




Article

The Standard for Assessing Water Resistance Properties of Lubricating Grease Using Contact Angle Measurements

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Abstract: Many grease-lubricated machines operate in wet environments, and are vulnerable to contamination because of water exposure. Reports suggest that even the presence of 1% water in grease reduces the life of a bearing by 90%. Nevertheless, only a few qualitative tests and standards are available to characterize the water resistance properties of greases. In this paper, we propose a standard for evaluating the water resistance properties of greases by studying their hydrophobic and hydrophilic nature via a custom-designed apparatus for measuring the grease contact angle. In this approach, a water droplet is dispensed onto the surface of the grease and the contact angle of the droplet is studied. For this purpose, an apparatus was designed, built, and tested with twelve different greases. To validate the efficacy of the test method and setup, tests were performed at two different locations by independent operators. From the obtained contact angle values, the authors propose categorizing a grease's water-resistance properties into five different grades that can be set as guidelines for the industrial user when selecting a grease for machinery operation in a wet environment. The classification of the water-repellent properties of greases, using the proposed standard is compared with existing ASTM standards used for evaluation of grease properties in the presence of water.



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1. Introduction

Grease is a complex substance composed of oil, thickening agents, and performance additives [1]. Depending on the National Lubricating Grease Institute (NLGI) grade, the thickener content can vary from 3–30%, with additives up to 10%, and the remainder composed of oils in ratios needed to target defined viscosity grades [2]. Even with these formulation advantages, grease lubrication performance can be compromised when exposed to prolonged water contamination [3–5]. In fact, bearing fatigue life can be compromised with as little as 0.03–1% water ingress [6,7]. Overall, water resistance properties are important when selecting a grease [8]. A grease that absorbs and suspends water within the thickener matrix will, over time, release water under repetitive bearing shear. This free water content can enter the bearing race, penetrate into microcracks formed under high-pressure conditions [4], and cause many types of bearing damage due to corrosion [9], erosion [10], micro pitting [11], hydrogen embrittlement [12], and ice formation at low temperatures [13]. Further, water in the grease can lead to either an increase or decrease in yield stress [14], reduction in adhesive and cohesive properties [15,16], faster formation of acids, causing flash vaporization, erosive wear, and hydrogen embrittlement due to hydrolysis [10], increase in the wearing of the bearing [17], etc. Acknowledging these important implications for grease performance in the presence of water contamination,

the industry uses various standardized tests to assess water resistance properties before making a grease selection.

There are a few non-technical tests, such as visual inspection, static and dynamic water absorption tests, crackle tests, etc., and other notable American Standard for Testing and Materials (ASTM) standards evaluating the grease performance in the presence of water. The details of the ASTM standards are below.

- DIN 51807-1 [18] (resistance of the lubricating grease to water) is a static water resistance test where a thin strip of grease on a glass strip is dipped in a test tube with water and heated for 3 h at 40 °C or 90 °C. After heating, the glass strip is visually inspected. An evaluation is made based on the scale established in the standard (0 = no change to 3 = major change). This approach is qualitative.
- ISO 11009:2000/ASTM D1264 [19,20] (standard test method for determining the water washout characteristics of lubricating greases) assesses the resistance capability of lubricating grease to water washout from a bearing operated at ~600 rpm, with an operating temperature of 38 °C and 79 °C. The standard mentions that this test is unsuitable for greases containing highly volatile components.
- ASTM D4049 [21] (standard test method for determining the resistance of lubricating grease to water spray) assesses the ability of grease to adhere to a surface when subjected to the impingement of a water spray. This test method suggests a correlation between the operating conditions of this test and water spray impingement in steel mill roll neck bearing service.
- ASTM D8022 [22] Wet roll stability test (standard test method for roll stability of lubricating grease in the presence of water) assesses the stability of grease within a rolling apparatus when exposed to water at lower shear and operated at 20–35 °C. The wet roll stability test result is the difference in the cone penetration values measured before and after working the grease.
- ASTM D7342 [23] Water stability test (standard test method for prolonged worked stability of lubricating grease in the presence of water) assesses the stability of grease in a standard grease worker when exposed to water. The rest of the procedure is the same as ASTM D8022.

In the above-discussed standards, DIN 51807-1 is a visual-based standard and approach that evaluates the grease performance qualitatively, and results may be inconclusive. ISO 11009:2000/ASTM D1264 and ASTM D4049 test the ability of grease to adhere to the bearing surface under the impingement of water, which does not always simulate the actual operating environment. ASTM D8022 and ASTM D7342 provide information on the shear stability of grease in the presence of water based on the penetration difference before and after the test is run. The drawback of these standards is that the consistency of several greases (like calcium sulphonate, lithium complex, aluminum complex, etc.) increases with the presence of water (grease becomes firmer) while several other greases (like poly urea, silicone, etc.) lose their consistency with water (grease becomes softer) [14,16]. From the authors' perspective, grease surface adhesion and shear stability do not fully represent the water resistance properties of grease operating in actual service.

