor vehicles were ignored. Only since the end of the 1970s has there been observed increased concern about the natural environment and also in this period, there have appeared laws and regulations concerning environmental issues. Some of these forced the reduction of pollution of automotive origin because

motor vehicles were identified as one of the primary sources of air pollutants. The majority of legislative initiatives concerning exhaust emission were introduced in the 1980s. Among others, they concerned the reduction of lead content in gasoline as well as particulates, sulphur, and aromatics in diesel fuel. At that time such terms as environmentally-friendly fuels, clean burning fuels, and reformulated fuels also appeared. These terms emerged, among others, from such large-scale research programmes as The European Programme on Emissions, Fuels and Engine Technologies (EPEFE) and The US and European Auto-Oil Programmes realized to investigate the

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impact of fuel reformulation on exhaust emissions – to help improve air quality. The projects provided the background for environmental regulations.

Automotive fuels are still mostly fossil derived and are complex mixtures containing hundreds of chemical components, mainly hydrocarbons (constituting the bulk of the fuel). They have to satisfy various engine types, different operating conditions, and fuel system technologies and meet efficiency and environmental requirements. Sulphur compounds are inherent constituents of crude oil. They are removed in refinery upgrading processes for their odour, acidic nature, and the fact that they are known to be strong catalytic poisons. A reduction in sulphur content provides benefits in terms of health and the environment. However, the severe hydroprocessing necessary to remove sulphur produces fuels that have a poor lubricating ability which may, in turn, lead to the failure of the engine injection equipment. Additionally, deterioration of lubricating ability results also from increased injection pressures (ca. 200-250 MPa – to reduce the size of fuel droplets) applied in modern engines. An additional factor that negatively influenced diesel fuel lubricating properties was viscosity decrease resulting from a limitation in the final boiling point.

To restore fuel lubricity, the refinery industry uses various lubricity enhancing additives. The best way to evaluate their effectiveness is to perform vehicle tests or full-scale injection pump tests. However, such tests are expensive, require a lot of fuel, and are time-consuming. Thus, the most popular are bench tests that are cost effective, quick, and usually require a small amount of fuel.

1. LUBRICITY

The history of lubrication is almost as old as the history of mankind. From ancient days, from the time when people started using tools, various natural substances were applied to prevent friction and wear on the sliding surfaces. For thousands of years, the most used lubricants were bitumen, tar, and oils from vegetable and animal sources (e.g. olive oil, tallow, castor oil, whale and sperm oils) [1]. With the Industrial Revolution, new manufacturing processes and machine tools brought about greater demand for lubricants. Therefore, the lubricant industry expanded. For instance, during the 1800s the whaling industry (sperm oil delivery) was the fifth largest industry in America [2]. Much faster development in the lubricant industry occurred with the Second Industrial Revolution. Rapid industrialization, mechanization, and automation of manufacturing processes and the development

of the automotive industry also led to much higher requirements for the quality of lubricating substances. Fortunately, in those days the age of crude oil began and the lubrication market started to offer products of the petroleum industry to ensure the smooth functioning of machines and vehicles. However, the first applications of mineral lubricants had already revealed that the majority of them are not as effective in reducing friction as animal oils and those of vegetable origin [3]. Under practical operating conditions, lubrication problems were observed over the regime of boundary lubrication. Boundary lubrication conditions usually occur at low speeds and high loads when the moving surfaces are separated from each other by a very thin fluid film adhering to them. Under such conditions, the bulk properties of the lubricant are insignificant and the viscosity of the lubricant is not a friction controlling parameter. The most important parameter is the physical and chemical interaction of the lubricant with the solid surface [4-5]. Boundary films are formed by physical bonding (van der Waals forces), chemisorption, and chemical reactions. In such conditions, the antiwear behaviour of the lubricant is determined by “lubricity”. This term appeared in the 20th century. Earlier, at the end of the 19th century, such phenomenon was described as “oiliness” (less often “body”, “greasiness” or “unctuosity”) a term introduced by the great American inventor Albert Kingsbury and related to differences in the lubrication behaviours of fluids with the same viscosity [6]. According to the definition adopted those days by the Society of Automotive Engineers: “Oiliness is a term signifying differences in friction greater than can be accounted for on the basis of viscosity when comparing different lubricants under identical test conditions.” [7] With the development of science and technology, the term “lubricity” has been modified but there is not established an unequivocal definition of lubricity and its quantitative measure, although many attempts have been made in this field, e.g. [8]. Lubricity consists of phenomena and processes occurring in the friction zone which depend on the kind of lubricant, solids, and atmosphere. As opposed to viscosity, which is an individual property, lubricity comprises all the lubrication phenomena which do not enter into the hydrodynamic theory and are not secondary mechanical effects [9-10]. It is a conventional concept indicating the ability to generate boundary layers that protect against excessive wear. Lubrication is insufficient when there is a lack of a resistant enough boundary layer able to completely separate surfaces moving against each other.

