



# Article A Methodological Approach to Assessing the Tribological Properties of Lubricants Using a Four-Ball Tribometer

Tareq M. A. Al-Quraan <sup>1,\*</sup>, Fadi Alfaqs <sup>2</sup>, Jamil Haddad <sup>2</sup>, Viktor Vojtov <sup>3</sup>, Anton Voitov <sup>3</sup>, Andrey Kravtsov <sup>3</sup>, Oleksandr Miroshnyk <sup>4,\*</sup>, and Andrii Kondratiev <sup>5</sup>

- <sup>1</sup> Department of Technical Sciences, Ma'an College, Al-Balqa Applied University, Al-Salt 19117, Jordan
- <sup>2</sup> Department Mechanical Engineering, Faculty of Engineering Technology, Al-Balqa Applied University, Amman 11134, Jordan; faalfaqs@bau.edu.jo (F.A.); drjamil@bau.edu.jo (J.H.)
- <sup>3</sup> Department of Transport Technologies and Logistics, State Biotechnological University, 61052 Kharkiv, Ukraine; vavoitovva@btu.kharkiv.ua (V.V.); klkavoitov@btu.kharkiv.ua (A.V.); kravcov\_84@btu.kharkiv.ua (A.K.)
- <sup>4</sup> Department of Electricity Supply and Energy Management, State Biotechnological University, 61052 Kharkiv, Ukraine
- <sup>5</sup> Department of Materials Science and Engineering of Composite Structures, O.M. Beketov National University of Urban Economy in Kharkiv, Marshal Bazhanov Str. 17, 61002 Kharkiv, Ukraine; andrii.kondratiev@kname.edu.ua
- \* Correspondence: tarjorggg@bau.edu.jo (T.M.A.A.-Q.); omiroshnyk@btu.kharkiv.ua (O.M.)

Abstract: Based on the analysis of standards for the testing of lubricants, both liquid and plastic, on a four-ball tribometer, and the analysis of the parameters by which lubricants are evaluated, this paper proposes a methodology and an integral parameter for the estimation of tribological properties. The methodological approach proposed in this paper allows for the integration of a variety of parameters provided in the standards for the testing of lubricants into one indicator. Herein, we show that the developed technique is based on the energy approach and takes into account the specific wear work of the test material (steel balls) in the lubricating medium to be investigated. The results of laboratory tests of a wide range of lubricants are presented: hydraulic fluids, motor and transmission oils of various purposes and classifications. It is shown that the magnitude of the integral parameter can be used to assess the effectiveness of anti-wear and anti-scuff additives in base lubricants, as well as the ranges of their applications. This allows for differentiation and quantitative evaluation of the effectiveness of such additives. The obtained results allow us to state that all tests according to the developed method are reproducible and homogeneous, which is confirmed using the Cochran criterion. The coefficient of variation during testing does not exceed 18%. We show that the presented methodology and the integral parameter can be used in the first stage of the laboratory selection tests of new lubricants and additives of various origins, reducing the costs of their development and implementation.

**Keywords:** lubricants; hydraulic oils; motor oils; gear oils; four-ball tribometer; anti-wear additives; extreme pressure additives

## 1. Introduction

An analysis of publications published in various scientific journals allows us to conclude that, recently, new types of lubricant additives and dopants have been actively developed. Much attention has been paid to additives based on nanomaterials, as well as environmentally friendly lubricants based on vegetable oils. The search for the optimal concentrations of additives, the use of additives in the base lubricant and the evaluation of the effectiveness of such additives require a large number of laboratory, standard and operational tests.

In order to reduce the time and costs of such studies, developers use laboratory tests on a four-ball tribometer. Such tests are the first stage of research and allow for the rapid



Citation: Al-Quraan, T.M.A.; Alfaqs, F.; Haddad, J.; Vojtov, V.; Voitov, A.; Kravtsov, A.; Miroshnyk, O.; Kondratiev, A. A Methodological Approach to Assessing the Tribological Properties of Lubricants Using a Four-Ball Tribometer. *Lubricants* **2023**, *11*, 457. https://doi.org/10.3390/ lubricants11110457

Received: 25 August 2023 Revised: 14 October 2023 Accepted: 16 October 2023 Published: 25 October 2023



**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). evaluation of the effectiveness of lubricants, the establishment of optimal concentrations and justification for areas of research.

The second stage is the testing of new lubricants on real tribo-system constructions in the form of standard tests or controlled operation. Therefore, the use of a four-ball tribometer in the first stage of research can reduce the time required for testing and increase the efficiency of such studies.

The methodology for the study of lubricants using a four-ball tribometer is set out in the standards of various countries: GOST 9490, ASTM D4172, ASTM D2596, ASTM D2783, ASTM D5183 and DIN 51350. The standards allow for the determination of several parameters, the physical meanings of which differ and are sometimes contradictory, making it difficult to formulate specific conclusions. In our opinion, the tribological properties of a lubricant should be evaluated by an integral indicator that takes into account the interaction of the lubricating medium (additives and dopants in the base lubricating medium) with friction surface materials under various loads, revealing the mechanisms of physical adsorption and chemisorption and the chemical reactions that affect the wear resistance of the tribo-system. Such an approach can increase the efficiency of the development of new lubricants and make the test procedure on a four-ball tribometer more informative.

An overview of various designs of tribometers that are used in the laboratory testing of materials and lubricants for friction and wear is presented in [1]. Among the variety of test equipment constructions, specific consideration is given to the four-ball tribometer. This device is used to determine the tribological characteristics of lubricants, both liquid and plastic.

According to the authors of [2], traditional tests on a four-ball tribometer make it possible to optimize base lubricant additives and dopants through an assessment of their effective actions. We believe that such a methodological approach makes it possible to reduce the number of preliminary tests and to detail the work of extreme pressure additives.