Using these standardized tests, different reports in the existing literature have classified calcium carbonate [4–25], aluminum complex [24], polyurea [26], and lithium complex formulated with synthetic oils [14] as greases with good water resistance properties. However, Leckner [16] found that higher water content in calcium sulphonate grease causes a thickening effect and loss of mechanical stability. In addition, a very recent review article [16] discusses how the presence of water can be fatal to a bearing operation. Yet an appropriate, quantitative criterion for assessing a grease's resistance to water remains elusive.

Given the shortcomings of the existing methods, the present work addresses the need for a quantitative assessment of the water-resistant properties of grease. The performance of grease in water depends on the cumulative effect of the formulation components, a complex analysis for which there is no simple solution. Thickener type and formation, base oil type and viscosity, and additive polarity all contribute to a grease's ability to lubricate

effectively if contaminated with water. No single test can predict lubrication performance in the presence of water. Several tests are often used to balance performance, touching on multiple conditions that could arise in grease applications. Further, these tests can be costly in terms of time and materials.

Against this background, the authors propose to employ a novel testing approach that uses the contact angle to characterize grease behavior in the presence of water. This technique leverages both the chemical and physical interaction with surface-active polar components within the grease structure to assess the behavior of grease in the presence of water. This new test requires a minimal grease sample (0.1–0.3 g) and can be performed in one minute. In this approach, a water droplet is dispensed on the surface of the grease and the contact angle of the water droplet is measured. Grease with a higher contact angle is water resistant or hydrophobic, while a lower contact angle is water absorbing, i.e., hydrophilic.

Considering seven commercially available greases, Lijesh et al. [14] categorized various greases by their water resistance and absorbing properties based on the contact angle results. In the present work, the authors extend on previous work [7,14] toward developing a standard for categorizing the water-resistant properties of grease by utilizing the contact angle approach. This work tests twelve greases on a custom-designed contact angle setup for grease and proposes a standard procedure. The standard will benefit both the grease manufacturer—in reducing the time and cost of quality control (QC) testing of the previous standardized tests—and the field user by providing a portable test that can be used to assess grease using a very small sample. Finally, the findings from the proposed novel standard is correlated with the following existing ASTM standards: (i) the water spray-off test as per ASTM D4049, and (ii) the water washout test performed at 79 °C, as per ASTM D1264 standard.

The outline of this paper is as follows. The details of the methodology and experimental setup employed are provided in Section 2. The results of contact angle values for the twelve greases are presented in Section 3, followed by a discussion of the results and proposed development of the standard in Section 4. To conclude, a summary and concluding remarks are presented in Section 5.

2. Materials and Methods

The complex and semi-solid nature of grease and the dependence of the contact angle on the surface topology of the test material necessitated developing a custom-designed contact-angle instrument and establishing standard operating procedures for the assessment of the water resistance properties of grease (Figure 1a). The instrument consists of the following: (i) a display to show the captured images and results, a microprocessor to control the camera, a micro-pump, screen, and monochromatic light (see Figure 1b), (ii) a camera with a microscopic lens to capture the video of the water droplet, a micropump capable of dispensing a water droplet of 10 μL , and the water reservoir (see Figure 1c), and (iii) a grease holder to provide a uniform thickness of the grease during every test (see Figure 1c). The camera employed in the present setup is a 12 MP with a 10 MP telephoto lens. The designed grease holder and micro-pump provide consistent and fast results. We observed the water droplet reaching equilibrium within 10 s.

Earlier studies assessing grease performance in the presence of water [16,27] faced difficulties related to consistently dispensing the same volume of water at the same location on the surface of the grease sample and achieving a uniform thickness of grease for every test. For this reason, in the present work, the setup was designed with a micro pump to dispense 10 μL water droplets and a grease holder capable of producing the same grease sample thickness during every test. The developed grease holder helps achieve a consistent grease topology during testing. In this developed instrument, a water droplet is dispensed onto the grease, and the contact angle between the droplet and grease is quickly measured using the developed software.