2. FUEL LUBRICITY EVALUATION

Demand for diesel and distillate fuels is growing worldwide. In the European Union, road transport plays a significant role in freight transportation, and the EU fuel market is still dominated by diesel. A similar trend is also observed in the US—refiners are shifting production from gasoline to diesel. Nowadays, fuel quality is more important than ever. Sophisticated constructions and rigorous environmental regulations require fuels of the highest quality to guarantee the proper engine and vehicle operation. Components of the fuel-injection system are built to very strict tolerances. In this connection, lubricity is a very important parameter of motor fuels, especially diesel and jet fuel. Thus, inadequate lubricity may shorten the service life of fuel injectors and high-pressure pumps leading to their failures.

To recommend guidelines regarding requirements for fuel quality, automobile and engine manufacturers from around the world published the Worldwide Fuel Charter (WWFC) in 1998. This document was addressed to governments and the refining industry with the objective of matching fuel quality with vehicle needs and emission standards and is regularly updated. The sixth edition of the WWFC was released in 2019. Recommendations presented in the WWFC also concern lubricity requirements [11].

Problems associated with inadequate fuel lubricity (sometimes called “slipperiness”) were observed for the first time in the mid-1960s in the US and Europe and were related to injection equipment failures in commercial and military aircraft turbine engines, particularly to wear and seizure of high-pressure piston-type fuel pumps [12]. In the beginning, these problems were called “stiction” [13]. They resulted from refinery processes applied by the petroleum industry in the production of aviation kerosene in order to upgrade the thermal stability required for the new generation of jet engines. The applied refining and treatment processes (hydrotreating and clay treatment) caused the removal from the fuel of many constituents responsible for effective lubrication. Earlier, to secure a satisfactory life of high-pressure fuel pumps, an addition of about 1% of lubricating oil to the fuel was practiced in aviation [14]. However, this practice was forbidden because of its negative effect on thermal stability. To help prevent problems caused by the poor lubricity of fuels in the field, it was recommended to blend, whenever possible, hydrotreated fuels with small amounts of nonhydrotreated fuels [15]. The blending

of 10-20 % of a conventionally treated fuel to a hydrotreated one was sufficient to obtain the desirable lubricity level [16]. To overcome lubricity problems the metallurgy of pumps was improved and corrosion inhibitors (Hitec E-515 in most cases) were blended into the fuel [17]. The corrosion inhibitors used prior to this time to combat the excessive corrosion in pipelines and to reduce the carryforward of corrosion products into aircraft fuel systems were found to restore the lubricity of fuels. Therefore their addition started to be obligatory. Since the 1970s, gear-type fuel pumps (less sensitive to lubricity variations) have become increasingly widespread in turbine engines and only isolated incidents related to fuel pump problems have been reported (in the 1980s and 1990s) [18].

From the very beginning in the 1960s started efforts aimed to understand the nature of fuel lubricity and to develop laboratory test methods for lubricity evaluation. Aviation kerosene like other crude oil derived fuels has a complex nature and thus its lubricating ability cannot be predicted from physical and chemical properties and a bench test is needed. An extensive study has been conducted on this subject by Esso in two complementary research projects: under the US Air Force contract [19-24] and one granted by the British government [16]. Also, manufacturers, like Lucas Aerospace, have carried out works to find a way to protect injection equipment from failure caused by insufficient fuel lubricity [14].

Appeldoorn and collaborators were one of the first to study the mechanisms of aviation fuel lubricity. They stated that oxidative corrosion was the primary wear mechanism in aviation equipment and that good fuel lubricity is related to the presence of naturally occurring trace polar compounds containing heteroatoms and heavy aromatics (the highest boiling fractions) rather than bulk fuel properties. They examined the effect on the lubricity of hydrocarbon type, dissolved oxygen, dissolved water, higher temperatures, and metallurgy. The research revealed that among hydrocarbon fuel constituents, heavy aromatics show higher lubricity as compared to paraffins and naphthenes. The authors stated that high molecular weight aromatics are most responsible for good lubricity and their removal in refining processes was the major cause of lubricity problems. Mixtures of heavy aromatics and paraffins behaved better than either component alone and only 2% of heavy aromatic hydrocarbons was sufficient to greatly reduce the wear and friction and increase the load-carrying capacity of

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paraffins. Water and oxygen dissolved in the fuel increased wear and friction through a corrosion process. Additionally, wear and friction were higher at elevated temperature, especially in the air. Furthermore, it was revealed that, although high sulphur fuels show good lubricating properties, sulphur compounds do not affect lubricity. This is not a cause and effect correlation. Only when oxygen and water are absent, are these compounds lubricity agents. In wet air, sulphur compounds (particularly disulfides and alkyl mercaptans) increase wear.