The authors of [3] used a four-ball tribometer to test the extreme pressure properties of lubricants according to ASTM D2596. The authors investigated various lubricants and their ability to prevent burrs. A feature of the tests was the change in the rotational speed of the tribometer rotor. The authors found that the change in the rotor speed and the acceleration time of the rotor changed the magnitude of the burr load. In our opinion, these studies show the necessity of meeting the requirements of the standards for testing using a four-ball tribometer, which set out the testing procedure. Otherwise, the results obtained in different laboratories and by different researchers will vary significantly, making it impossible to analyze such data and create databases. Changes to the methods set out in the standards should be substantiated.

The anti-wear and extreme pressure properties of motor oils were studied in [4–6] using a four-ball tribometer according to ASTM D4172 and ASTM D2783. The tribological characteristics of fresh and used SAE 15W40 and SAE 20W50 engine oils were evaluated at different operating times in the engine [5]. The presented results allow us to state that the chosen test method is highly informative. This method allows for the evaluation of the effectiveness of additives in motor oils, such as colloidal particles and ionic liquids [4], as well as carbon nanotubes [6]. These works confirm the high sensitivity and information contents of tests conducted on a four-ball tribometer.

A study of the efficiency of the use of graphene-based organic additives based on graphite, which provide a tribo-chemical reaction during friction with metal surfaces, was described in [7]. The authors of the work showed the high efficiency of using a four-ball tribometer in the study of such additives. According to the test results, organic additives are physically or chemically adsorbed on rubbing metal surfaces with the formation of monolayers, which indicates the high information content of the chosen method.

The results of tests of greases conducted on a four-ball tribometer were presented in [8–10]. For example, the addition of ash from rice husk was shown in [8]. In [9,10], additives to lithium grease in the form of talc nanoparticles were studied. The main conclusion of the works listed above was that tests using a four-ball tribometer can achieve highly reproducible results.

Therefore, we can state that the use of a four-ball tribometer in the first stage of the qualifying tests of oils, lubricants, additives and dopants can help to obtain reliable information about the selected areas of research, introducing corrections into research in the first stages of testing and reducing the financial costs and time spent on controlled exploitation [9–12].

A good result was achieved using a four-ball tribometer in the study of the tribological characteristics of vegetable oils. The investigation of sunflower vegetable oil with nano-additives at various concentrations was shown in [13], which allowed the authors to establish the optimal concentration. The purpose of the studies presented in the article was to assess the anti-wear characteristics of vegetable-based lubricants. The test results from the presented work enable us to assert the high information value of employing a four-ball tribometer in studying the anti-wear and extreme pressure properties of vegetable-based lubricants.

The evaluation of the tribological characteristics of various lubricants, both liquid and plastic, with the addition of nanoparticles, where the main tool is a four-ball tribometer, was described in [14–21]. The tribological properties of  $SiO_2$  nanoparticles used as additives to a base oil were studied in [15]. In this work, the authors used a four-ball tribometer to evaluate the anti-wear properties of the base oil in comparison with  $SiO_2$  nanoparticles in the base oil. A strong correlation was observed between the anti-wear properties of oils with additives, their solubility, and the additive content in the base oil. This affirms the high information value of the chosen research method [16].

The tribological properties of a paraffin lubricant with ZnO and CuO nanoparticles were studied in [17]. Using a four-ball tribometer, the extreme pressure and anti-wear properties of lubricants with nano-additives were assessed according to the ASTM D2596 and ASTM D2266 standards.

In [18], the tribological performance of castor grease with and without two-dimensional (2D) lamellar nanomaterials was evaluated using a four-ball tribo-tester following the ASTM standards. The test results demonstrated high measurement reproducibility.

Nanoparticles, specifically activated carbon, in lithium grease, were discussed in [19,20], while hybrid nano-oils were studied in [21,22]. The possibility of using nanoparticles and polytetrafluoroethylene simultaneously was considered by the authors of [23].

The tribological characteristics of lubricants containing carbon spheres were discussed in [24]. In all these works, experimental studies were conducted using a four-ball tribometer. The authors of these works highlight the high reproducibility of the experimental results.

The works in [25,26] consisted of experimental studies on the tribological characteristics of lubricants using a four-ball tribometer with polytetrafluoroethylene additives. Appropriate concentrations of polytetrafluoroethylene additives in base oils were established.

All the works mentioned relied on tests conducted using a four-ball tribometer [14–26]. In [27,28], the authors proposed an energy parameter, namely the specific work of the wear of a test material composed of ball-bearing steel in the tested lubricating medium, to assess the tribological characteristics of lubricants. The study demonstrated that the value of the specific wear work can serve as a comprehensive energy parameter for the evaluation of the lubricating properties of liquid and plastic materials. The presented methodology and parameters for the assessment of the effectiveness of anti-wear and extreme pressure additives include the mandatory determination of the wear index and its inclusion in the general formula. In our opinion, this methodological approach reduces the information content of the test results obtained from a four-ball tribometer [24,29,30]. Excluding the wear indicator from the general formula for the estimation of the integral parameter can significantly enhance the method's information content.

According to the authors of the referenced works [14–28], testing on a four-ball tribometer provides highly reproducible results across multiple experiments and offers valuable data. In our view, conducting tests on a four-ball tribometer should be the primary and essential step in the study of lubricants, both liquid and plastic. These tests help to identify areas for further research in terms of developing promising lubricants and determining the initial optimal content of additives and dopants in the base lubricant [31]. A prerequisite for such studies, constituting the second stage of testing, is the implementation of bench tests on actual machinery or controlled operation tests. During this phase, optimal concentrations will be refined, and factors such as the friction losses and wear rates of the primary tribo-systems in the machinery will be determined when employing new lubricants. For example, in works [32,33], plant-based additives were examined as environmentally friendly additives to lubricants using a four-ball tribometer. Studies involving nanomaterial-based additives, such as fullerenes [34,35], were conducted in works [34–40]. Such investigations necessitate rapid and efficient laboratory tests to identify directions for further research. The work [28] demonstrates the utilization of an integral parameter in assessing the tribological properties of lubricants in mathematical models.

