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Improving The Tribological Characteristics of In-House Made Acid-Activated Iraqi Bentonite Lubricating Greases Using Graphite and Polytetrafluoroethylene Nanoplatelets as Additives

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ABSTRACT

Traditional soap-based greases such as lithium and calcium are no longer able to meet the requirements of high-performance lubrication due to their high cost and environmentally harmful nature, thus there is a need for new solid, non-organic, non-soapy, and inexpensive greases. This paper presents a comparative study and analysis of the effect of adding polytetrafluoroethylene (PTFE) and graphite to Iraqi Bentonite Lubricating Greases Activated with Nitric Acid (AIBLG-HNO3) of which 24-types were synthesized in laboratory using two types of local base oil stock 60 and 150, Iraqi bentonite as thickener, and two types of soft and hard paraffin waxes. The tribological characteristics such as corrosion resistance and diameter of wear scars (WSDs) that take place due to friction between metal surfaces inside the equipment were studied. It was concluded that adding either PTFE or graphite nanocomposites should be preferably at a weight percentage of not less than 15 wt.%. Moreover, in comparison with the parent Iraqi Bentonite Lubricating Greases (IBLG) samples free of additives, the results also indicate a decrease in WSDs by 90% to 93% using base oil 150 for four samples and in the presence of either soft and/or hard wax. As a general rule, using base oil 150 in Iraqi lubricating greases gives better specifications to the manufactured greases than using base oil 60. Accordingly, this study can open a new approach that contributes to the utilization of the abundant and cheap Iraqi bentonite clay as new thickeners capable of meeting the needs of the local industry.

1. Introduction

Lubricating greases are complex multiphase systems composed of a base oil, often a mineral or synthetic oil, which is trapped within a solid network created by a thickening agent. The formulation and manufacturing process of lubricating greases are important factors that influence their rheological and tribological characteristics. It possesses an extensive range of utilizes, including numerous types and brands, and is predominantly employed for bearings and gear transmissions. Recently, several researchers have provided summaries of advancements in machine learning-based studies on friction and wear [1-4], on the other hand, additional researchers have made valuable efforts in various domains including dry friction, elastohydrodynamic lubrication, grease, and lubricating oil to develop lubricants that are both high-performing and cost-effective, while also ensuring long-term efficiency and longevity of equipment [5-14]. These investigations provided a range of

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samples for tribological research on this subject and significantly advanced the progress of tribology. Due to the wide range of greases available and their many applications, each kind has unique performance and quality indicators [15-17]. Hence, it is essential to choose an appropriate grease for each specific piece of equipment. Neglecting to do so can lead to mechanical failures and serious consequences such as fatal accidents [18]. Traditional soap greases, such as lithium or calcium-based grease, have poor heat resistance and are therefore used for general plain bearings that operate at fairly low speed and low load where the temperature does not rise above 70°C during service, whereas non-soap grease thickened with bentonite clay is thermally stable, insensitive to water (i.e., has high oxidative stability), corrosion resistance, and able to withstand a wider range of temperature, which is consequently called "no-drop point grease" or "no-melting point grease" because it does not lose its structure even at very high temperatures and continues to work up to +260°C [19-22].

In fact, Nano clays are regarded as eco-friendly substances and have the potential to be utilized as very efficient thickening agents [23]. Montmorillonite is a particularly interesting material among many forms of clays, primarily because it is both cost-effective and readily available for numerous engineering purposes [24]. Montmorillonite is formed through inorganic layered silicates (octahedral aluminum oxide layer and tetrahedral silicon oxide layer). Several of these layered platelets are arranged into particles. The macro-properties of the substance are determined by these layered silicates. Non-modified montmorillonite is hydrophilic, however, the replacement of ions present among the layers by organic cations might enhance the compatibility of clay with a wide variety of hydrophobic substances. Because of its small size, it interacts with matter on the atomic or molecular scale [25].

Tribology research is mainly focused on the investigation of friction, wear scars, and lubrication occurring between surfaces that contact with each other. Due to the continuous industrialization of human society, the scope of tribology research has expanded significantly [26]. A significant percentage of fuel energy is consumed in overcoming mechanical losses caused by friction. This is observed in various applications such as paper factories, passenger automobiles, trucks, and buses. According to a detailed study by Holmberg et al. [27-29], leading to a notable surge in CO_2 emissions [30-31].

Greases provide viable options for lubricant formulators when conventional liquid materials are insufficient in terms of performance, particularly in high-temperature environments and low sliding speeds with significant contact stresses on machinery's bearing points [32-35]. Several types of solid lubricants have been identified as potential lubricant additives in practical applications [36-38]. Due to the progress of technology in the industry, mechanical equipment will always operate in highly challenging environments and working conditions, including extreme temperatures, sandstorms, salt spray, acid or alkali atmospheres, fast or low speeds, and heavy loads. The resultant friction, abrasion, and corrosion have detrimental impacts on the efficiency and lifespan of equipment [39-41]. Hence, it is imperative to implement efficient safeguards in order to enhance the unfavorable conditions for the operation of mechanical machinery. Grease is a viscous lubricant that has the properties of lubrication and sealing. The inherent consistency and adhesiveness of this substance differentiate it from both liquid and solid lubricants. Additionally, it may be applied to friction pairs without any leakage caused by gravity. Therefore, it has special advantages as a material that is resistant to corrosion [42-43]. The most important kind of additives are corrosion inhibitors and plant extracts can be utilized as eco-friendly corrosion inhibitors on surfaces [44-46].

This research aims to study the effect of adding some additives on the properties of four different types of laboratory-prepared environmentally friendly inorganic greases to make them more suitable according to their standard specifications for use in industrial applications for equipment operating under high stresses, which exposes their metals to friction and corrosion, by adding graphite and polytetrafluoroethylene (PTFE) as improved additives. Different weight percentages of additives have been added to the greases to obtain the most effective ratio that enhances grease tribological characteristics such as anti-wear and friction resistance. To diminish the production cost, raw Iraqi bentonite clay was used as a thickener after treating it with an acidic solution. Two types of base oils produced in Iraqi refineries and two types of locally made paraffin wax were also used. This study will open the horizon for developing and using the abundantly available and inexpensive Iraqi bentonite clays as new thickeners in the manufacture of high-efficiency greases in the near future to cover the industrial need for these important products.

2. Methodology

2.1 Material

Base oil (Stock 60 and Stock 150) and primary additives (i.e., Soft and Hard Waxes) were purchased from the Middle Refineries Company, Al Doura Refineries in Iraq for the preparation of greases. The raw Iraqi bentonite (RIB) material used in this study was provided by the State Company for Geological Survey and Exploration in Iraq, which consists of about 80% montmorillonite clay ($(OH)_{12}\cdot Al_4\cdot Si_8\cdot O_{16}\cdot nH_2O$) of the Fe-Mg-Ca type. Iraqi Bentonite (IB) used as a grease thickener was treated in the laboratory under the name of nitric acid-activated Iraqi bentonite ($AIBHNO_3$) and has a specific surface area of 195.783 m^2/g, average particle size of 29.03 nm, and degree of crystallinity of 128.62% according to the analyses conducted at the University of Technology, Baghdad, Iraq.

The following materials were also used: (i) Nitric acid (HNO_3) with 69% purity and 122 ˚C BP, (ii) Methanol (CH_3OH) with 99.8% purity and 64.7 ˚C BP both purchased from LOBA CHEMIE Co., Ltd- India and (iii) DI Water (H_2O) with 99.99% purity and 99.97 ˚C BP supplied from University of Technology-Iraq. Other additives (i.e., graphite and polytetrafluoroethylene nano-platelets) usage in the preparation of grease were provided by Central Drug House (CDH) Co., Ltd., and Sichuan Fudi New Energy Co., Ltd., and were used directly without further purification. The physical properties of Iraqi base oil stock are shown in Table 1, the physical characteristics of home-made paraffin wax by Midland Refineries Company MRC / Daura Refineries are shown in Table 2, the characteristics of graphite nanoplatelets are shown in Table 3, and the characteristics of the PTFE nanoplatelets are shown Table 4.

Table 1: Physical properties of Iraqi base oil stock.