A variety of wear tests designed to evaluate lubricants (e.g. Almen-Wieland test machine, Timken bearing rig, SAE and four-ball extreme pressure testers, Bendix CRC Lubricity Simulator, and Vickers Vane Pump) were employed in the search for a fuel lubricity bench test [25]. However, the obtained results did not provide satisfactory results. Generally, the tests were too severe. This is not surprising, given that the most important property to investigate in the lubricity assessment is the resistance to the breakdown of the boundary layer. Difficulty in the development of fuel lubricity bench tests arises from the fact that fuel injection systems comprise many tribological configurations of contact and various metallurgy. Also, failure modes and mechanisms leading to these failures vary for individual contacts. They depend on load, speed, temperature, humidity, and fuel properties. Additionally, these factors may act independently or synergistically. Thus, the test conditions must be a compromise between many variables. The test should be sensitive to lubricity agents naturally occurring in the fuel as well as to lubricity enhancing additives.

The first test rigs sensitive to the lubricity of aviation kerosene were the Ball-on-Cylinder machine used in the US and, similar in principle, the Pin-on-Disc tester (developed by Esso) used in the UK. Both of these were capable of differentiating between good and poor lubricity fuels and detecting the presence of additives. However, the Ball-on-Cylinder (BOC) machine gained wide acceptance and was most commonly used. This tester was developed in 1965 by Exxon Research and Engineering Company and was a modification of the tester designed by M. Furey [26] in 1961 to investigate lubricants. Several BOC rigs were used by the late 1970s and many laboratory reports were published. However, the test rigs differed in metallurgy and applied procedures (temperature, speed, load, and time of the test) and it was difficult to compare results obtained by individual laboratories. Thus, the Coordinate Research Council Aviation Fuel Lubricity Group established The Ball-on-Cylinder Lubricity Evaluator (BOCLE)

Task Force to prepare an operating procedure and establish its precision [27-28]. Several inter-laboratory test programs (three round robins) were carried out in the UK and US. Earlier, preliminary research programs ruled out other test rigs proposed as lubricity evaluators. An additional motivation to advance the research activity was the fact that the US Congress passed the so-called Energy Security Act in 1980 to reduce dependence on foreign energy resources by producing synthetic fuels. In effect, the US Army Forces decided to introduce the shale derived fuel for jet engines [29]. However, a high degree of hydrotreatment applied in refinery processing of shale oil resulted in the extremely poor lubricating ability of the product and it was necessary to reintroduce lubricity additives in airbases. To assess the additive effectiveness in the shale derived JP-4 turbine fuel a semi-automated version of the Ball-on-Cylinder machine was built and distributed to airbases. In the period 1968-1990, the BOC test was modified several times and in 1990 it was accepted as an ASTM standard for measurement of aviation fuel lubricity as the Ball-on-Cylinder Lubricity Evaluator (BOCLE) [30]. The device consists of a stationary steel ball loaded (9.81 N) against a steel cylinder rotating at a fixed speed. A part of the cylinder (approximately one-third) is immersed in the tested fuel. Fuel lubricity is determined by the mean diameter of the wear scar generated on the ball after a 30 minute test conducted in a controlled atmosphere. The smaller the wear scar diameter (WSD), the better the lubricity. Currently, the specifications for aviation turbine fuel limit the wear scar diameter, measured in the BOCLE test, to 0.85 mm. However, the determination of lubricity is required only for fuels containing more than 95% hydroprocessed material where at least 20% of this is severely hydroprocessed, as well as for all fuels containing synthetic components.

A modified technique based on the BOCLE procedure was proposed by Hadley and Blackhurst [31] in order to determine the scuffing load. The procedure followed the standard method but instead of a steady load, a series of 1 minute runs with incremental load (from 1 to 3.1 kg in 0.1 kg intervals) was applied. A new ball was used for each test. Results of investigation on aviation turbine fuels provided by this procedure revealed good agreement with those provided by the TAFLE test.

The first test originally dedicated to fuel lubricity evaluation was the Lucas Dwell Test developed in 1971 by R.T. Aird and S.L. Forgham [14]. The test rig consisted of a loaded cylindrical aluminium bronze pin sliding on a rotating steel disc covered with the

fuel film. Construction materials applied in this rig represented the metallurgy of the Lucas piston pump, popular in aircraft engines: pump bores and pistons, respectively. Fuel lubricity was measured in the number of revolutions (termed the Dwell number) the disc has made to reach the coefficient of friction equal to 0.4. The higher the Dwell number, the better the lubricating ability of the fuel. In 1969 the British Ministry of Defence decided to coordinate lubricity studies and formed the Fuel Lubricity Panel – a working group to develop a standard method of aviation fuel lubricity evaluation. However, the results showed that Lucas Dwell Test suffered from a lack of repeatability and reproducibility and thus was not suitable for refinery control tests [32]. The test was also evaluated in the US by the Aviation Fuel Lubricity Group (CRC) but the obtained results showed its insensitivity to known lubricity enhancers. This method gained some interest in the 1970s, mainly in Great Britain.