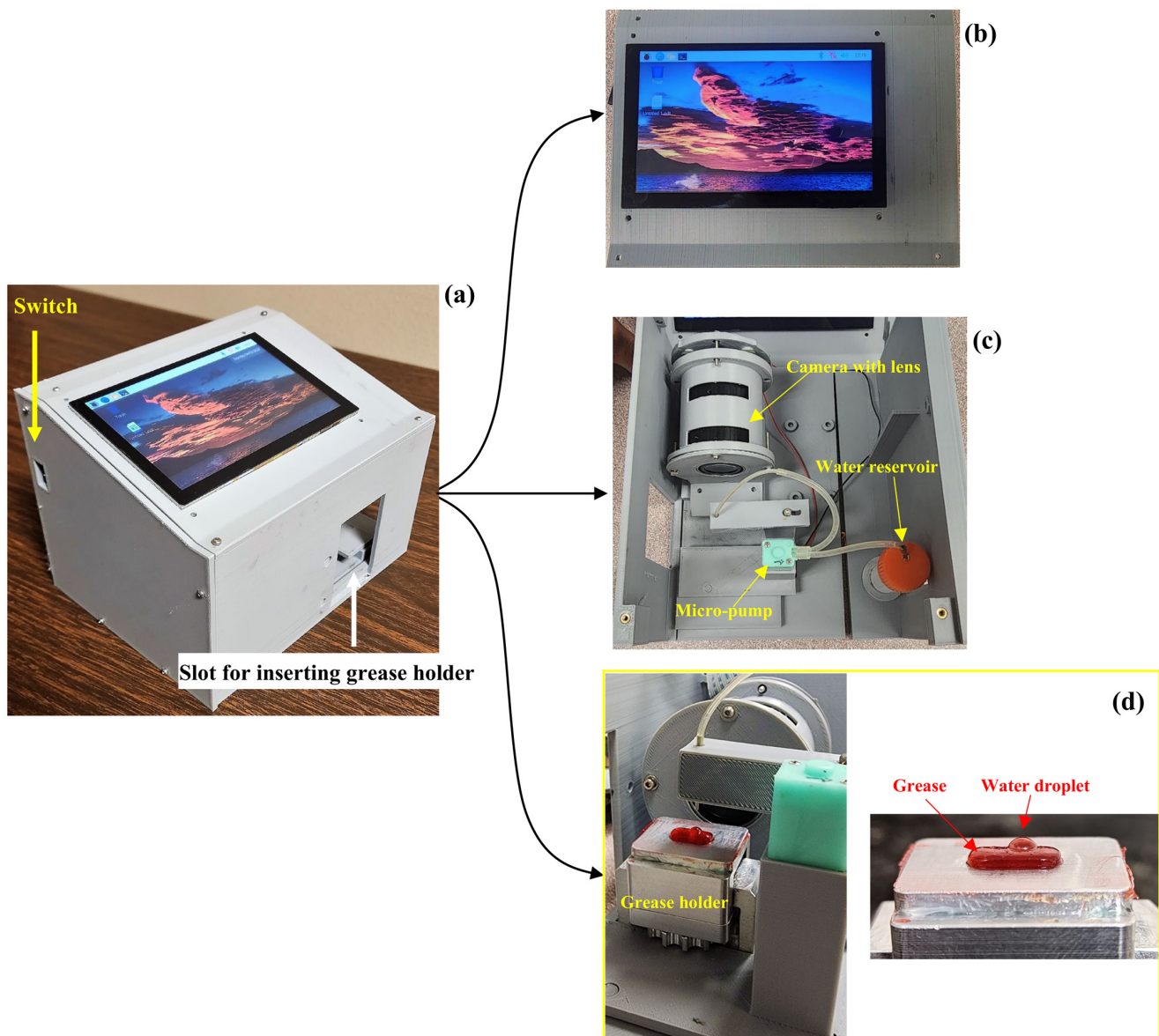


Figure 1. Custom-designed contact angle setup with their components. (a) isometric view of the contact angle setup, (b) display and microcontroller, (c) top view of the setup showing the camera and lens, micropump, and water reservoir, and (d) grease holder with grease and water droplet.

The steps followed to achieve repeatable results are described below and shown in Figure 2.