Physical property	Base Oil Stock 60	Base Oil Stock 150
Viscosity at 40 ˚C, c.St.	61.09	318.58
Viscosity at 100 ˚C, c.St.	8.05	25.12
[a] Viscosity index (V. I.)	95 Min.	93 Min.
[b] COC flash point, ˚C	220 Min.	260 Min.
Pour point, ˚C	-6 Max.	-3 Max.
[c] Color	1..5	3
Water content %vol.	NiL	NiL

[a] An index used to describe the viscosity-temperature behavior of lubricating oils.
[b] COC: Cleveland open cup method.
[c] The color of oil is measured by Lovibond apparatus according to ASTM D 1500 Color Scale.

Table 2: Physical characteristics of homemade paraffin wax by Midland Refineries Company MRC / Daura Refineries.

Physical Characteristics	Soft wax	Hard wax
Penetration at 25 ˚C	34	20
API gravity	37.3	34
Melting point, ˚C	48	60 – 66
Oil content, wt.%	1.5	2
Color by ASTM D-1500	2.5	3

Table 3: Characteristics of graphite nanoplatelets.

Characteristics	Specification
Formula	C
Color	Black
Specific gravity, g/cm^3	2.26
Crystalline structure	Hexagonal
Melting point, °C	>3500

Table 4: Characteristics of the PTFE nanoplatelets.

Characteristics	Specification
Color	white powder
Specific gravity, g/cm^3	0.6
Particle size, nm	8
Surface area, m^2/g	10
Whiteness	≥98
Melting point, ˚C	327

2.2 Preparation of Iraqi Bentonite Lubricating grease (IBLG)

Activated Iraqi bentonite was utilized as a thickening in producing lubricating greases. 2 M nitric acid (HNO_3) solution was used to activate the raw Iraqi bentonite (RIB) at 100 °C and mixed continuously for 8 h. In fact, acid-activated bentonite clays have different physical and chemical properties than unactivated clays. Acid activation usually modifies the surface area of the bentonite and changes the percentages of its components. Intuitively, the acid treatment removes a percentage of the aluminum layers to which the cation is bound. By performing energy dispersive X-ray spectroscopy (EDS) using OXFORD INSTRUMENT PLC, UK, it was found that the percentage of aluminium within the bentonite framework decreased from 14.2 wt. % in RIB to about 6.1 wt. % in $AIBHNO_3$, where the H^+ ion replaces the metal ions bound to the removed aluminium atoms, creating a clay with a highly active structural framework that helps the organic base oil to bind to the protons to form lubricating greases. Base oil stock (BOS) 60 and 150 were also used as dispersing fluid. In addition, both soft and hard waxes were used as additives to prevent the separation that happens between the thickener and the base oil. In other words, these waxes were employed to enhance the stability, solidity, and consistency of the produced grease. Methanol alcohol is utilized as a polar activator to effectively control the dispersion of the thickening in the base oil, ensuring a uniform mixture. Figure 1 illustrates in detail preparation process of Iraqi bentonite lubricating grease. In general, Iraqi bentonite lubricating grease was produced by combining the base oil and a dispersing aid (i.e., 1 wt. % methanol), in a stirred vessel at a temperature of 25 ˚C. The mixture was continuously mixed at a speed of 2000 rpm for thirty minutes at a temperature of 40 ˚C to achieve a uniform mix without any air bubbles. The mixer used is a 2020 RZR mechanical overhead stirrer made in Germany by Heidolph Instruments GmbH & Co. KG suitable for standard applications and has medium to high viscosity with a speed range between 40-2000 rpm, a torque range between 100-400 Ncm, a capacity of 25 liters, and a four-impeller pitched-blade type. Likewise, the nitric acid activated Iraqi bentonite ($AIBHNO_3$) slowly added to the base oil (i.e., 62% base oil and 27% thickener) in the vessel under continuous stirring at 2000 rpm for 1 h. Afterwards, a wax was added to the mixture of thickener and base oil at 10 wt. %. The mixture was then continuously stirred for thirty minutes. In order to enhance the consistency of the Iraqi bentonite lubricating grease. Graphite and polytetrafluoroethylene (PTFE) were then added to base oil, AIB, and wax with three different weight ratios (i.e., 5, 10, and 15 wt. %) under continuous mixing for 30 min at a temperature of 40 ˚C. This process aims to enhance the grease's resistance to water washout and protection against corrosion of metallic materials, reduce friction noise, and improve the load-carrying capacity properties of the grease. Finally, the resulting greases (i.e., IBLG 1 and IBLG 2), as given in Table 5, were left to cool slowly to room temperature to obtain 28 types of laboratory-manufactured Iraqi greases, as exhibited in detail in Figure 2.

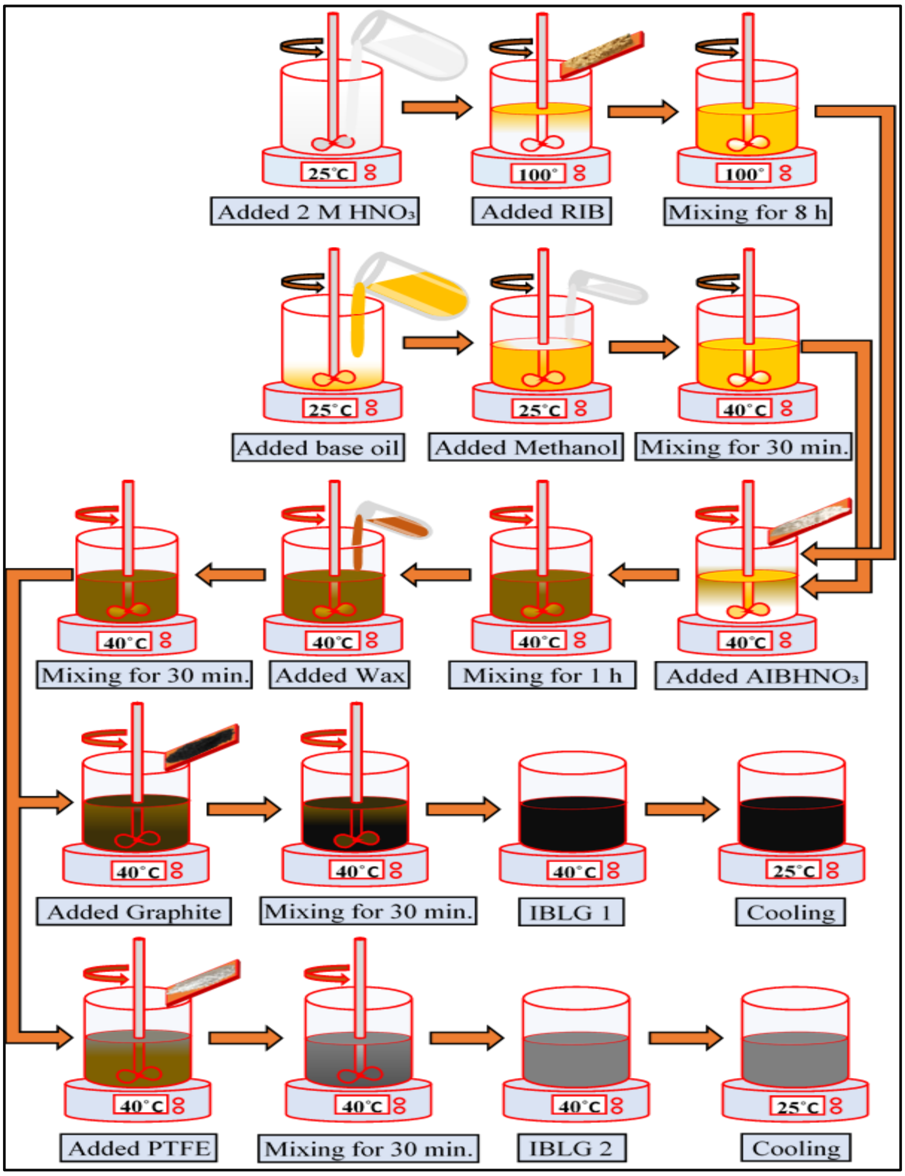

Figure 1. A schematic process for improving the characteristics of Iraqi bentonite lubricating grease (IBLG) prepared from activated Iraqi bentonite.

Table 5: Identifying the names of laboratory-prepared samples of Iraqi bentonite lubricating grease (IBLG) according to the identification code for each one.