In the year 1985, J.W. Hadley (Shell Research) [33] built the Thornton Aviation Fuel Lubricity Evaluator (TAFLE) on the basis of the Amsler machine. The successive, improved versions of the apparatus were named Mark I, Mark II, Mark III, and Mark IV. The friction pair comprises two steel cylinders mounted in parallel axes, one vertically above the other, creating a line contact. The upper, stationary cylinder was loaded against the rotated lower cylinder. In the final version, the upper cylinder had lower hardness and surface roughness. The procedure, carried out under a controlled environment and temperature, consisted of a sequence of 15 minute tests at incrementally raised loads. The fuel was passed over the specimens under fully flooded conditions. Fuel lubricity was measured as the friction failure load – the load (in kg) at which the maximum coefficient of friction became greater than or equal to 0.4. TAFLE apparatus has not been widely used because of a complex procedure that required an operator with advanced tribological experience and knowledge. However, it was successfully applied in individual cases up to the 1990s to estimate aviation and diesel fuel lubricity properties, e.g. in Sweden [34]. There exists only a prototype of this apparatus.

Implementing the concept of a single fuel on the battlefield for both aircraft and ground vehicles, the US Department of Defence enacted in 1988 "The Single Fuel Forward" policy. The legislation mandated that all military vehicles must be operable with kerosene-based fuel (JP-8/Jet A-1) to enhance operational flexibility. It was decided that

the conversion of diesel fuel to aviation kerosene should be achieved without extensive modifications to existing CI engines. Also, NATO forces operate under a single fuel policy. However, although the logistic benefits of a single fuel on the battlefield are numerous, the substitution of aviation kerosene for diesel fuel revealed a lot of issues to investigate. One of them concerned the compatibility of kerosene with reciprocating piston engine systems. Additionally, during the operations of Desert Shield and Desert Storm in 1990-1991 many injection pump failures were reported, mainly the Stanadyne rotary fuel injection pump, found to be the most sensitive to poor lubricity fuel [35]. Thus, a bench test was needed to evaluate the effect of additives to improve the load-carrying capacity of JP-8 when used in diesel-powered ground equipment. A broad study sponsored by the US Army was conducted on this subject at the Southwest Research Institute. Two bench tests were assessed as promising candidates: the Cameron-Plint High-Frequency Reciprocating machine and the modified BOCLE test [35]. The standard BOCLE procedure did not reflect the lubricating ability of fuels in highly loaded contacts. To compare results, full-scale pump tests were also performed [36-39]. The Cameron-Plint High-Frequency Reciprocating machine [40] was used to simulate the wear mechanisms and metallurgical properties found in the Stanadyne rotary fuel injection pump. The contact configuration applied in this test was ball-on-plate. The test specimens were fully immersed in the fuel. The loaded (15-25 N) ball oscillated against the fixed horizontal plane at frequencies ranging from 5 to 50 Hz. The amplitude of the stroke varied from 2.38 to 15.1 mm. Test duration varied from 1 hour to 10 hours. Fuel lubricity was determined by the scar diameter on the ball. The wear volume was also calculated. It was stated that such a test could be used as a screening tool to find additives for the enhancement of JP-8 lubricity. The second test was a modified BOCLE technique based on that developed by Hadley and Blackhurst and called US Army Scuffing Load Wear Test (US Army SLWT) or the US Army Scuffing BOCLE. It was stated that this procedure has the potential to determine the scuffing load capabilities of fuels and will be developed. An additional advantage of such a solution was that the BOCLE apparatus had widespread availability in the petroleum industry. In a further study [41-42] the BOCLE test procedure was modified in order to reproduce the pre-dominant wear mechanisms (oxidative corrosion and scuffing) that occurred in diesel fuel injection equipment.

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The modifications related to the friction couple, test conditions as well as evaluation criteria. The modified procedure is commonly referred to as SLBOCLE (Scuffing Load BOCLE) and is the most popular in the United States where it was accepted in 1999 as a standard test method for evaluating the lubricity of diesel fuels [43]. The contact configuration applied in this test is a ball-on-rotating cylinder, similar to that used in the BOCLE procedure. A non-rotating steel ball is loaded against a partially immersed polished (instead of the ground one) steel cylinder rotating at 525 rpm for 60 seconds. The load is increased incrementally, starting with 500 g. For each sequential load, the test cylinder is moved at least by 0.75 mm and a new ball is used. The lubricating properties of diesel fuel are determined as the minimum applied load required to produce a friction coefficient greater than 0.175. The test is conducted at a temperature of 25°C and at a relative humidity of 50%.

The fact that the BOCLE apparatus was commercially available induced many investigators to develop its modifications suitable for diesel fuel lubricity evaluation. One such modification was a procedure commonly known as Lubrizol/Hadley Scuffing BOCLE or the Constant Load Scuffing BOCLE [44]. This technique measured the scuffing performance of diesel fuels by applying a constant load that was established at 7 kg. The test duration was 2 minutes and the rotational speed was 300 rpm.