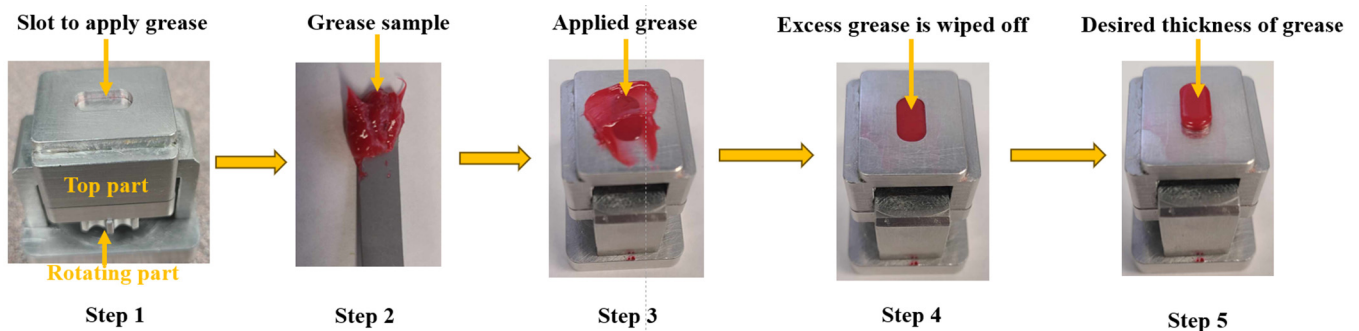


Figure 2. Steps for achieving a uniform thickness of grease in the grease holder.

Step 1: The top of the grease holder is moved up by rotating the rotating part, creating a slot in the center to apply the grease.

Step 2: 0.1–0.3 gms grease is taken for testing.

Step 3: The grease sample is filled in the slot.

Step 4: The excess grease is wiped off.

Step 5: The top part is moved down, creating a projection of grease to be tested.

Figure 3a shows an image capture from the video at 30 frames. The captured image is converted to greyscale (see Figure 3b); from the greyscale image, the edge points of the water droplet are identified (see Figure 3c). The slopes between the points are identified from the edge points, and the points with maximum slope values are determined and used as reference contact points. The contact angle is the angle between a linear fit line from the contact points and the data points of the water droplet. Finally, the calculation is made and displayed (see Figure 3d). All the above-mentioned image-processing techniques are performed using the Python platform.

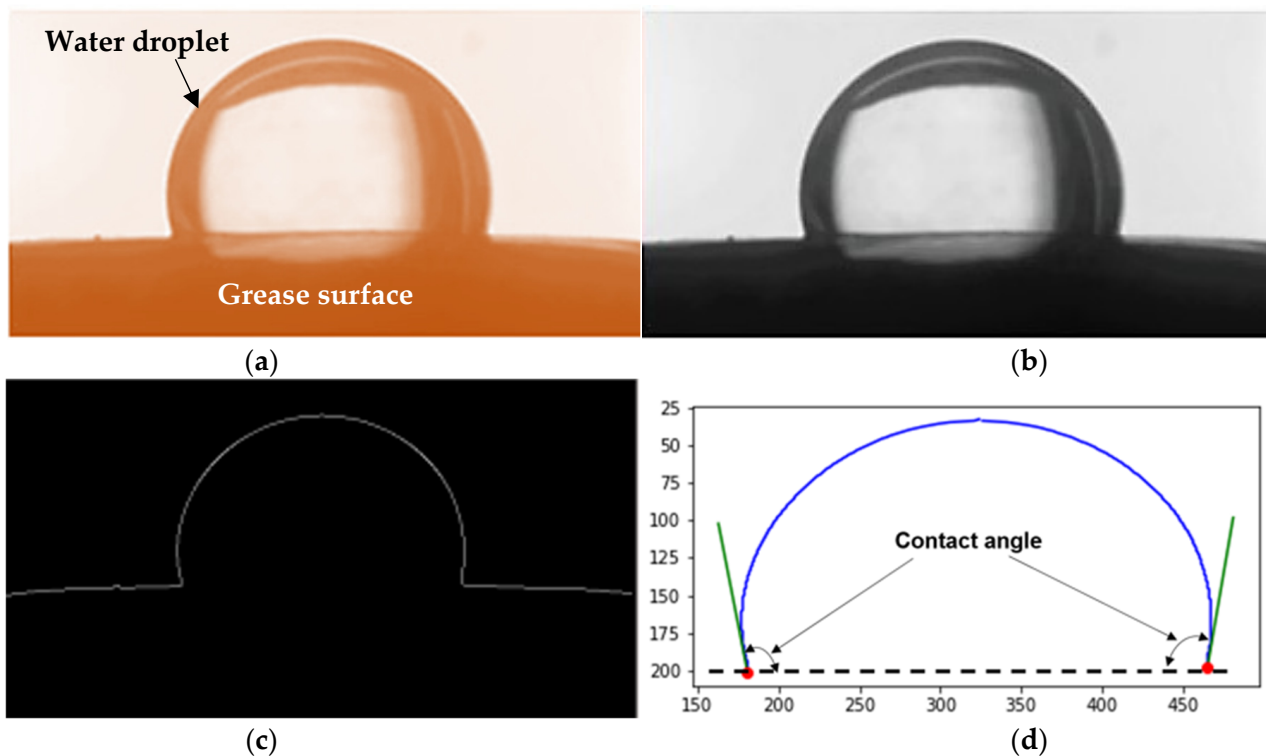


Figure 3. Image processing of the water droplet to determine the contact angle. (a) image at 300 frames, (b) greyscale Image at 300 frames, (c) edge points of a water droplet, (d) shape of droplet for analysis.