An analysis of the methods using a four-ball tribometer, as outlined in ASTM D4172 and ASTM D2783, reveals several crucial indicators.

Point B on the ASTM D2783 characteristic curve, which characterizes a lubricant, determines whether the lubricant possesses anti-wear properties. These properties are typically conferred by surfactants and their physical adsorption to the friction surface. A shift in point B to the right on the graph indicates the presence of a surfactant in the lubricant, functioning as an anti-wear additive. The primary mechanism of interaction between such additives and the friction surface is physical adsorption.

Conversely, the ASTM D2783 standard does not mandate tests at point A. For instance, GOST 9490 prescribes test conditions at point A, which entails a load of 196 N or 20 kg. This load applies to all lubricants except gear oils. For gear oils, the load at point A is increased to 392 N or 40 kg. Consequently, an AB segment is established, signifying specific physical attributes. This range represents the performance of anti-wear additives in a lubricant with a broader AB range situated lower on the graph field, indicating the superior anti-wear properties of the lubricant.

Section 2, as presented in the ASTM D2783 standard, is the BC segment, which characterizes the rapid increase in the wear scar on the three lower balls as the load increases. Since the load increases in accordance with the standard's load series, the temperature in the friction zone also rises. The elevated temperature causes the physical adsorption of lubricant molecules to the friction surface to diminish. Consequently, the wear scar and temperature on the friction surface increase rapidly. This rise in temperature indicates chemical reactions between the extreme pressure additives and the friction surface material. On the curve, there is a transition from point B to point C, accompanied by deceleration (or a halt) in the growth of the wear scar. The BC segment signifies the presence and effective operation of extreme pressure additives in the lubricant. If extreme pressure (EP) additives are not available, at point C, the scoring of the friction surfaces begins, and the balls may weld together. However, a large BC segment leads to a high wear rate in the materials due to active chemical processes occurring on the friction surfaces. The seizure of the friction surfaces is eliminated, and the wear rate of the materials is increased. A smaller BC segment indicates superior extreme pressure properties of the lubricant.

Section 3, represented by the CD segment, characterizes the performance of the extreme pressure additives in the lubricant. With an increase in the test load, following the standard's load series, the temperature in the friction zone increases significantly. The temperature is the driving force behind the chemical reactions on the friction surfaces that prevent the scuffing or welding of the balls. The physical meaning of the CD segment indicates the effectiveness of the extreme pressure additives in the lubricant. The wider the CD segment, the better the extreme pressure properties of the lubricant. However, the height of point D on the graph negatively affects the wear resistance. Due to the active reactions of the chemical elements in extreme pressure additives with the metal surface, the size of the wear scar increases significantly. Such a lubricant prevents seizure but results in a high wear rate. Ideally, point D should be positioned as far to the right as possible on the graph field and below. The next load after point D is the welding load, at which either the four-ball pyramid welds together in an oil bath or the four-ball tribometer automatic shuts down. This occurrence signifies the loss of extreme pressure properties in the lubricant.

In our view, a test on a four-ball tribometer can serve as the initial stage in evaluating liquid and plastic lubricants, offering a preliminary assessment of their tribological characteristics. This method enables the determination of the directions for further research in the development of new lubricants at a minimal cost.

The purpose of this investigation was to establish an integral parameter for the evaluation of the tribological properties of lubricants, providing a differentiated assessment of the contribution of anti-wear and extreme pressure additives or dopants.

## 2. Materials and Methods

The testing unit, following the four-ball scheme, is depicted in Figure 1. It consists of three fixed lower balls (1) and one upper rotating ball (2) secured with a collet (3). The upper rotating ball (2) is subjected to pressure, represented by the load N, against the three lower balls (1).



**Figure 1.** Scheme of a four-ball friction unit. 1—three lower fixed balls; 2—rotating upper ball; 3—collet; *N*—load.

The external view of the four-ball assembly is shown in Figure 2, and a four-ball tribometer is illustrated in Figure 3.



**Figure 2.** External view of the four-ball assembly. 1—fixed lower balls; 2—a glass for a lubricant to be tested; 3—nut for fixture of the three lower balls.



Figure 3. External view of a four-ball tribometer.

The foundation for the development of an integral parameter for the assessment of the tribological properties of lubricants, with a differentiated evaluation of the contribution of anti-wear and extreme pressure additives, was presented in [27]. The study introduced an energy parameter—the specific wear work of the test material (ball-bearing steel balls) in the tested lubricating medium. This parameter characterizes the amount of friction work required to remove a unit volume of material from the friction surface, expressed as  $J/m^3$ , in the form of wear particles. The specific wear work serves as an integral energy parameter whose value can be used to evaluate the anti-wear and extreme pressure properties of lubricants. This manuscript is a continuation of the work presented in [26] and offers an improved technique for the assessment of tribological properties. This work takes into account the requirements outlined in the ASTM D4172 and ASTM D2783 standards.

The specific wear work A is calculated using the following expression:

$$A = \frac{f \cdot P \cdot l}{d^3}, \ J/m^3 \tag{1}$$

where *f*—the coefficient of friction during tests on a four-ball tribometer;

*P*—the load chosen based on the load range specified in the ASTM D2783 standard, dimension N;

*l*—the friction path during testing, dimension m;

*d*—the average diameter of the wear spots on the three lower balls during the test, dimension m.

This parameter can be applied to evaluate the anti-wear and extreme pressure properties of liquid and plastic lubricants.