The scuffing performance of diesel fuels was also assessed in the test developed by D. Cooper [45] based on the Cameron-Plint reciprocating tribometer (Plint TE-77). The contact configuration in this rig was roller-on-flat. A loaded roller (75 N) oscillated (1 Hz) mechanically against the fixed plate of lower hardness. The stroke length was 15 mm. The test duration was 1 hour and during this time the temperature raised linearly from ambient to 60 °C. Scuffing severity was assessed by the depth of the scar generated on the plate. Fuels giving the scar depth over 1,5 µm were qualified as possessing insufficient lubricating quality. The obtained results correlated well with those of the US Army SLWT. The test also enabled the assessment of the mild wear performance of fuels by the measurement of the scar width on the roller. However, in this case, the correlation with pump failures was weak.

Failures of injection equipment in CI engines related to the substitution of diesel fuel with kerosene coincided with those related to the application of low sulphur diesel fuel. Until the late 1980s, there were not reported any serious lubricity problems related to diesel fuel. Generally,

petroleum diesel fuel has natural lubricity and this is why, in the past, the less-processed diesel fuels showed good lubricating properties. For a long time, typical sulphur levels in diesel fuel were around 0.2 – 0.5% wt (2000 -5000 ppm). However, concern over the environmental impact of automotive vehicles has led to severe restrictions on sulphur and aromatic content in diesel fuel specifications following the decision to remove lead from gasoline. Limitation of the sulphur content was necessary to enable the introduction of advanced exhaust after-treatment systems. One of the first were regulations adopted in 1988 in California which set limits on sulphur content (500 ppm) and on aromatic hydrocarbon content (10%). Then, between 1991 and 1995, low levels of sulphur and aromatics were specified throughout the Nordic countries (Sweden, Finland, Denmark, and Norway) [34, 46-47]. The amount of sulphur in diesel fuels was reduced from 2000-5000 ppm, even down to 10 ppm (Swedish Class I). Swedish specification also included the total aromatics limit (5% in Swedish Class I). A high reduction level of sulphur compounds and aromatic hydrocarbons in diesel fuels was obtained by applying severe hydroprocessing. First, the feedstock was hydrotreated to remove most sulphur and nitrogen which would poison the catalyst used in the further hydrogenation. Then, hydrogen was added to aromatic and polyaromatic compounds to produce mainly naphthenes. Within a few years the Swedish Class I was the major diesel fuel used in Sweden. Additionally, tax incentives encouraged drivers to use low sulphur diesel. California introduced low-sulphur fuels in 1993. Shortly after the refined fuels entered the market, problems related to proper lubrication of injectors and pumps (in passenger and light duty vehicles) were identified. Using a non-additive-treated Swedish Class I diesel resulted in rotary distribution pump failures at less than 8000 km [48]. Failures of rotary or distributor type pumps were also reported in Canada. They started in the winter of 1989/90 and involved the use of a winter grade diesel fuel [49-51]. To provide good cold flow characteristics, Canadian winter diesel fuel had a lower viscosity and pour point than those supplied in the US or mainland Europe, which resulted in reduced lubricating ability.

Anticipating that the trend of increasingly severe fuel treatment would in effect lead to lubricity problems with automotive fuels, H.A. Spikes and D. Wei started an investigation into the wear-preventing characteristics of diesel fuels in the early 1980s [52]. The aim of their work was to develop

a bench test to differentiate between various diesel fuels, determine their lubricating ability, and estimate the relative effectiveness of natural trace components in contributing to diesel lubricity. To realize this aim, they employed a high frequency reciprocating machine developed in the late 1970s at Imperial College in London [53]. In this device, a loaded (2.2 N) upper steel ball oscillated with a stroke of 0.5 mm against a lower steel plate. Test specimens were manufactured from the same steel but they differed in hardness. A hard ball was sliding over a softer flat. The Vickers hardness of the ball was 845 HV and of the flat – 190 HV. The oscillation frequency was 50Hz. Thus, the velocity was 25 mm/s at midstroke. The contact was fully immersed in fuel and tests were conducted at room temperature. A new ball was used for each test. Fuel lubricity was measured by the wear scar diameter on the ball after the 75 minute test. Results obtained in the investigation revealed that polyaromatics and oxygen-containing polar impurities determine, to a large extent, diesel fuel lubricity and that most sulphur impurities are pro-wear.

The procedure applied by Wei and Spikes, later known as High Frequency Reciprocating Rig (HFRR), was widely practiced after some modifications by C. Bovington et al. [54]. In a search for a satisfactory bench test that would show a good correlation with field experience, they validated various testers. The HFRR technique seemed to be the most promising because it reproduced both wear types (adhesive and fretting) present in the pumps and met the two major requirements established for such a test, i.e. producing low levels of frictional heating in the contact and fully flood the rubbing contact.

The HFRR test consists in high frequency reciprocating motion (stroke length – 1 mm) of a vertically loaded (200 g) steel ball over a static steel plate immersed in the investigated fuel (sample – 2 ml) at the specified test temperature (25 or 60°C) and controlled humidity for 75 minutes. A measure of diesel fuel lubricity is the mean wear scar diameter (measured parallel and perpendicular to the sliding direction) corrected to the standardized water vapour pressure of 1.4 kPa and denoted as WS1.4 (Wear Scar). Generally, the test is performed at 60°C. Lower test result values reflect better lubricity.