Code No.	Identification	Code No.	Identification
0000	BOS 60, $AIBHNO_3$, and soft wax.	11	BOS 150, $AIBHNO_3$, soft wax, and 10% PTFE.
000	BOS 150, $AIBHNO_3$, and soft wax.	12	BOS 150, $AIBHNO_3$, soft wax, and 15% PTFE.
00	BOS 60, $AIBHNO_3$, and hard wax.	13	BOS 60, $AIBHNO_3$, hard wax, and 5% graphite.
0	BOS 150, $AIBHNO_3$, and hard wax.	14	BOS 60, $AIBHNO_3$, hard wax, and 10% graphite.
1	BOS 60, $AIBHNO_3$, hard wax, and 5% PTFE.	15	BOS 60, $AIBHNO_3$, hard wax, and 15% graphite.
2	BOS 60, $AIBHNO_3$, hard wax, and 10% PTFE.	16	BOS 150, $AIBHNO_3$, hard wax, and 5% graphite.
3	BOS 60, $AIBHNO_3$, hard wax, and 15% PTFE.	17	BOS 150, $AIBHNO_3$, hard wax, and 10% graphite.
4	BOS 150, $AIBHNO_3$, hard wax, and 5% PTFE.	18	BOS 150, $AIBHNO_3$, hard wax, and 15% graphite.
5	BOS 150, $AIBHNO_3$, hard wax, and 10% PTFE.	19	BOS 60, $AIBHNO_3$, soft wax, and 5% graphite.
6	BOS 150, $AIBHNO_3$, hard wax, and 15% PTFE.	20	BOS 60, $AIBHNO_3$, soft wax, and 10% graphite.
7	BOS 60, $AIBHNO_3$, soft wax, and 5% PTFE.	21	BOS 60, $AIBHNO_3$, soft wax, and 15% graphite.
8	BOS 60, $AIBHNO_3$, soft wax, and 10% PTFE.	22	BOS 150, $AIBHNO_3$, soft wax, and 5% graphite.
9	BOS 60, $AIBHNO_3$, soft wax, and 15 % PTFE.	23	BOS 150, $AIBHNO_3$, soft wax, and 10% graphite.
10	BOS 150, $AIBHNO_3$, soft wax, and 5% PTFE.	24	BOS 150, $AIBHNO_3$, soft wax, and 15% graphite.

Figure 2. Photographs of laboratory-prepared samples of Iraqi bentonite lubricating grease (IBLG) according to the identification code.

2.3 Characterization techniques

ASTM Copper Strip Corrosion Standard is used to evaluate the detection of copper corrosion in lubricating grease, according to the standards of Normal ab France Instrument Co., Ltd. ASTM Four-Ball Standard is used to evaluate the wear-preventative characteristics of lubricating grease, according to the requirements of Koehler Instrument Co., Inc.

2.3.1 Corrosion test

To detection of copper corrosion caused by lubricating grease, the ASTM D-4048 test technique was employed [47]. The copper strip is fully submerged in a lubricating grease sample and subjected to heating in an oil bath under controlled conditions (temperature maintained at 100 ˚C ± 1 ˚C for a duration of 24 hours ± 5 minutes). After the heated stage ends, the strip is taken out and cleaned using kerosene. It is then compared to the copper strip standard.

2.3.2 Four-ball anti-wear test

ASTM D2266 standard is utilized to evaluate the wear-preventative characteristics of lubricating grease [48]. Three of steel balls with a diameter of 12.7 mm are clamped together and covered with the greases that will be assessed. A fourth steel ball with a diameter of 12.7 mm, also known as the top ball, is pressed with a force of 392 N into the space formed by the three clamped balls to form three-point contact. The ambient temperature of the grease samples is controlled at 75°C, and then the top ball is rotated at a speed of 1200 rpm for 60 minutes. Greases are evaluated by measuring the mean diameter of the scars formed in the three bottom-clamped balls.

3. Results and discussion

3.1 Copper strip corrosion

The findings of the copper corrosion testing indicate that when immersed in Iraqi bentonite grease, copper strips show no color changes in comparison to the standard copper strip. Whether before or after adding all percentages of PTFE as an additive and for all four types of grease pre-prepared from Iraqi bentonite. These greases (i.e., From sample code 1 to sample code 12 in Table 5) gave a degree of copper corrosion that can be seen in 1a according to Figure 3 and Table 6 which explains the copper strip corrosion standard classifications, indicating that the laboratory prepared Iraqi bentonite greases have excellent corrosion resistance, as the addition of PTFE did not cause any corrosion, due to the generation of a lubrication film on the surface of the prepared grease to protect the metal surfaces that will be covered with the grease inside the equipment from any corrosion.

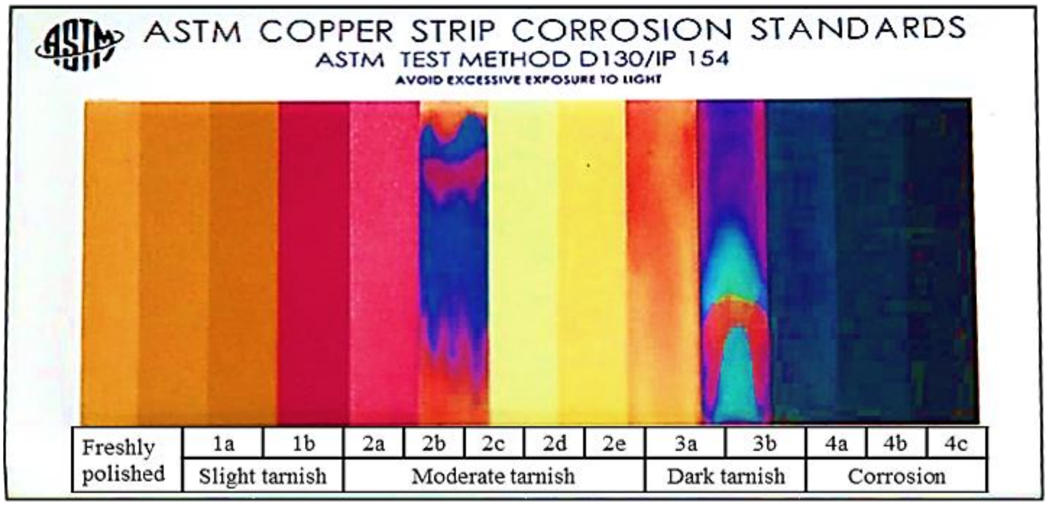

Figure 3: Copper Strip Corrosion Standards (CSCS) Test according to ASTM D-4048 method.

Table 6: The Copper Strip Corrosion Standard (CSCS) [a] Classifications.

Classification	Designation	Description
Freshly polished strip [b]		
1	Slight tarnish	A. Light orange B. Dark orange
2	Moderate tarnish	A. Claret red B. Lavender C. Multicolored with lavender, blue, silver, or both, overlaid on claret red. D. Silvery E. Brassy or gold
3	Dark tarnish	A. Magenta overcast on the brassy strip B. Multicolored with red and green showing, but no ray
4	Corrosion	A. Transparent black, dark gray, or brown with peacock green barely showing. B. Graphite or lusterless black C. Glossy or jet black

[a] The (CSCS) is a colored reproduction of strips characteristic of these descriptions.
[b] The freshly polished strip is included in the series only as an indication of the appearance of a properly polished strip before a test run; it is not possible to duplicate this appearance after a test, even with a completely noncorrosive sample.

However, the results showed that adding graphite to the greases prepared from Iraqi bentonite as an additive does not provide good corrosion resistance compared to PTFE, some grease samples (i.e., From sample code 13 to sample code 24 in Table 5) gave copper corrosion degrees of 1b as in samples identified by code 17 and 19, and 2c as in samples identified by code 14, 15, 16, 20 and 21. This indicates that the pre-prepared grease has poor corrosion resistance, or that the grease causes a slight type of corrosion on the metal surfaces that it will cover within the equipment in which it is used. In fact, PTFE has unparalleled characteristics, such as non-reactivity, hydrophobicity, low coefficient of friction, and excellent insulating properties [49]. Because of its enormous flexibility, PTFE significantly enhances compactness and adhesion [50]. Accordingly, the hydrophobic characteristics of PTFE are efficiently utilized to improve corrosion resistance [51] and water resistance [52], while graphite is a naturally hydrophilic inorganic material, that contains metal oxides [53]. These oxides can promote corrosion when used as thickeners in the manufacture of lubricating greases.