Figure 4 shows the graphical user interface developed in the Python program for recording the video of the water droplet followed by the outputs yielded by the above-mentioned image processing technique.

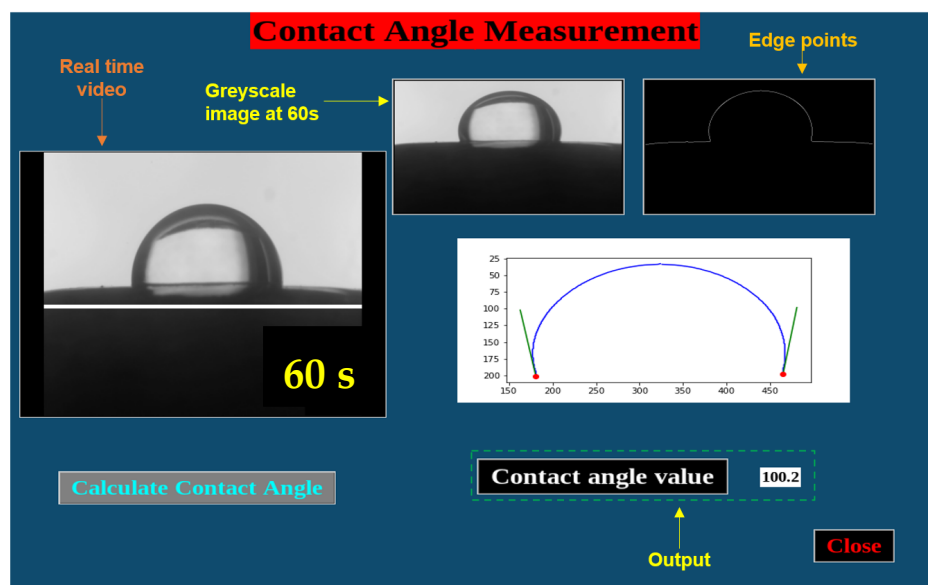


Figure 4. Graphical user interface developed in Python for measuring the contact angle.

3. Results

Sixteen commercially available greases were examined to test the proposed standard for measuring a grease’s water resistance properties. The greases considered are of six different NLGI grades (00 to 3), six types of thickeners (aluminum complex (AlC), calcium sulphonate (CaS), lithium (Li), lithium complex (LiC), poly urea (PU), and silicon (Si)), three types of base oil types (bio-based oil, mineral and synthetic), and three different viscosities (100, 220 and 460 cSt). The contact angles of each grease sample were assessed for three trials on fresh grease samples. The data for all sixteen greases along with their different compositions, are provided in Table 1.

Table 1. Contact angle values obtained for sixteen greases.

Grease Type	NLGI Grades	Base Oil Type	Grease Thickeners	Base Oil Viscosity @ 40 °C cSt	Contact Angle (°)		
					Trial 1	Trial 2	Trial 3
1	3	Mineral	LiC	220	88	88.4	87.9
2	2	Bio-based oil	Li	220	58.2	59.2	59.6
3	2	Mineral	AlC	226	86.8	87.4	87.2
4	2	Mineral	CaS	460	89.4	89.6	89.2
5	2	Mineral	LiC	220	83.4	85.4	84
6	2	Mineral	LiC	460	83	84.4	84
7	2	Mineral	Poly Urea	220	71.2	70.2	70.8
8	2	Synthetic	LiC	100	89.2	88.4	88.8
9	2	Synthetic	LiC	220	100.8	101.2	101.4
10	2	Synthetic	Poly Urea	220	84.4	86.6	86.8
11	2	Synthetic	Silicone	220	89.6	90.4	90.1
12	1.5	Synthetic	LiC	460	94.2	93.8	93.8
13	1.5	Synthetic	LiC	460	88.4	88.8	87.9
14	1	Mineral	LiC	220	77.8	77.2	77.4
15	0	Mineral	LiC	220	72.5	72.1	72.5
16	00	Mineral	LiC	220	67.8	68.4	68.4

The mean values of the three-sample data set with errors are plotted in Figure 5a. This figure shows that the contact angle values for the sixteen greases varied from >60° to <100° with a maximum error of ~2° for Grease type 9. Further, the standard deviation calculated considering trial readings is plotted in Figure 5b. The maximum mean value of the standard deviation is 0.86°, observed for Grease 5.