Another attempt to monitor diesel fuel lubricity was a modification (by Falex Corporation) of the well-known four-ball test [55]. The modified version was called BOTD (Ball-*nn*-Three-Discs). Initially, it was Ball-on-Three-Seats (BOTS) configuration [56]. Replacement of the three lower balls with the conforming seats created a larger contact area and

was intended to reduce contact stress. However, such geometry requires high precision and it was difficult to manufacture the contact curve with the required dimensional tolerances. Thus, the seats were replaced by discs creating a point contact configuration. The test consisted in a 30 minute run at room temperature. The upper ball rotating at 60 rpm was loaded with 3 kg. The friction pair was immersed in the investigated fuel (40 ml). The average wear scar diameter obtained on the three discs was a measure of fuel lubricity. The carried out investigation demonstrated its sensitivity to lubricity additive evaluation [57].

Lubricity additives have been regularly used by the refinery industry to maintain the good lubricating ability of diesel fuels since the early 1990s. They comprise a range of surface active chemicals, like carboxylic acids and their salts, amides, alcohols, ethers, and esters. Another way to restore diesel fuel lubricity is blending it with high lubricity fuel. The most common practice is blending low sulphur diesel with biodiesel (FAME). Good lubricating properties of biodiesel are provided by oxygen functional groups. Biodiesel is usually blended at a level of 1–2% [58-59].

In 1997, a modified Timken test was developed in the Department of Mechanical Engineering at the University of Saskatchewan in Canada to study the lubricity properties of the biodiesel additized winter fuel. It was called Roller on Cylinder Lubricity Evaluator (ROCLE) [60]. During the test, the wear scar area on the roller and the coefficient of friction were recorded. A dimensionless Lubricity Number (LN) based on wear scar area, applied stress, Hertzian theoretical contact stress, and coefficient of friction was applied as a measure of the fuel lubricating quality. Lubricity Number of 1.0 was established as the pass/fail value for diesel fuel of sufficient lubricity. After some improvements were done by J.W. Munson the test is now known as the Munson Roller on Cylinder Lubricity Evaluator (M-ROCLE) [61]. The carried out investigation demonstrates the high sensitivity of this bench test with respect to additive concentration in diesel fuel. The M-ROCLE procedure was applied to evaluate a number of vegetable-based (soy, flax, sunflower, mustard, rapeseed, and canola) lubricity additives [62]. Canola based additives performed the best in the carried out tests.

Transition to low sulphur diesel generated the necessity to establish a standard test method and a fuel specification. To gather necessary data to select the best laboratory test for diesel fuel lubricity evaluation and to define a minimum acceptable

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lubricity level, an international round robin program (started in 1993) under the auspices of the International Organization of Standardization (ISO/SC7/TC22 Working Group 6) and Coordinating European Council (CEC/PFOOG steering group) was conducted [63]. The program involved original equipment manufacturers, fuel producers, additive suppliers, and independent testing laboratories from Europe and North America. Four bench tests were chosen for evaluation. They were HFRR, US Army Scuffing BOCLE, Lubrizo/Hadley Scuffing BOCLE, and BOTS. To compare the obtained results, together with laboratory investigation, full-scale in-house tests on pumps and injectors were conducted by four manufacturers: Bosch, Lucas, Stanadyne, and Cummins. Taking into consideration the correlation to injection equipment, discrimination power between high and low lubricity fuels, response to additized fuels, repeatability, reproducibility as well as cost and ease of operation, the HFRR technique was selected as the ISO [64] (in 1995) and CEC [65] (in 1996) test method for diesel fuel lubricity. The minimum acceptable mean wear scar diameter was recommended as 450 μm for the test conducted at 60°C and 380 μm for the test conducted at 25°C. For the SLBOCLE - the minimum acceptable load was recommended as 3000 grams and for the BOTD – the minimum acceptable wear scar diameter was recommended as 450 μm [51]. The CEC standard is now obsolete.

In 1999, the HFRR test also became an ASTM standard [66]. The latest version was updated in 2018. A revision of the ASTM standard, in 2011, specified the application of a microscope with a digital camera to capture and record the image of the wear scar and the original method using a microscope for visual observation of the WSD became ASTM D7688 test method [67]. Also, the ISO 12156 standard, after a revision in 2016, defines two methods for the measurement of the wear scar: method "A" – digital camera and method "B" – visual observation. ISO 12156 contains two parts. Part 1 specifies a test method and defines two ways for the wear scar measurement. Part 2 specifies the performance requirement necessary to ensure the reliable operation of diesel fuel injection equipment ($WSD \leq 460 \mu\text{m}$).