The methodological approach involves employing this parameter according to Formula (1) for all three ranges, AB, BC, and CD, that characterize the lubricant. Such a methodological approach allows for differentiation in the effectiveness of anti-wear and extreme pressure additives. The sum of these properties results in an integral parameter reflecting the tribological properties of the lubricant [29,30].

As mentioned previously, the AB range is utilized to assess anti-wear properties, and the specific wear work for this range, denoted as  $A_{AB}$ , is calculated using the following expression:

$$A_{AB} = \sum_{i=196}^{P_B} \frac{f_i \cdot P_i \cdot l_i}{d_i^3}, \, J/m^3$$
(2)

where  $\sum_{i=196}^{P_B}$  —the sum of the values of tests from a load of 196 N (20 kg) to the load preceding the point where the wear scar diameter increases. Such a load is denoted as  $P_B$ . According to the aforementioned standards for four-ball tribometer testing, this increase in diameter should be at least 0.1 mm.

 $f_i$ —the friction coefficient value at loads ranging from 196 N to  $P_B$ .

 $P_i$ —the load value from 196 N to  $P_B$ , N, applied according to the load range specified in ASTM D2783. Tests must be carried out sequentially at all loads specified in the standard.

 $l_i$ —the friction path during testing, with the standard recommending a testing duration of 10 s, resulting in a friction path of l = 5.88 m.

 $d_i$ —the average diameter of the wear spots on the three lower balls under loads ranging from 196 N to  $P_B$ , N, dimension m.

It should be noted that if the test duration remains consistent at 10 s, then the friction path remains the same, l = 5.88 m.

The higher the value of the specific wear work within the AB segment, the better the anti-wear properties of the lubricant.

The second segment, *BC*, which signifies the shift from anti-wear properties to extreme pressure properties, can be determined using the following expression:

$$A_{BC} = \sum_{P_{B+1}}^{P_C} \frac{f_k \cdot P_k \cdot l_k}{d_k^3}, \, \text{J/m}^3$$
(3)

where  $\sum_{P_{B+1}}^{P_C}$  —the total value of tests from the load at which the wear scar diameter increases

by at least 0.1 mm, denoted as  $P_{B+1}$ , up to the load where the increase in the wear scar diameter ceases, denoted as  $P_C$ .

 $f_k$ —friction coefficient value under loads ranging from  $P_{B+1}$  to  $P_C$ .

 $P_k$ —the load value from  $P_{B+1}$ , N, to  $P_C$ , N, applied according to the load series specified in ASTM D2783. Tests should be performed sequentially at all loads outlined in the standard.  $l_k$ —the friction path during testing, equal to 5.88 m.

 $d_k$ —the average diameter of the wear spots on the three lower balls under loads ranging from  $P_{B+1}$ , N, to  $P_C$ , N, dimension m.

Based on experience gained from testing various lubricants, it is observed that the range of the BC segment typically encompasses no more than two loads from the load range.

The greater the specific wear work value in the BC segment, the faster the EP additives of the lubricant come into effect, and the more effective their action.

The third segment, denoted as CD, characterizes the performance of extreme pressure additives and can be determined using the following expression:

$$A_{CD} = \sum_{P_{C+1}}^{P_D} \frac{f_j \cdot P_j \cdot l_j}{d_j^3}, \ J/m^3$$
(4)

where  $\sum_{P_{C+1}}^{P_D}$  —the total value of tests from the load where the growth in the wear scar ceases,

denoted as  $P_{C+1}$ , up to the load that precedes the welding load, denoted as  $P_D$ .

 $f_i$ — the friction coefficient value under loads ranging from  $P_{C+1}$  to  $P_D$ .

 $P_j$ —the load value from  $P_{C+1}$ , N, to  $P_D$ , N, applied according to the load series specified in ASTM D2783. Tests must be conducted sequentially at all loads outlined in the standard.  $l_i$ —the friction path during testing, equal to 5.88 m.

 $d_j$ —the average diameter of the wear spots on the three lower balls under loads ranging from  $P_{C+1}$ , N, to  $P_D$ , N, dimension m.

Based on the experience of testing various lubricants, as well as additives and dopants used with them, it is observed that not all lubricants under high loads ensure the welding of balls during testing. Some lubricants do not result in welding. Instead, there is accelerated wear of the three lower and one upper balls. In such cases, the standard recommends stopping the test with the load when the wear scar exceeds 3 mm.

The greater the value of the specific wear work in the CD segment, the better the extreme pressure properties of the lubricant. This area can be referred to as the "carrying capacity" of the lubricant, as extreme pressure additives ensure the operation of tribosystems under increased loads and elevated temperatures of the friction surfaces.

With the expressions for the calculation of the specific work of friction on three segments of the curve characterizing the lubricant's performance—see Formulas (2)–(4)—we can derive an expression for the calculation of the integral parameter of the lubricant's tribological properties, represented by the formula

$$A = A_{AB} + A_{BC} + A_{CD}, \text{ J/m}^3$$
(5)

The physical interpretation of this integral parameter for the assessment of the tribological properties of lubricant A, with a differentiated assessment of the contribution from each of these segments, is illustrated in Figure 4.



**Figure 4.** Physical interpretation of the integral parameter for assessment of the tribological properties of lubricants.

The developed methodological approach allows us, within the framework of the ASTM D2783 standard, to determine the presence of antiwear and extreme pressure properties of

a lubricant, as well as to differentially evaluate the contribution of each of the three sections to the integral properties.

#### 3. Results of the Experiments

The purpose of the experimental studies was to confirm the information content and reproducibility of the developed methodological approach and the integral parameter in assessing the tribological properties of various lubricants.

For testing, hydraulic fluids (hydraulic oils), engine oils and gear oils were chosen.

Hydraulic fluids and motor and transmission oils belong to the category of liquid lubricants. The purpose of our research was to develop an integral parameter to evaluate the tribological properties of liquid lubricants only. Excluding from this list, for example, hydraulic fluids that simultaneously serve as lubricants for hydraulic units, would make our study incomplete. Therefore, our study examined all types of liquid lubricants.