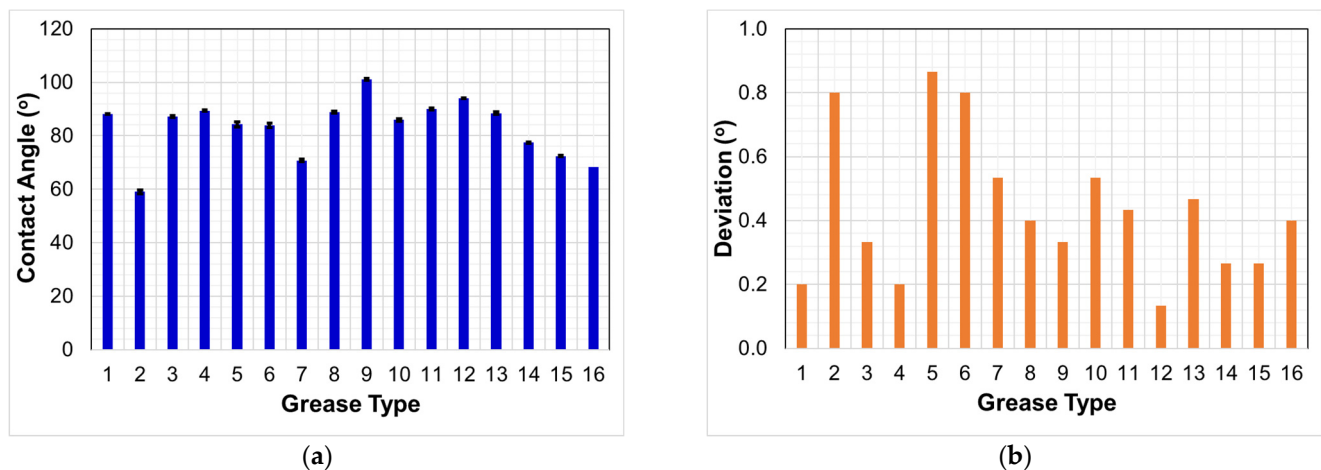


Figure 5. Average contact angle values determined from different trials and standard deviation (a) average contact angle values with error, and (b) mean deviation of the contact angle values between the trials.

4. Discussion

In the present paper, we attempt to examine the water resistance properties of greases using a contact angle approach [16]. In this approach, a water droplet is dispensed onto the grease surface and the contact angle is measured. The technique employed leverages the chemistry behind the interaction between surface-active polar components on the grease surface and the dispensed water droplet. This approach is developed considering the behavior of water on the grease surface which is strongly dependent on the availability of polar components and the arrangement of surface-active thickeners and additives in the grease [11].

Tests were performed on sixteen grease types during three trials and each test was performed on fresh grease samples. The contact angle values are provided in Table 1. It can be inferred from the table that the developed setup, grease holder, and proposed procedure provided a repeatable contact angle value. The obtained average values of the contact angle are plotted in Figure 5a. Comparing Table 1 and Figure 5a, the following observations were made:

- The highest contact angle ($\sim 101^\circ$) indicating higher water resistance properties of the grease is observed for Grease type 9, with Lithium complex as a thickener, NLGI grade 2, and synthetic oil as a base oil with 220 cSt.
- The lowest contact angle is observed for Grease type 2, with Lithium as a thickener, NLGI grade 2, and bio-based oil as a base oil with 220 cSt. This indicates that the proper selection of grease thickener and base oil is necessary for achieving good water resistance properties. Thus, grease thickener and base oil type should be considered for improved water resistance properties.
- Comparing the ISO 460 mineral oil greases with NLGI 2 consistencies, the contact angle value is observed to be the highest for the CaS thickener. It is well known that grease with CaS as a thickener provides the best performance in the presence of water [8].
- Comparing grease types 1, 5, 14, 15, and 16, the contact angle values are observed to increase with a concurrent increase in the NLGI grease grades (see Figure 6).

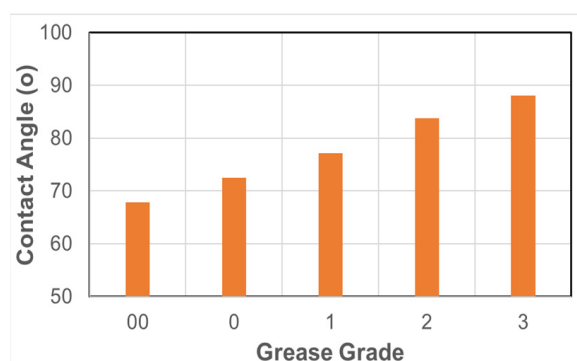


Figure 6. Contact angle value of different grades of LiC grease with mineral oil as the base oil with 220 cSt.