Moreover, Figure 4 shows illustrative images of the effect of additives on the corrosion tests of copper strips conducted on different types of laboratory-prepared bentonite greases, while Table 7 demonstrates the impact of additives on the corrosion of copper strips in various types of greases. The results obtained are consistent with those of previous research investigations published in the literature in terms of methodology in the analysis of solid-based lubricating greases [42, 54-56].

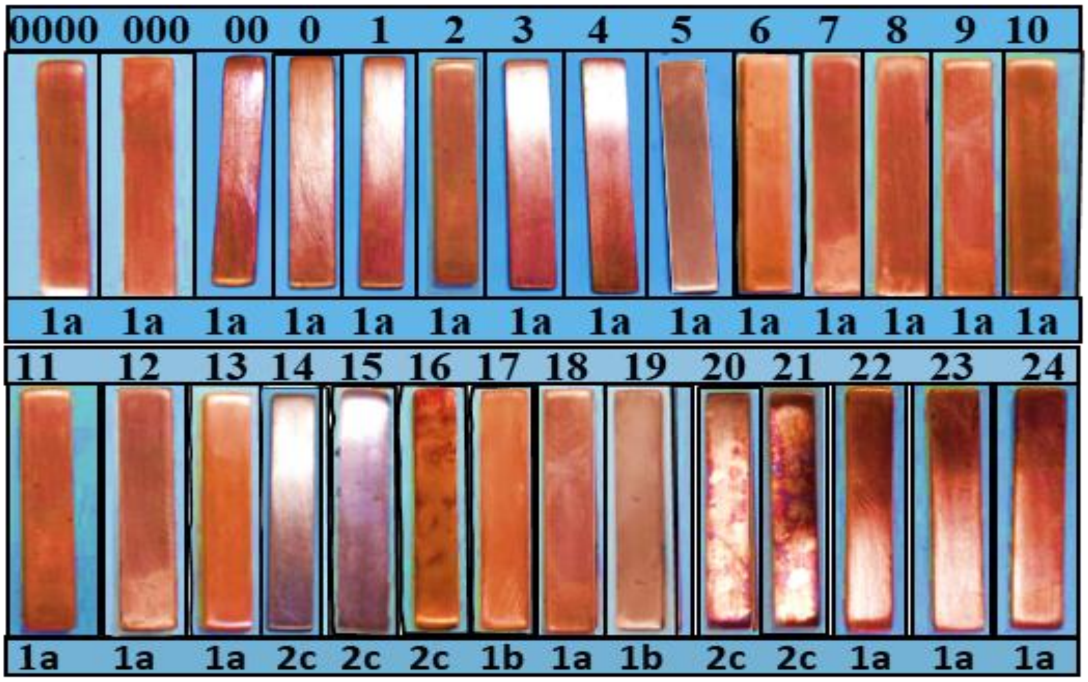

Figure 4. Illustrative images demonstrate the impact of additives on copper strip corrosion tests conducted on various types of pre-prepared Iraqi bentonite greases.

Table 7: The impact of additives on the corrosion of copper strips in various types of prepared greases.

Sample Code No.	Copper Strip Corrosion	Sample Code No.	Copper Strip Corrosion	Sample Code No.	Copper Strip Corrosion
0000	1a	7	1a	17	1b
000	1a	8	1a	18	1a
00	1a	9	1a	19	1b
0	1a	10	1a	20	2c
1	1a	11	1a	21	2c
2	1a	12	1a	22	1a
3	1a	13	1a	23	1a
4	1a	14	2c	24	1a
5	1a	15	2c		
6	1a	16	2c		

3.2 Four-Ball Wear Test

The wear scar diameters (WSDs) on the friction pair are directly related to the effectiveness of the grease resistance to the corrosion (i.e., anti-wear factor) that occurs in the equipment metals. There is a direct relationship between the WSD value and friction, as the WSD value decreases when friction decreases in the presence of lubricating greases. The test results indicated that adding both PTFE and graphite nanocomposites to the structures of the laboratory-prepared IBLGs made them have a high resistance to friction compared to the results of the WSD values obtained using lubricating greases free of additives as shown in Tables 8 and 9, where the test results showed the mean wear scar diameters (WSDs) of four balls when using Iraqi bentonite lubricating greases before and after using different percentages of additives, while Figures 5 and 6 show the analytical images that were measured for the wear scar diameters of the bottom balls, which result from the tribological test, using a microscopic instrument.

Table 8: Results obtained from the effect of adding PTFE in different percentages to Iraqi lubricating greases on the diameters of the four-ball wear scar.

Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)
0	4.5	00	4.0	000	6.75	0000	5.5
1	1.0	4	0.63	7	0.9	10	0.7
2	0.7	5	0.55	8	0.65	11	0.5
3	0.75	6	0.45	9	0.6	12	0.45

Table 9: Results obtained from the effect of adding graphite in different percentages to Iraqi lubricating greases on the diameters of the four-ball wear scar.

Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)	Sample Code No.	four-ball WSDs (mm)
0	4.5	00	4.0	000	6.75	0000	5.5
13	0.95	16	0.80	19	0.75	22	0.63
14	0.75	17	0.85	20	0.65	23	0.55
15	0.60	18	0.45	21	0.55	24	0.45

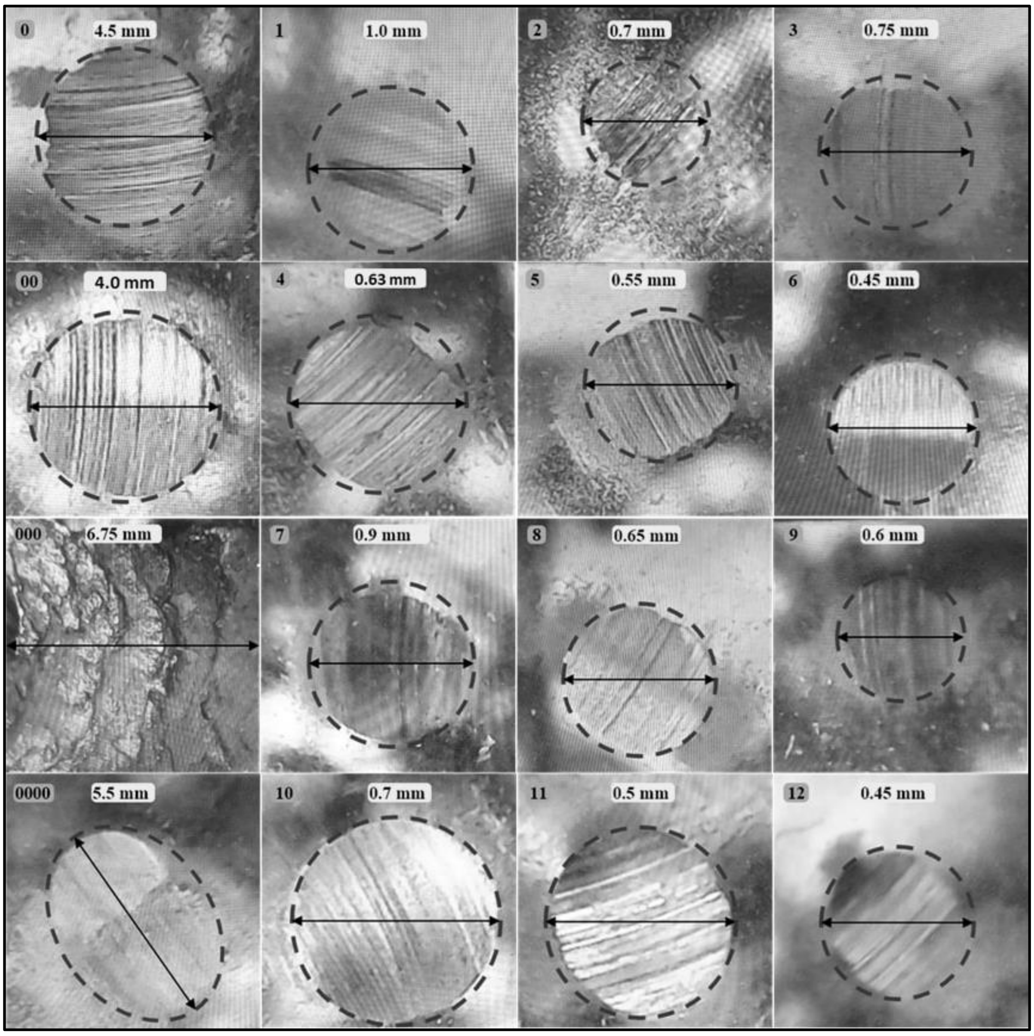

Figure 5. Analytical images of the four spherical wear scar diameters (mm) of prepared Iraqi grease before and after PTFE addition.