As hydraulic liquids simultaneously perform the functions of lubricants, we selected working liquids (ISO 6743-4), the tribological properties of which are presented in Table 1. The tribological properties of motor oils are presented in Table 2, and those of transmission oils in Table 3. Each type of lubricant is represented by at least three types. For instance, hydraulic fluids include four types (Table 1), motor oils include seven types (Table 2) and transmission oils include three types (Table 3).

Lubricant Classification	$A_{AB} imes 10^{11}$ , J/m $^3$	$A_{BC} imes 10^{11}$ , J/m $^3$	$A_{CD} imes 10^{11}$ , J/m $^3$	$A imes 10^{11}$ , J/m $^3$
ISO-L-HH	43.54	2.12	0	45.66
ISO-L-HL	57.64	8.87	0.69	67.2
ISO-L-HM	75.45	11.26	1.84	88.59
ISO-L-HV	83.13	13.5	2.3	98.93

Table 1. Tribological properties of hydraulic oils.

Table 2. Tribological properties of motor oils.

Lubricant Classification	$A_{AB} imes 10^{11}$ , J/m $^3$	$A_{BC} imes$ 10 <sup>11</sup> , J/m <sup>3</sup>	$A_{CD} imes 10^{11}$ , J/m $^3$	$A imes 10^{11}$ , J/m <sup>3</sup>
SAE 20W-30 API SF	92.93	13.13	2.49	108.55
SAE 10W-30 API SH	113.2	16.12	2.89	132.21
SAE 20W-40 API CD	124.2	17.8	3.1	145.1
SAE 20W-40 API SJ/CF	159.7	19.5	3.72	182.92
SAE 20W-40 API CF-4	198.7	21.7	4.5	225.27
SAE 10W-40 API SL/CF	224.65	23.4	4.87	252.92
SAE 5W-30 APICH	258.26	25.2	5.18	288.64

Lubricant Classification	$A_{AB} imes 10^{11}$ , J/m $^3$	$A_{BC} imes 10^{11}$ , J/m $^3$	$A_{CD} imes 10^{11}$ , J/m $^3$	$A imes 10^{11}$ , J/m $^3$
SAE 85W-90 API GL-3	67.55	13.44	8.08	89.07
SAE 80W-90 API GL-4	165.72	24.64	12.43	202.79
SAE 75W-90 API GL-5	213.25	31.75	15.54	260.54

Table 3. Tribological properties of gear oils.

The choice of oil brands was intended to cover the entire range of tribological properties of lubricants, from minimum values to maximum, in accordance with the requirements of the ASTM D4172 and ASTM D2783 standards. With this methodological approach, we considered the entire range of properties that exist today in the liquid lubricant market. We do not refer to specific brands of lubricants but to their classification according to SAE and API.

The abovementioned lubricants were tested in accordance with the ASTM D2783 standard, using the specified load range. Testing for hydraulic and motor oils began with a load of 196 N or 20 kg, with a sequential load series. For gear oils, testing commenced with a load of 392 N or 40 kg.

Based on the results of three repetitions, we calculated the average diameter values of the drift flames on the three lower balls, the root mean square values of the registration values of the diameters of the drift flames during the experimental studies and the coefficient of variation in the measurement of the diameter.

The root mean square deviation of the diameter values of the wear spot during experimental studies was determined using the following formula:

$$S_d = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (d_i - d_{av})^2}, m,$$
 (6)

where  $d_i$ ,  $d_{av}$ —the diameter of each wear spot in the sample and the average value for the number of repetitions n, respectively, dimension m.

*n*—the number of results obtained during the testing of the coating material.

The coefficient of variation in the diameter measurements of wear spots was determined as follows:

$$v_d = \left(\frac{S_d}{d_{av}}\right) \cdot 100\%. \tag{7}$$

The experimental sample of wear spot diameter values obtained during the experimental studies was assessed for uniformity and reproducibility across multiple experiments using Cochran's C test:

$$G_{p} = \frac{S_{d(\max)}^{2}}{\sum_{i=1}^{n} S_{d(i)}^{2}},$$
(8)

where  $S_{d(\max)}^2$ —the maximum value of the dispersion of the wear spot diameters ( $d_i$ ) measured during the experiment;

 $S_{d(i)}^2$ —the value of the variance of the *i*-th experiment for the parameters to be measured during the experiment— $d_i$ .

The hypothesis was tested using the following condition:

$$G_p < G_{table},\tag{9}$$

where  $G_{table}$ —the tabular value for Cochran's C test, with a predefined confidence factor q = 0.90.

11 of 15

If condition (9) is met, it indicates that the obtained experimental results exhibit uniformity and reproducibility.

#### 4. Discussion

As indicated in the tables above, the integral parameter A—see Formula (5)—dimension J/m<sup>3</sup>, exhibits significant variation across different lubricants. For instance, hydraulic oils have A values ranging from 45.66 to  $98.93 \times 10^{11}$  J/m<sup>3</sup>, engine oils show values between 108.55 and  $288.64 \times 10^{11}$  J/m<sup>3</sup> and gear oils exhibit values ranging from 89.07 to  $260.54 \times 10^{11}$  J/m<sup>3</sup>.

The most pronounced result was obtained when examining the anti-wear and extreme pressure properties separately. The value of the  $A_{AB}$  range indicates the presence of anti-wear additives or dopants in the lubricant. Notably, motor oils (as shown in Table 2) tend to display the maximum values of this range, while hydraulic oils (as seen in Table 1) exhibit the minimum values. This range serves as a useful metric for the evaluation of the effectiveness of anti-wear additives and dopants in a lubricant.