- Comparing grease types 5 & 9 and 7 & 10, the contact angle values are observed to be high for grease with synthetic oil as the base oil. Comparing grease types 5 & 9 and 7 & 10, the contact angle values are observed to be high for grease with synthetic oil as the base oil. Synthetic base oils are generally susceptible to hydrolysis compared to mineral oil, i.e., synthetic base oils remain stable in damp environments as they do not emulsify when exposed to water [10,28]. Further, the saturation of water content in mineral oil is often about 200–300 ppm moisture, while for synthetic oil it can be close to 1000 ppm [10].
- Grease types 12 and 13 had the same grease composition but were from different companies. The difference in the contact angle values indicates that the chemistry followed for developing a grease, results in different water resistance properties of that grease.

Having established consistent results, an attempt is made to develop a novel standard that can be used as a guideline by industry and testing laboratories in determining the water resistance properties of grease.

4.1. Development of the Standard

In the present work, the authors propose classifying the contact angle values into five different grades based on the mean contact angle results. The grades range from 1 (poor water resistance properties) to 5 (excellent water resistance properties). This is summarized in Table 2. According to the proposed classifications, Grade 1 grease (contact angle $< 60^\circ$) exhibits poor water resistance properties, while grease with Grade 5 (contact angle $> 90^\circ$) exhibits good water resistance properties.

Table 2. Contact angle corresponding to different grades proposed for water-resistance properties of grease.

Grades	Contact Angle ($^\circ$)	Water-Resistance Characterization
1	$< 60^\circ$	Very Poor
2	$60\text{--}70^\circ$	Poor
3	$70\text{--}80^\circ$	Average
4	$80\text{--}90^\circ$	Good
5	$> 90^\circ$	Excellent

Following the proposed procedure for grading the grease, the sixteen greases are characterized into five different grades according to the mean contact angle results, and the grades are provided in Table 3. From this table, it is observed that grease types 2 and 15 fall into a Grade 1 classification (poor water resistance). Four of the greases (9, 11 & 12) fell into Grade 5, reflecting excellent water resistance.

Table 3. Mean contact angle values and corresponding grades.

Grease Type	NLGI Grades	Base Oil Type	Grease Thickeners	Base Oil Viscosity @ 40 °C cSt	Average Contact Angle Values (°)	Proposed Grades
1	3	Mineral	LiC	220	88.1	4
2	2	Bio-based oil	Li	220	59	1
3	2	Mineral	AlC	226	87.13	4
4	2	Mineral	CaS	460	89.4	4
5	2	Mineral	LiC	220	84.27	4
6	2	Mineral	LiC	460	83.8	4
7	2	Mineral	Poly Urea	220	70.73	3
8	2	Synthetic	LiC	100	88.8	4
9	2	Synthetic	LiC	220	101.33	5
10	2	Synthetic	Poly Urea	220	85.93	4
11	2	Synthetic	Silicone	220	90.03	5
12	1.5	Synthetic	LiC	460	93.93	5
13	1.5	Synthetic	LiC	460	88.37	4
14	1	Mineral	LiC	220	77.47	3
15	00	Mineral	AlC	244	55.17	1
16	00	Mineral	LiC	220	68.2	2

4.2. ASTM Water Resistance Standard (ASTM D4049 and ASTM D1264)

The existing ASTM standards for the evaluation of grease in the presence of water are ASTM D4049 and ASTM D1264. These standards provide information regarding the adherence properties of the grease to the bearing surface. They do not provide information on the water-repelling or attractive properties of the grease. On the other hand, the contact angle approach provides a quantitative way of characterizing the water-repelling characteristics of the grease. Furthermore, for a practitioner, adapting the proposed standard derived through assessing contact angle values will be faster if the contact angle values are correlated to the existing ASTM D4049 and ASTM D1264 standards. For this purpose, the measured contact angle values are compared with the water spray-off test, as per ASTM D4049, and the water washout test performed at 79 °C, as per the ASTM D1264 standard. Both these standards provide the result in percentage of weight loss. The average contact, the weight % loss of grease during water spray-off, and water washout tests from the website of the commercial greases are provided in Table 4. The values are marked NA (not available) for greases with details not provided on their company websites.

Table 4. Average contact angle values and their respective weight % loss of grease during water spray-off and water washout tests as reported by company websites.