To provide good fuel properties, lubricity requirements were introduced into diesel fuel specifications. The maximum HFRR wear scar acceptable by EN 590 is 460 μm and by ASTM D975 – 520 μm , although, the Worldwide Fuel Charter recommends 400 μm for markets with advanced requirements for emission control (categories 4 and

5). The minimum load, measured in the SLBOCLE method, which should provide sufficient diesel fuel lubricity, is 3100 grams.

Apart from the dosing level of the additive, the test results depend on the chemistry of the base fuel. Introduction into the market and broad commercial availability of the paraffinic diesel fuel produced through the Fischer-Tropsch synthesis from natural gas (GTL), biomass (BTL) or coal (CTL), or through hydrotreatment process from vegetable oils (HVO), brought about a concern for its potentially harmful effect on injection equipment due to very low lubricating properties. To prevent possible problems and secure a sufficient lubricity level, suppliers of fuel injection systems postulated applying an extra lubricity evaluation test and incorporating it into the paraffinic diesel fuel specification. They suggested the SLBOCLE test with a minimum limit of 3500 g. However, the carried out studies [68-70] involving the application of both HFRR and SLBOCLE techniques demonstrated that the HFRR test is sufficient to control paraffinic fuels lubricity and it is not necessary to use SLBOCLE as an additional test. The eventual application of the SLBOCLE technique would require a substantial improvement in its repeatability and reproducibility.

Although both HFRR and SLBOCLE methods are approved as standards, the correlation between them is rather poor. The reason may be that the lubricity evaluation is based on different phenomena (wear and scuffing). The HFRR test shows better response to the additive concentration and is the most often applied fuel lubricity test. However, in many cases the HFRR and the injection pump rig test results do not correlate well with each other. For instance, the HFRR test may underestimate the lubricity properties of a fuel [46]. There are also observed some problems in military applications where, according to the "The Single Fuel Forward" policy all compression ignition engines operate with kerosene-based fuel containing synthetic components. In such situation, the HFRR test does not discriminate between neat and additized fuels at approved levels. To ensure adequate lubricity evaluation it was decided to develop a new laboratory test. Currently, the work being performed concerns modification of the HFRR apparatus by changing the test geometry from a point contact (ball on flat) to a line contact (pin on flat) [71]. The modified version is called High Frequency Reciprocating Rig – Line Contact (HFRR-LC). In such alteration, reduction of the Hertzian contact pressure and increasing the active test surface area will probably improve test sensitivity.

For many years, lubricity related problems did not concern gasoline, although reducing the sulphur content in gasoline started in the 1980s. Because gasoline pumps operate at lower pressures than diesel fuel pumps, the lubricity regarding requirements is also not as high as those regarding diesel fuel, and lubricity is not included in gasoline specifications. However, there is concern that the more common application of direct injection (which requires high injection pressures) in spark-ignition engines together with refinery upgrading processes (mainly desulphurization and reduction of olefin and aromatic levels) may lead to premature wear and failure. Gasoline lubricity has been an object of investigation since the late 1990s. However, only few reports are available [72-76] and no dedicated gasoline lubricity test rig is developed. Generally, to study gasoline lubricity, the HFRR test method is applied. However, because of the high gasoline volatility, the conventional test is slightly modified. A larger sample of the fuel is used and the test temperature is 25°C.

CONCLUSIONS

Fuel lubricity is typically evaluated in laboratory bench tests. They are cost effective, quick, and usually require only a small amount of fuel and for several decades they have been applied by the refinery industry. Over a dozen bench-test procedures developed for fuel lubricity assessment were described in detail in the paper. The test rigs are tribotesters with various contact configurations and metallurgy. The procedures define various test conditions and measures of lubricity and have various repeatability and reproducibility. Some of the procedures are nowadays only of historical significance.

Although all procedures are designed to assess the same fuel property, the correlation between them is poor. This is probably because the lubricity evaluation is based on different failure modes and mechanisms leading to these failures and the established test conditions do not comprise a variety of conditions occurring in fuel pumps and injectors.

The most important feature of a bench test is the correlation with injection equipment and sensitivity to lubricity agents naturally occurring in the fuel as well as to lubricity enhancing additives. Also, the cost and ease of operation are important. For this reason, the TAFLE apparatus was not widely used. Its sophisticated procedure requires an operator with advanced tribological experience and knowledge.

As for now, three fuel lubricity tests were accepted

as standards: BOCLE – for aviation turbine fuels and SLBOCLE and HFRR – for diesel fuel. Among these, the HFRR test is the most commonly used. There is currently no standard for gasoline lubricity, although some reports claim that there is a need for such a test.

To provide adequate lubricating abilities of fuels, the lubricity requirements were incorporated into specifications. However, manufacturers of injection equipment recommend further limitations, particularly in the case of diesel fuel produced for markets with advanced requirements for emission control.