On the other hand, the value of the  $A_{CD}$  range signifies the presence of extreme pressure additives or dopants within the lubricant. Here, gear oils (Table 3) tend to exhibit the maximum values. For instance, the  $A_{CD}$  range varies within 8.08–15.54×10<sup>11</sup> J/m<sup>3</sup>. In contrast, hydraulic oils (Table 1) display the minimum values, ranging from 0 to 2.3 ×10<sup>11</sup> J/m<sup>3</sup>. This range is instrumental in assessing the effectiveness of EP additives and lubricant dopants.

Furthermore, the value of the  $A_{BC}$  range provides insights into the effectiveness of the transition from antiwear to extreme pressure properties within the lubricant. Gear oils (Table 3) generally show higher values in the  $A_{BC}$  range, signifying a more effective transition to extreme pressure additives. This suggests that the gear oils selected for testing possessed a superior set of extreme pressure additives compared to the engine oils presented.

The laboratory test results for various lubricants assessed on a four-ball tribometer led to the following conclusion (as summarized in Table 4).

- 1. All test results were reproducible and uniform, with the calculated values of Cochran's C test (Formula (8)) being less than the tabulated values at a given confidence level of probability q = 0.95. This applies to scenarios where the number of test repetitions was 3 and the number of logged values was 9. Consequently, the condition was satisfied; see Formula (9).
- 2. The coefficient of variation during testing did not exceed 18%; see Formula (7). This indicates the minimal scatter of the test results.
- 3. The technique presented in this article and the integral parameter for the evaluation of the tribological properties of lubricants—see Formula (5)—can be used at the initial stage of the qualifying laboratory tests of new lubricants, additives, and dopants of various origins.

The physical interpretation of the tribological properties of lubricants for various purposes, along with characteristic areas of operation for various additives, is shown in Figure 5.

The format adopted for the presentation of the research results in Figure 5 allows us to illustrate the physical meaning of the integral parameter used to assess the tribological properties of liquid lubricants. The presented Figure 5 clearly differentiates among various categories of lubricants. The *x*-axis represents the load range that the lubricant can withstand, characterizing its extreme pressure properties. Greater values indicate better anti-scuff properties. The *y*-axis reflects the ability of the lubricant to prevent wear, with higher integral parameter A values signifying better anti-wear properties.

The developed technique and the integral parameter for the estimation of the tribological properties of lubricants differ from those that are already known because they are based on an energy approach—the specific work of wear determined on the test material (balls made of ball-bearing steel) in a lubricating medium, as presented in this research. This parameter characterizes the amount of friction work required to remove a unit volume of material from the friction surface (dimension J/m<sup>3</sup>) in the form of wear particles. This approach enables the evaluation of the lubricant as a whole and the differentiation of its components, including its anti-wear and extreme pressure properties.

Lubricant	$S_d  imes 10^{-3}$ ,	<i>v<sub>d</sub></i> ,	G <sub>p</sub>	G <sub>table</sub>
	III	/0	•	
ISO-L-HH	0.155	17.32	0.432	0.478
ISO-L-HL	0.137	17.24	0.413	0.478
ISO-L-HM	0.113	16.26	0.412	0.478
ISO-L-HV	0.09	14.53	0.388	0.478
SAE 20W-30 API SF	0.052	13.23	0.410	0.478
SAE 10W-30 API SH	0.043	12.52	0.410	0.478
SAE 20W-40 API CD	0.041	11.83	0.394	0.478
SAE 20W-40 API SJ/CF	0.04	11.54	0.380	0.478
SAE 20W-40 API CF-4	0.04	11.72	0.345	0.478
SAE 10W-40 API SL/CF	0.039	10.49	0.340	0.478
SAE 5W-30 APICH	0.039	10.28	0.340	0.478
SAE 85W-90 API GL-3	0.055	14.32	0.410	0.478
SAE 80W-90 API GL-4	0.047	13.55	0.380	0.478
SAE 75W-90 API GL-5	0.042	10.45	0.375	0.478

**Table 4.** Lubricant statistical parameters.



**Figure 5.** Physical interpretation of the integral parameter for assessment of the tribological properties of lubricants: 1—ISO-L-HM hydraulic oil; 2—engine oil SAE 5W-30, API CH; 3—gear oil SAE 75W-90, API GL-5.

Further development of this method for the testing of lubricants on a four-ball tribometer aims to enhance the efficiency of screening tests during the development of new lubricants, such as vegetable oils. Lubricant additives using nanomaterials, such as fullerenes, are actively being developed, as discussed in [26]. Additionally, this parameter can be utilized in modeling friction and wear processes in tribo-systems to monitor the lubricating medium, as presented in [24,27].

## 5. Conclusions

Based on the analysis of standards for the testing of lubricants—both liquid and plastic—an energy parameter is proposed: the specific work of wear of the test material (steel balls) in the lubricating medium to be investigated. Unlike known parameters, this energy parameter is associated with the work of friction expended in removing the volume of material from the friction surface during testing. Experimental evidence demonstrates that this proposed parameter evaluates the range of operation of anti-wear and extreme pressure additives in the lubricant, allowing for the differentiation of their effectiveness.

The results of laboratory tests on various lubricants using a four-ball tribometer verify that, according to the developed method, all tests are reproducible and uniform, as confirmed by Cochran's C test. The coefficient of variation during testing does not exceed 18%, indicating minimal variation in the test results.

The technique presented in this article and the integral parameter for the assessment of the tribological properties of lubricants can be applied in the initial stages of laboratory selection tests for new lubricants, additives, and dopants of various origins.