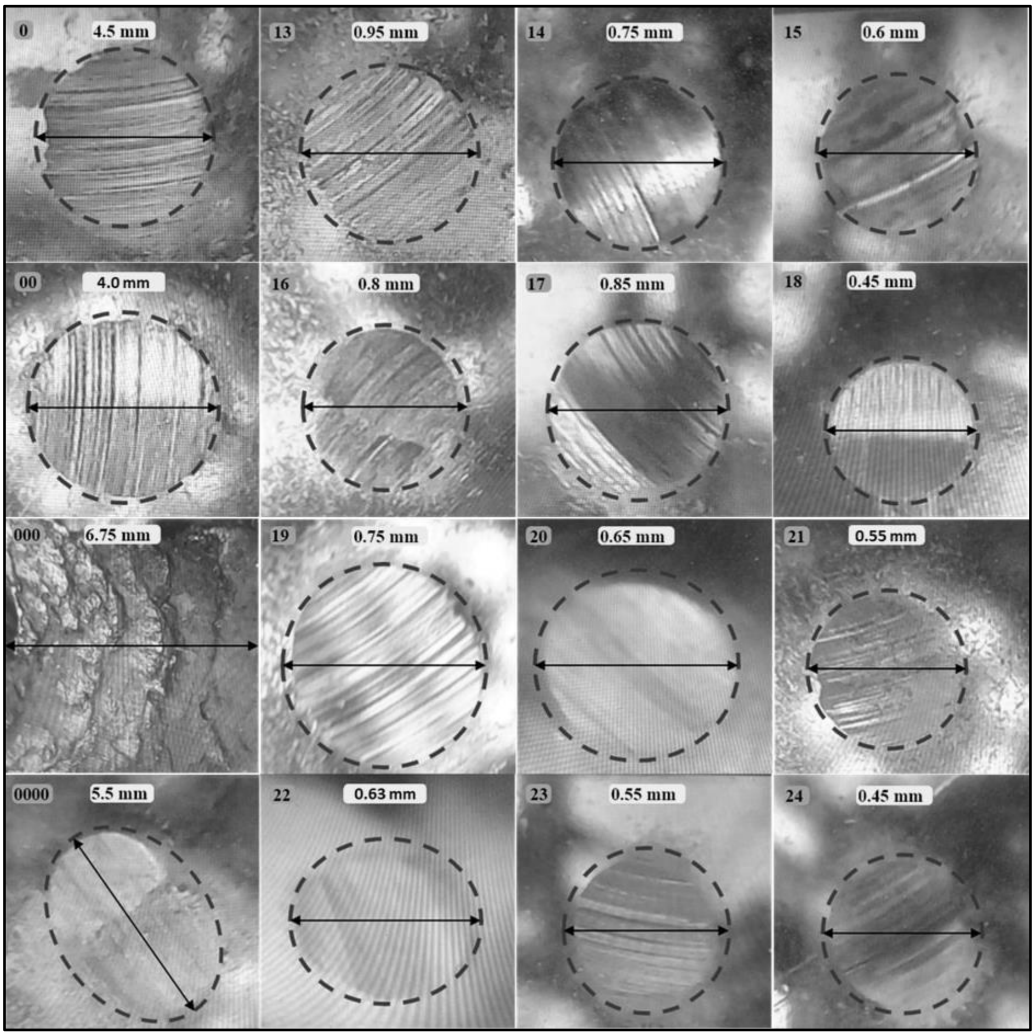

Figure 6. Analytical images of the four spherical wear scar diameters (mm) of prepared Iraqi grease before and after graphite addition.

Moreover, Figures 7 and 8 show a comparison between the mean wear scar diameters (WSDs) before and after adding PTFE and graphite nanoplatelets as additives to IBLG at three different weight percentages (i.e. 5, 10, and 15 wt. %). In general, it can be seen that the mean WSD of IBLG without any additives is significantly greater compared to the grease samples with additives. The friction resistance of the grease samples also increases with increasing the percentages of additives used until it reaches the lowest values of four-ball wear scar diameters using 15 wt. % additives, which indicates that the wear properties of IBLG are excellently improved by adding graphite and PTFE nanoplatelets. These additives increase the thickness of the boundary lubrication films formed on the surfaces of the lubricating greases and also increase their structural resistance to corrosion on the metal surfaces inside the industrial equipment in which the greases are used.

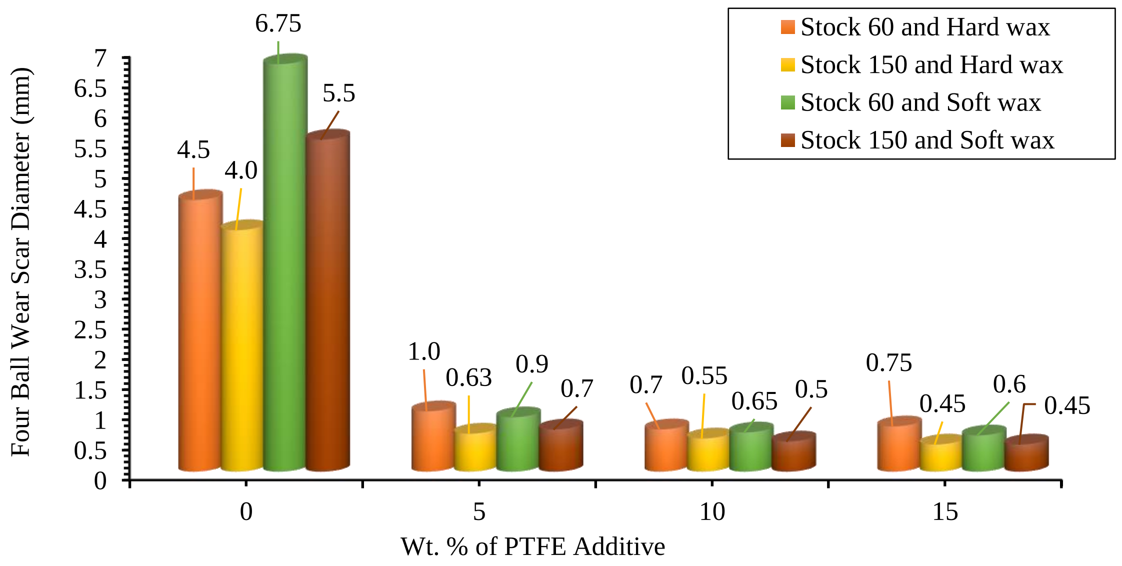

Figure 7. Evaluation of the WSDs results of steel ball carried out by four-ball tester before and after adding PTFE at 392 N.

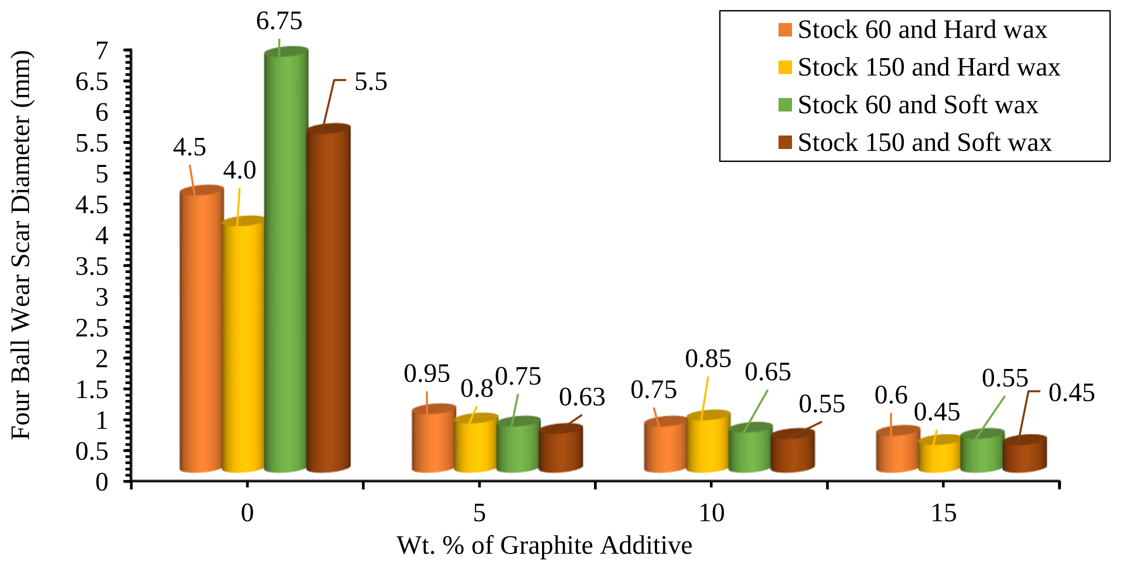

Figure 8. Evaluation of the WSDs results of steel ball carried out by four-ball tester before and after adding graphite at 392 N.