Grease Type	Average Contact Angle Values (°)	Proposed Grades	Water Washout, Loss wt%,	Water Spray Off, Loss wt%,
1	88.1	4	5	10
2	59	1	NA	NA
3	87.13	4	5.78	NA
4	89.4	4	NA	NA
5	84.27	4	5	10
6	83.8	4	NA	NA
7	70.73	3	1.9	NA
8	88.8	4	6	NA
9	101.33	5	1.5	NA
10	85.93	4	7	26
11	90.03	5	<1	NA
12	93.93	5	<3	6.5
13	88.37	4	7	NA
14	77.47	3	8	15
15	55.17	1	NA	NA
16	68.2	2	37	NA

Conclusions from Table 4

Grade 5: The proposed standard identified Grease types 9, 11, and 12 as Grade 5, with excellent water resistance properties. The water washout loss weight percentages for these greases are observed to have the lowest magnitude of <3 and the percentage loss of weight during water spray-off for Grease type 12 is 6.5. In other words, the greases identified as having excellent water resistance properties from the proposed standard also provided the best performance in the presence of water as per ASTM D4049 and ASTM D1264.

Grade 4: For Grease types 1, 3, 5, 8, 10, and 13, the observed weight loss during the water washout test is reported to be in the range of 5 to 7%, while the weight loss during water spray-off for Grease types 1, 5, and 13 is observed to be in the range of 10 and 26. These greases have good water washout properties but are lower than Grease types 9, 11, and 12. According to the proposed standard, these greases are classified as one grade lower than Grade 5, i.e., Grade 4 grease, which is proposed to have good water resistance properties.

Grade 3: Grease type 14 had water spray off and a water washout weight loss percentage of 8 and 15, respectively. The water spray-off test showed a lower water washout property than the earlier considered greases; however, the water washout test showed a better value than grease type 10. The proposed standard identifies this grease as a Grade 3 grease with average water resistance properties. For Grease type 7 (poly-urea (PU) based grease), a contradictory observation between the proposed standard and ASTM D4049 is seen. Cyriac et al. [2] observed that poly-urea-based grease with different base oils absorbed 70–80% of water, proving that the poly-urea as the thickener is the reason for the high percentage of water absorption. In a similar test, PU-based grease was observed to absorb more water than CaS-based and LiC-based greases [14]. The higher absorption property of poly-urea thickener resulted in a lower contact angle value. However, this grease also has good adherence properties and is known to have lower weight loss during the water washout test.

Grade 2: Among the considered 16 greases, grease type 16 is reported to have the highest weight loss percentage during the water washout test and the proposed approach graded the grease as Grade 2, which is considered to have poor water-resistant properties. The low water resistance is due to the grease's lower grade (NLGI Grade 00).

Grade 1: Grease types 2 and 15 are identified as Grade 1 greases, with very poor water resistance properties. Unfortunately, the relevant website did not provide water washout or spray-off results for these greases.

Finally, it can be concluded that, for most of the tested greases in this study, the identified water resistance properties of the greases using the proposed standard (Table 2) agree with the results obtained from ASTM D4049 and ASTM D1264 standards. This can be attributed to the fact that grease with higher water-repelling properties is unreactive to water and tends to stick firmly to the bearing surface, while poor water-repelling grease absorbs more water, reacts with water, and loses its adherence properties. It should be noted, however, that some greases, such as polyurea-based greases, behave differently. The probable weight loss percentage range for water spray-off and washout test for the identified different Grades of water-repellent greases using the proposed approach is provided in Table 5.

Table 5. Contact angle corresponding to different grades proposed for the water-resistance properties of grease.

Grades	Contact Angle (°)	Water Spray Off, Loss wt%,	Water Washout, Loss wt%,
1	<60°	-	-
2	60–70°	>37	-
3	70–80°	>7 & <36	>26
4	80–90°	>3 & <7	>6.5 & <26
5	>90°	<3	<6.5

5. Conclusions

The authors have presented an alternative test method to measure a grease lubricant's water resistance properties. In this test method, a water droplet is dispensed onto the grease surface and the contact angle of the water droplet is measured. This approach is unique, with the benefits of using smaller sample sizes, shorter test time, and a reduced test grease quantity. Results show the test method and apparatus yield repeatable results. Based on the results, the authors were able to propose a standard to classify grease based on water resistance properties. The water resistance properties identified using the proposed standard were in accordance with the results reported by ASTM D4049 and ASTM D1264 standards. Furthermore, the proposed standard, using contact angle values, was able to determine the water resistance properties of greases, which were not measurable using the existing standards.

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