Author Contributions: Conceptualization, T.M.A.A.-Q., F.A., J.H. and V.V.; methodology, T.M.A.A.-Q., O.M., A.V., F.A., A.K. (Andrey Kravtsov) and J.H.; software, V.V., O.M., A.K. (Andrey Kravtsov) and F.A.; validation, O.M. and A.K. (Andrii Kondratiev); formal analysis, T.M.A.A.-Q. and V.V.; investigation, F.A., O.M. and A.V.; resources, T.M.A.A.-Q., O.M., A.V. and A.K. (Andrey Kravtsov); data curation, J.H., O.M. and A.V.; writing—original draft preparation, T.M.A.A.-Q., F.A., J.H., A.K. (Andrei Kondratiev). and V.V.; writing—review and editing, T.M.A.A.-Q., O.M., A.K. (Andrei Kondratiev) and A.V.; visualization, F.A., J.H., A.K. (Andrey Kravtsov) and O.M.; supervision, V.V., O.M. and A.V. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

**Data Availability Statement:** The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

### References

- 1. Stachowiak, G.W.; Batchelor, A.W.; Stachowiak, G.B. 3—Tribometers. Tribol. Ser. 2004, 44, 25–78. [CrossRef]
- Ji, X.; Chen, Y.; Zhao, G.; Wang, X.; Liu, W. Tribological Properties of CaCO<sub>3</sub> Nanoparticles as an Additive in Lithium Grease. *Tribol. Lett.* 2011, 41, 113–119. [CrossRef]
- 3. Joysula, S.K.; Dube, A.; Patro, D.; Veeregowda, D.H. On the Fictitious Grease Lubrication Performance in a Four-Ball Tester. *Lubricants* **2021**, *9*, 33. [CrossRef]
- 4. Beheshti, A.; Huang, Y.; Ohno, K.; Blakey, I.; Stokes, J.R. Improving tribological properties of oil-based lubricants using hybrid colloidal additives. *Tribol. Int.* 2020, 144, 106130. [CrossRef]
- 5. Yadav, G.; Tiwari, S.; Jain, M.L. Tribological analysis of extreme pressure and anti-wear properties of engine lubricating oil using four ball tester. *Miner. Process.* 2018, *5*, 248–253. [CrossRef]
- 6. Xie, H.; Wei, Y.; Jiang, B.; Tang, C.; Nie, C. Tribological properties of carbon nanotube/SiO<sub>2</sub> combinations as water-based lubricant additives for magnesium alloy. *J. Mater. Res. Technol.* **2021**, *12*, 138–149. [CrossRef]
- Kumar, N.; Saini, V.; Bijwe, J. Tribological investigations of nano and micro-sized graphite particles as an additive in lithium-based grease. *Tribol. Lett.* 2020, 44, 124. [CrossRef]
- Akhtar, K.; Khalid, H.; Haq, I.U.; Malik, A. Improvement in tribological properties of lubricating grease with quartz-enriched rice husk ash. *Tribol. Int.* 2016, 93, 58–62. [CrossRef]
- Saini, V.; Bijwe, J.; Seth, S.; Ramakumar, S. Potential exploration of nano-talc particles for enhancing the anti-wear and extreme pressure performance of oil. *Tribol. Int.* 2020, 151, 106452. [CrossRef]