On the other hand, Figures 9 and 10 show the mean wear scar diameters (WSDs) before and after adding PTFE and graphite nanoplatelets to IBLG in the form of a graph. Figures 11 and 12 show the amount of variation in WSD results due to the effect of adding three different weight percentages of PTFE and graphite nanoplatelets to IBLG on the produced grease characteristics (i.e., resistance to friction and/or anti-wear). The lowest WSDs of 0.45 mm were obtained at the highest PTFE additive percentage, which reduced the WSDs by 90% for sample identified by code No. 6 (i.e., BOS 150, AIBHNO$_3$, hard wax, and 15% PTFE), and approximately 93% for sample identified by code No. 12 (i.e., BOS 150, AIBHNO$_3$, soft wax, and 15% PTFE) compared to the values of 4.5 mm for sample identified by code No. 0 (i.e., BOS 150, AIBHNO$_3$, and hard wax) and 6.75 mm for sample identified by code No. 000 (i.e., BOS 150, AIBHNO$_3$, and soft wax), respectively, of the additive-free IBLG parent samples. Similarly, the lowest WSDs of 0.45 mm were obtained at the highest graphite additive percentage, which reduced the WSDs by 90% for sample identified by code No. 18 (i.e., BOS 150, $AIBHNO_3$, hard wax, and 15% graphite), and approximately 93% for sample identified by code No. 24 (i.e., BOS 150, $AIBHNO_3$, soft wax, and 15% graphite) compared to 4.5 mm for sample identified by code No. 0 (i.e., BOS 150, $AIBHNO_3$, and hard wax) and 6.75 mm for sample identified by code No. 000 (i.e., BOS 150, $AIBHNO_3$, and soft wax), respectively, of the original IBLG samples without additives. The methodology used to compare the results of the analyses in this study is consistent with the methodology and results of the published literature [57-59].

In addition, comparing the results obtained from the four-ball wear test with the results of the copper strip corrosion, it can be noted that they are identical, as all samples (i.e., 6, 12, 18, and 24) gave copper corrosion degrees of 1a, as shown in Figure 4 and/or Table 7, which means that they provide good anti-wear and the best possible friction resistance. The results obtained in this study indicate that using base oil stock 150 for the purpose of manufacturing highly efficient Iraqi lubricating greases that work under severe operating conditions is better than using base oil stock 60, whether the wax used is hard or soft wax. It can also be concluded in general that the additions of both PTFE and graphite nanocomposites should be preferably at a weight percentage of not less than 15 wt. %.

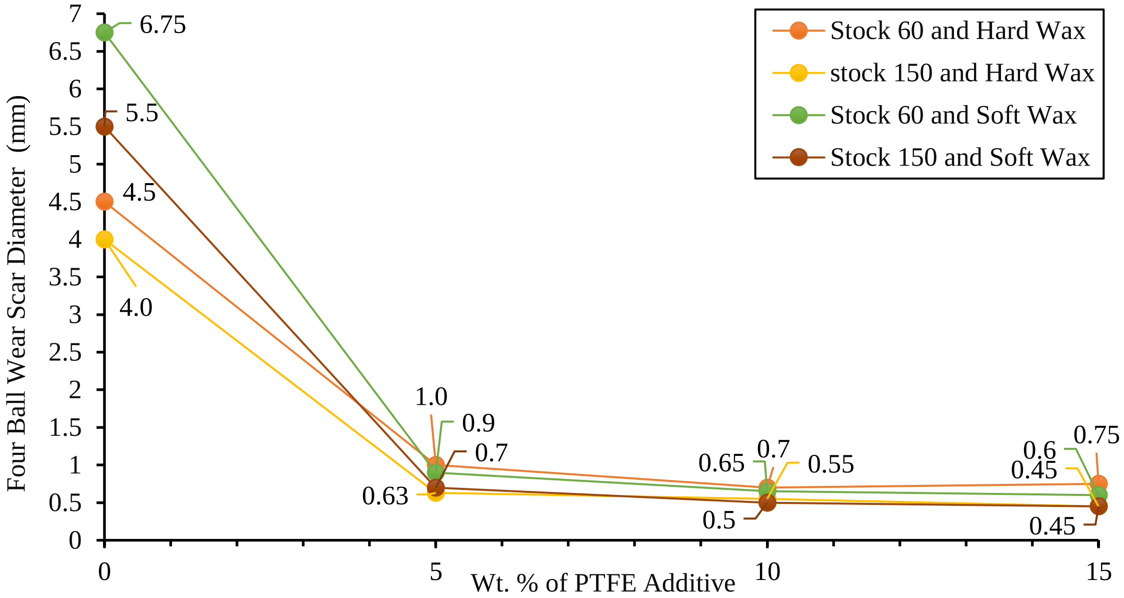

Figure 9: Effect of PTFE additive percentages in lubricating greases on the results of mean wear scar diameters (WSDs) compared to the original lubricating grease without additives.

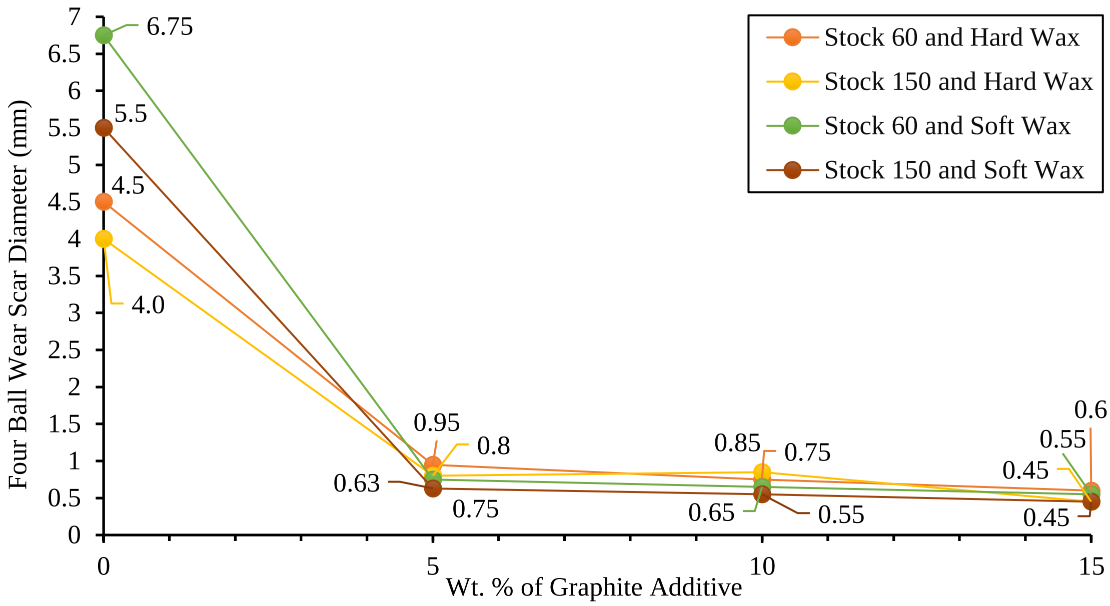

Figure 10. Effect of graphite additive percentages in lubricating greases on the results of mean wear scar diameters (WSDs) compared to the original lubricating grease without additives.