In recent years, the importance of fuel lubricity is raising. Modern injection systems of the common rail type operate at tremendous pressures. To allow this, pump and injector components are made to a strict standard. Insufficient lubrication may cause wear of the mating components and in result failures of the injection equipment may occur. Thus, adequate lubricity is the crucial factor for proper and efficient engine operation. In such situation, test methods applied for lubricity measurement should be very sensitive, both to the base fuel and additive chemistries and the additive concentration. Although the HFRR test is the most commonly applied procedure in fuel lubricity evaluation, the results obtained with it not always correlate to high pressure pump wear, for instance – when compression ignition engines operate with kerosene-based fuel containing synthetic components. Thus, to provide more reliable fuel lubricity evaluation some research work may be done in order to modify the existing procedure.

ABBREVIATIONS

BOC	– Ball-on-Cylinder
BOCLE	– Ball-on-Cylinder Lubricity Evaluator
BOTD	– Ball-on-Three Discs
BOTS	– Ball-on-Three Seats
BTL	– Biomass-to-Liquid
CEC	– Coordinating European Council
CTL	– Coal-to-Liquid
GTL	– Gas-to-Liquid
HFRR	– High Frequency Reciprocating Rig
HVO	– Hydrotreated Vegetable Oil
M-ROCLE	– Munson Roller on Cylinder Lubricity Evaluator
ROCLE	– Roller on Cylinder Lubricity Evaluator
SLBOCLE	– Scuffing Load BOCLE
TAFLE	– Thornton Aviation Fuel Lubricity Evaluator
US Army SLWT	– U.S. Army Scuffing Load Wear Test
WSD	– Wear Scar Diameter
WWFC	– Worldwide Fuel Charter

SMARNOŚĆ PALIW I JEJ OCENA LABORATORYJNA

W artykule przedstawiono tematykę właściwości smarych ciekłych paliw węglowodorowych oraz przegląd laboratoryjnych metod oceny smarności. Celem przeprowadzenia niniejszej analizy była chęć podkreślenia ważności problematyki oceny smarności paliw, w szczególności za pomocą relatywnie szybkich metod laboratoryjnych. Niewłaściwa smarność może prowadzić do nadmiernego zużycia elementów układu wtrysku paliwa, a w pewnych przypadkach – nawet do uszkodzenia aparatury wtryskowej, co w konsekwencji powoduje wyższe koszty wymiany, krótszą żywotność, zmniejszone osiągi silnika i wzrost emisji. Obecnie, w sytuacji kiedy normy emisji są coraz bardziej restrykcyjne, smarność jest szczególnie istotnym parametrem określającym jakość paliw. Smarność określa właściwości przeciwozużyciowe w warunkach tarcia granicznego. Tarcie zachodzi wówczas pomiędzy bardzo cienkimi warstwami substancji smarującej zaadsorbowanymi na powierzchniach współpracujących. Najważniejszą rolę w tworzeniu takich warstw odgrywają związki polarne oraz węglowodory aromatyczne występujące w ropie naftowej. Jednakże, w wyniku procesów rafineryjnych stosowanych podczas produkcji paliw, większość związków polarnych jest usuwana, co w efekcie prowadzi do obniżenia smarności. Problem smarności paliw pojawił się po raz pierwszy w latach sześćdziesiątych XX wieku i był spowodowany stosowaniem głębokiego rafinowania oraz procesów uszlachetniania stosowanych w produkcji ropy lotniczej. Konsekwencją stosowania tak wytwarzanego paliwa było wiele przypadków uszkodzeń aparatury wtryskowej turbinowych silników lotniczych. Później, w latach osiemdziesiątych, problemy wynikające z niedostatecznej smarności paliw pojawiły się po wdrożeniu przez Stany Zjednoczone i NATO tzw. koncepcji jednolitego paliwa pola walki (The Single Fuel Forward), która polega na stosowaniu we wszystkich pojazdach wojskowych paliwa przeznaczonego standardowo do silników odrzutowych. Z kolei, problem smarności oleju napędowego pojawił się na początku lat dziewięćdziesiątych, kiedy zaczęto wprowadzać ograniczenia dotyczące zawartości siarki i węglowodorów aromatycznych w tym paliwie. Parafinowy olej napędowy produkowany metodą syntezy Fischera-Tropscha lub w procesach uwodnienia, który jest coraz powszechniej stosowany, również charakteryzuje się niską smarnością. Aby nadać paliwom odpowiednie właściwości smarne stosowane są różnego rodzaju dodatki. Ocenę skuteczności działania tych dodatków prowadzi się najczęściej za pomocą metod laboratoryjnych. Od momentu pojawienia się problemów wynikających z niedostatecznej smarności paliw zostało opracowanych wiele metod laboratoryjnych oceny tego parametru. Przeprowadzono również kilka programów badań międzylaboratoryjnych mających na celu wybranie testów wykazujących największą korelację z badaniami w warunkach rzeczywistych. Spośród ocenianych testów tylko BOCLE, HFFR oraz SLBOCLE uzyskały status metod badań normowych.

Słowa kluczowe: BOCLE, HFFR, SLBOCLE, smarność paliw, smarowanie graniczne

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