- 10. Kumar, N.; Saini, V.; Bijwe, J. Exploration of Talc nanoparticles to enhance the performance of Lithium grease. *Tribol. Int.* 2021, *162*, 107107. [CrossRef]
- 11. Havrylenko, Y.; Kholodniak, Y.; Halko, S.; Vershkov, O.; Miroshnyk, O.; Suprun, O.; Dereza, O.; Shchur, T.; Śrutek, M. Representation of a Monotone Curve by a Contour with Regular Change in Curvature. *Entropy* **2021**, *23*, 923. [CrossRef] [PubMed]
- Khasawneh, A.; Qawaqzeh, M.; Miroshnyk, O.; Danylchenko, D.; Minakova, K.; Potryvai, A. Methodology for Accounting for the Influence of Dust Cover on the Performance of a Photovoltaic System for Matlab. In Proceedings of the 20th IEEE International Conference on Modern Electrical and Energy Systems, MEES 2021, Kremenchuk, Ukraine, 21–24 September 2021. [CrossRef]
- 13. Fox, N.J.; Tyrer, B.; Stachowiak, G.W. Boundary Lubrication Performance of Free Fatty Acids in Sunflower Oil. *Tribol. Lett.* 2004, 16, 275–281. [CrossRef]
- 14. Zhao, F.Y.; Li, G.T.; Zhang, G.; Wang, T.M.; Wang, Q.H. Hybrid effect of ZnS sub-micrometer particles and reinforcing fibers on tribological performance of polyimide under oil lubrication conditions. *Wear* 2017, *380–381*, 86–95. [CrossRef]
- 15. Rawat, S.S.; Harsha, A.P.; Deepak, A.P. Tribological performance of paraffin grease with silica nanoparticles as an additive. *Appl. Nanosci.* **2019**, *9*, 305–315. [CrossRef]
- Havrylenko, Y.; Kholodniak, Y.; Halko, S.; Vershkov, O.; Bondarenko, L.; Suprun, O.; Miroshnyk, O.; Shchur, T.; Śrutek, M.; Gackowska, M. Interpolation with Specified Error of a Point Series Belonging to a Monotone Curve. *Entropy* 2021, 23, 493. [CrossRef]
- 17. Rawat, S.S.; Harsha, A.P.; Das, S.; Deepak, A.P. Effect of CuO and ZnO nano-additives on the tribological performance of paraffin oil-based lithium grease. *Tribol. Trans.* 2020, *63*, 90–100. [CrossRef]
- Rawat, S.S.; Harsha, A.P.; Khatri, O.P. Tribological investigations of two-dimensional nanostructured lamellar materials as additives to castor-oil-derived lithium grease. J. Tribol. 2022, 144, 091902. [CrossRef]
- 19. Vyavhare, K.; Aswath, P.B. Tribological Properties of Novel Multi-Walled Carbon Nanotubes and Phosphorus Containing Ionic Liquid Hybrids in Grease. *Front. Mech. Eng.* **2019**, *5*, 15. [CrossRef]
- 20. Iegorov, O.; Iegorova, O.; Miroshnyk, O.; Savchenko, O. Improving the accuracy of determining the parameters of induction motors in transient starting modes. *Energetika* **2020**, *66*, 15–23. [CrossRef]
- Saini, V.; Bijwe, J.; Seth, S.; Ramakumar, S. Augmenting the lubrication performance of nano-oils through synergistic cofunctioning of nanoparticles. *Tribol. Int.* 2023, 182, 108332. [CrossRef]
- 22. Kondratiev, A.; Píštěk, V.; Smovziuk, L.; Shevtsova, M.; Fomina, A.; Kučera, P.; Prokop, A. Effects of the temperature–time regime of curing of composite patch on repair process efficiency. *Polymers* 2021, *13*, 4342. [CrossRef]
- Saini, V.; Bijwe, J.; Seth, S.; Ramakumar, S. Role of base oils in developing extreme pressure lubricants by exploring nano-PTFE particles. *Tribol. Int.* 2020, 143, 106071. [CrossRef]
- 24. Panickar, R.; Sobhan, C.B.; Chakravorti, S. Investigations on tribological properties of non-catalytic CVD synthesized carbon spheres in lubricant. *Diam. Relat. Mater.* 2020, *106*, 107834. [CrossRef]
- Saini, V.; Bijwe, J.; Seth, S.; Ramakumar, S. Interfacial interaction of PTFE sub-micron particles in oil with steel surfaces as excellent extreme-pressure additive. J. Mol. Liq. 2021, 325, 115238. [CrossRef]
- Solanki, R.; Saini, V.; Bijwe, J. Exploration of PTFE sub-micron particles for enhancing the performance of commercial oils. *Surf. Topogr. Metrol. Prop.* 2021, 9, 025005. [CrossRef]
- Vojtov, V.; Biekirov, A.; Voitov, A. The quality of the tribosystem as a factor of wear resistance. *Int. J. Eng. Technol.* 2018, 7, 25–29. [CrossRef]
- Al-Quraan, T.M.A.; Alfaqs, F.; Alrefo, I.F.S.; Vojtov, V.; Voitov, A.; Kravtsov, A.; Miroshnyk, O.; Kondratiev, A.; Kučera, P.; Píštěk, V. Methodological Approach in the Simulation of the Robustness Boundaries of Tribosystems under the Conditions of Boundary Lubrication. *Lubricants* 2023, 11, 17. [CrossRef]
- 29. Al-Quraan, T.M.A. Influence Evaluation of the RVS Friction Geomodifier on Tribotechnical Parameters of the Contact in Non-Stationary working Conditions. *Tribol. Ind.* 2020, 42, 121–130. [CrossRef]
- Al-Quraan, T.M.A. A Manifestation of Non Hydrodynamic Factors in Steady State Conditions of Friction. J. Mech. Eng. 2017, 14, 109–124. Available online: https://ir.uitm.edu.my/id/eprint/21124 (accessed on 31 December 2017).
- Kondratiev, A.V.; Kovalenko, V.O. Optimization of design parameters of the main composite fairing of the launch vehicle under simultaneous force and thermal loading. *Space Sci. Technol.* 2019, 25, 3–21. [CrossRef]
- 32. Ali, A.I.; Hamid, M.K.A.; Abas, M.A.B.; Said, M.F.M.; Mazali, I.I.; Daud, Z.H.C.; Opia, A.C. Experimental analysis of tribological performance of base ficus carica vegetable oil with different polymers as additives using four ball tribometer. *Mater. Today Proc.* 2023, *in press.* [CrossRef]
- Kumar, A.; Chaudhary, R.; Singh, R.C. Tribological performance of various blends of commercial SAE 40 oil and novel apricot oil-based bio-lubricant using a four-ball tester tribometer. *Int. J. Mater. Prod. Technol.* 2023, 67, 166–177. [CrossRef]
- Tarun, S.; Keishing, S.; Sharma, S. Tribological analysis of nanoparticles in lubricants using four ball tester. *Int. Res. J. Mod. Eng. Technol. Sci.* 2023, 5, 5064–5066. [CrossRef]
- 35. Anurag, S.; Prashant, C.; Mamatha, T.G. A review on tribological performance of lubricants with nanoparticles additives. *Mater. Today Proc.* **2020**, *25*, 586–591. [CrossRef]
- Shahnazar, S.; Bagheri, S.; Hamid, S. Enhancing lubricant properties by nanoparticle additives. *Int. J. Hydrog. Energy* 2015, 41, 3153–3170. [CrossRef]

- 37. Ali, I.; Basheer, A.; Kucherova, A.; Memetov, N.; Pasko, T.; Ovchinnikov, K.; Pershin, V.; Tkachev, A. Advances in carbon nanomaterials as lubricants modifiers. *J. Mol. Liq.* **2019**, 279, 251–266. [CrossRef]
- 38. Yao, Y.; Wang, X.; Guo, J.; Yang, X.; Xu, B. Tribological property of onion-like fullerenes as lubricant additive. *Mater. Lett.* **2008**, *62*, 2524–2527. [CrossRef]
- Rapoport, L.; Feldman, Y.; Homyonfer, M.; Cohen, H.; Sloan, J.; Hutchison, J.L.; Tenne, R. Inorganic fullerene-like material as additives to lubricants: Structure–function relationship. *Wear* 1999, 225–229, 975–982. [CrossRef]
- Yunusov, F.A.; Breki, A.D.; Vasilyeva, E.S.; Tolochko, O.V. The Influence of Nano Additives on Tribological Properties of Lubricant Oil. Mater. Today Proc. 2020, 30, 632–634. [CrossRef]

**Disclaimer/Publisher's Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.