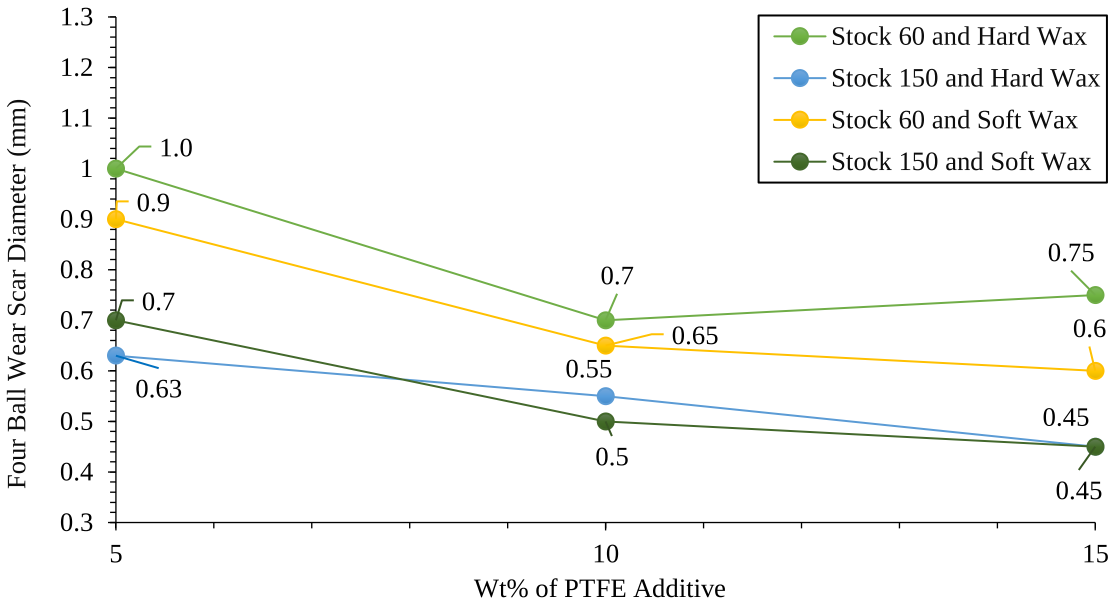

Figure 11. Amount of variation in WSD results of the four-ball test (mm) due to adding different percentages of PTFE to laboratory-prepared Iraqi lubricating greases.

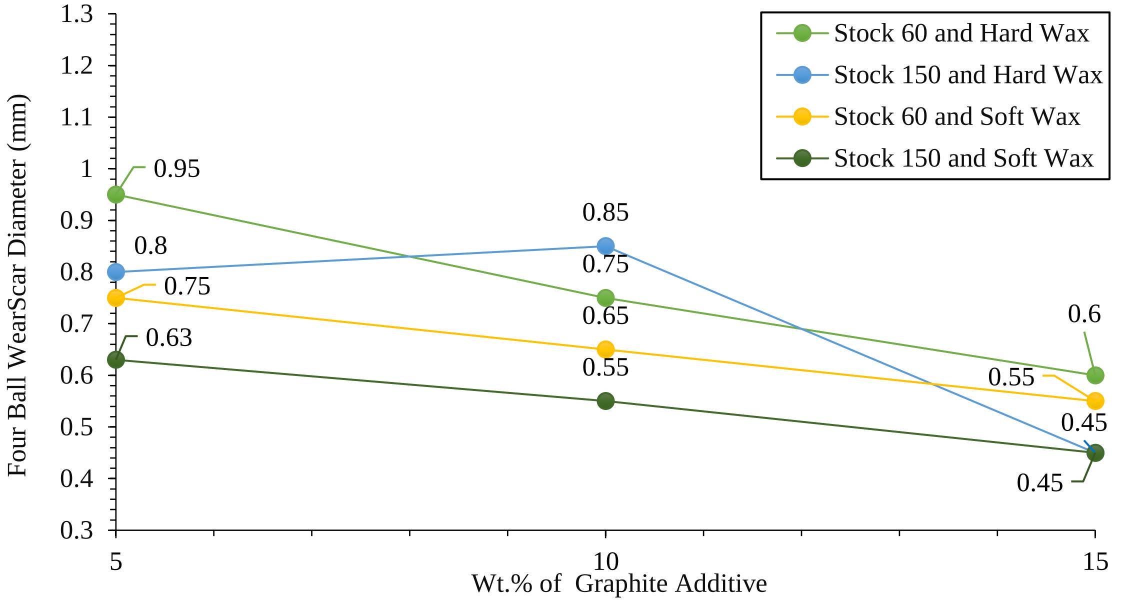

Figure 12. Amount of variation in WSD results of the four-ball test (mm) due to adding different percentages of graphite to laboratory-prepared Iraqi lubricating greases.

4. Conclusions

The effect of two types of Iraqi base oil Stock (BOS) and two types of locally manufactured paraffin waxes as well as the impact of weight percentages of nanoplate additives on the corrosion resistance properties and wear scar diameters (WSDs) of 24 types of laboratory-prepared Iraqi bentonite greases, were studied. A comparative analysis of the tribological characteristics of the basic bentonite grease with the addition of three percentages of PTFE and graphite nanocomposites was conducted. In the copper corrosion test, the color of the copper strip did not change after adding different percentages of PTFE, and the copper strip corrosion degrees were class 1a for all samples, indicating that Iraqi bentonite greases have excellent corrosion resistance to protect metal surfaces. However, when graphite nanoplatelets were added, there was a change in the color of the copper strip for some samples (i.e., it was of grade 1a with slight corrosion noted from grades 1b and 2c), indicating that this type of bentonite grease gives lower corrosion resistance when compared to the types in which PTFE nanoplatelets is used as an additive. In addition, the study showed that adding PTFE and graphite nanoplatelets can enhance the ability to anti-wear in severe operating conditions, as the WSD value decreased due to the decrease in friction between the balls in the presence of grease. The wear test showed the lowest WSD when the percentage of PTFE and graphite was about 15 wt. %. The results of the study also indicated that using base oil stock 150 for the purpose of manufacturing Iraqi lubricating greases is better than using base oil stock 60, whether the wax used is hard or soft wax. Finally, the study concluded that the greases prepared from BOS 150, $AIBHNO_3$, hard wax, and 15% PTFE as well as BOS 150, $AIBHNO_3$, hard wax, and 15% graphite had reduced WSDs by 90% compared to their parent grease. While the greases prepared with BOS 150, $AIBHNO_3$, soft wax, and 15% PTFE as well as BOS 150, $AIBHNO_3$, soft wax, and 15% graphite had reduced WSDs by approximately 93%.

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Reference

[1] Y. Meng *et al.*, “A review of advances in tribology in 2020–2021,” *Friction*, vol. 10, no. 10, pp. 1443–1595, Oct. 2022, doi: 10.1007/s40544-022-0685-7.

[2] M. Marian, A. Almqvist, A. Rosenkranz, and M. Fillon, “Numerical micro-texture optimization for lubricated contacts—A critical discussion,” *Friction*, vol. 10, no. 11, pp. 1772–1809, Nov. 2022, doi: 10.1007/s40544-022-0609-6.

[3] A. Rosenkranz, M. Marian, F. J. Profito, N. Aragon, and R. Shah, “The Use of Artificial Intelligence in Tribology—A Perspective,” *Lubricants*, vol. 9, no. 1, p. 2, Dec. 2020, doi: 10.3390/lubricants9010002.

[4] N. Yin, Z. Xing, K. He, and Z. Zhang, “Tribo-informatics approaches in tribology research: A review,” *Friction*, vol. 11, no. 1, pp. 1–22, Jan. 2023, doi: 10.1007/s40544-022-0596-7.

[5] V. Pandiyan, M. Akeddar, J. Prost, G. Vorlaufer, M. Varga, and K. Wasmer, “Long short-term memory based semi-supervised encoder—decoder for early prediction of failures in self-lubricating bearings,” *Friction*, vol. 11, no. 1, pp. 109–124, Jan. 2023, doi: 10.1007/s40544-021-0584-3.

[6] M. Perčić, S. Zelenika, and I. Mezić, “Artificial intelligence-based predictive model of nanoscale friction using experimental data,” *Friction*, vol. 9, no. 6, pp. 1726–1748, Dec. 2021, doi: 10.1007/s40544-021-0493-5.

[7] M. Ulas, O. Altay, T. Gurgenc, and C. Özel, “A new approach for prediction of the wear loss of PTA surface coatings using artificial neural network and basic, kernel-based, and weighted extreme learning machine,” *Friction*, vol. 8, no. 6, pp. 1102–1116, Dec. 2020, doi: 10.1007/s40544-017-0340-0.

[8] O. Altay, T. Gurgenc, M. Ulas, and C. Özel, “Prediction of wear loss quantities of ferro-alloy coating using different machine learning algorithms,” *Friction*, vol. 8, no. 1, pp. 107–114, Feb. 2020, doi: 10.1007/s40544-018-0249-z.

[9] F. König, C. Sous, and G. Jacobs, “Numerical prediction of the frictional losses in sliding bearings during start-stop operation,” *Friction*, vol. 9, no. 3, pp. 583–597, Jun. 2021, doi: 10.1007/s40544-020-0417-9.

[10] H. H. Parikh and P. P. Gohil, “Experimental investigation and prediction of wear behavior of cotton fiber polyester composites,” *Friction*, vol. 5, no. 2, pp. 183–193, Jun. 2017, doi: 10.1007/s40544-017-0145-y.

[11] X. Gao, K. Dai, Z. Wang, T. Wang, and J. He, “Establishing quantitative structure tribo-ability relationship model using Bayesian regularization neural network,” *Friction*, vol. 4, no. 2, pp. 105–115, Jun. 2016, doi: 10.1007/s40544-016-0104-z.

[12] R. Egala, G. V. Jagadeesh, and S. G. Setti, “Experimental investigation and prediction of tribological behavior of unidirectional short castor oil fiber reinforced epoxy composites,” *Friction*, vol. 9, no. 2, pp. 250–272, Apr. 2021, doi: 10.1007/s40544-019-0332-0.

[13] M. S. Hasan, A. Kordijazi, P. K. Rohatgi, and M. Nosonovsky, “Triboinformatic modeling of dry friction and wear of aluminum base alloys using machine learning algorithms,” *Tribol Int*, vol. 161, p. 107065, Sep. 2021, doi: 10.1016/j.triboint.2021.107065.

[14] S. N. A. Mohd Sofi, M. A. Abd Aziz, N. S. Anang Japar, N. W. Abdu Rahman, A. R. Abdulhalim, and M. Y. Mohd Yunus, “Preparation and characterization of grease formulated from waste transformer oil,” *IOP Conf Ser Mater Sci Eng*, vol. 702, no. 1, p. 012034, Nov. 2019, doi: 10.1088/1757-899X/702/1/012034.

[15] L. Heirendt, H. H. T. Liu, and P. Wang, “Aircraft landing gear greased slider bearing steady-state thermo-elastohydrodynamic concept model,” *Tribol Int*, vol. 82, pp. 453–463, Feb. 2015, doi: 10.1016/j.triboint.2014.04.001.

[16] N. De Laurentis, A. Kadiric, P. Lugt, and P. Cann, “The influence of bearing grease composition on friction in rolling/sliding concentrated contacts,” *Tribol Int*, vol. 94, pp. 624–632, Feb. 2016, doi: 10.1016/j.triboint.2015.10.012.

[17] Z. Cao, Y. Xia, and X. Ge, “Conductive capacity and tribological properties of several carbon materials in conductive greases,” *Industrial Lubrication and Tribology*, vol. 68, no. 5, pp. 577–585, Aug. 2016, doi: 10.1108/ILT-07-2015-0113.

[18] X. Feng, Y. Xia, P. Xie, and X. Li, “Classification and spectrum optimization method of grease based on infrared spectrum,” *Friction*, vol. 12, no. 6, pp. 1154–1164, Jun. 2024, doi: 10.1007/s40544-023-0786-y.

[19] C.J. Boner, “Manufacture and application of lubricating greases,” *New York, Reinhold publishing corp. 3rd ed.*, 1971.

[20] R. Keith Mobley., *Plant engineer’s handbook*. Butterworth-Heinemann, 2001.

[21] Syed Q. A. Rizvi, *A Comprehensive Review of Lubricant Chemistry, Technology, Selection, and Design*. 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959: ASTM International, 2009. doi: 10.1520/MNL59-EB.

[22] S. N. A. Mohd Sofi, M. A. Abd Aziz, N. S. Anang Japar, N. W. Abdu Rahman, A. R. Abdulhalim, and M. Y. Mohd Yunus, “Preparation and characterization of grease formulated from waste transformer oil,” *IOP Conf Ser Mater Sci Eng*, vol. 702, no. 1, p. 012034, Nov. 2019, doi: 10.1088/1757-899X/702/1/012034.

[23] S. SINHARAY and M. BOUSMINA, “Biodegradable polymers and their layered silicate nanocomposites: In greening the 21st

century materials world," *Prog Mater Sci*, vol. 50, no. 8, pp. 962–1079, Nov. 2005, doi: 10.1016/j.pmatsci.2005.05.002.

[24] H. Nourmoradi *et al.*, "Surfactant modified montmorillonite as a low cost adsorbent for 4-chlorophenol: Equilibrium, kinetic and thermodynamic study," *J Taiwan Inst Chem Eng*, vol. 59, pp. 244–251, Feb. 2016, doi: 10.1016/j.jtice.2015.07.030.

[25] C. K. Saurabh, S. Gupta, J. Bahadur, S. Mazumder, P. S. Variyar, and A. Sharma, "Mechanical and barrier properties of guar gum based nano-composite films," *Carbohydr Polym*, vol. 124, pp. 77–84, Jun. 2015, doi: 10.1016/j.carbpol.2015.02.004.

[26] N. Yin, Z. Xing, K. He, and Z. Zhang, "Tribo-informatics approaches in tribology research: A review," *Friction*, vol. 11, no. 1, pp. 1–22, Jan. 2023, doi: 10.1007/s40544-022-0596-7.

[27] K. Holmberg, R. Siilasto, T. Laitinen, P. Andersson, and A. Jäsberg, "Global energy consumption due to friction in paper machines," *Tribol Int*, vol. 62, pp. 58–77, Jun. 2013, doi: 10.1016/j.triboint.2013.02.003.

[28] K. Holmberg, P. Andersson, and A. Erdemir, "Global energy consumption due to friction in passenger cars," *Tribol Int*, vol. 47, pp. 221–234, Mar. 2012, doi: 10.1016/j.triboint.2011.11.022.

[29] K. Holmberg, P. Andersson, N.-O. Nylund, K. Mäkelä, and A. Erdemir, "Global energy consumption due to friction in trucks and buses," *Tribol Int*, vol. 78, pp. 94–114, Oct. 2014, doi: 10.1016/j.triboint.2014.05.004.

[30] A. Razzaq, T. Fatima, and M. Murshed, "Asymmetric effects of tourism development and green innovation on economic growth and carbon emissions in top 10 GDP countries," *Journal of Environmental Planning and Management*, vol. 66, no. 3, pp. 471–500, Feb. 2023, doi: 10.1080/09640568.2021.1990029.

[31] H. K. Nasif and A. Daham Wiheeb, "Absorption-Desorption Characteristics of the Synthesized Deep Eutectic Solvents for Carbon Dioxide Capture," *Diyala Journal of Engineering Sciences*, pp. 115–129, Sep. 2024, doi: 10.24237/djes.2024.17308.

[32] R. H. Savage, "Graphite Lubrication," *J Appl Phys*, vol. 19, no. 1, pp. 1–10, Jan. 1948, doi: 10.1063/1.1697867.

[33] M. Chhowalla and G. A. J. Amaratunga, "Thin films of fullerene-like MoS2 nanoparticles with ultra-low friction and wear," *Nature*, vol. 407, no. 6801, pp. 164–167, Sep. 2000, doi: 10.1038/35025020.

[34] "Bowden, Frank Philip, and David Tabor. The friction and lubrication of solids. Vol. 1. Oxford university press, 2001.".

[35] M. Dienwiebel, G. S. Verhoeven, N. Pradeep, J. W. M. Frenken, J. A. Heimberg, and H. W. Zandbergen, "Superlubricity of Graphite," *Phys Rev Lett*, vol. 92, no. 12, p. 126101, Mar. 2004, doi: 10.1103/PhysRevLett.92.126101.

[36] B. M. Kamel, A. Mohamed, M. El Sherbiny, K. A. Abed, and M. Abd-Rabou, "Tribological properties of graphene nanosheets as an additive in calcium grease," *J Dispers Sci Technol*, vol. 38, no. 10, pp. 1495–1500, Oct. 2017, doi: 10.1080/01932691.2016.1257390.

[37] A. Hernandez Battez, J. E. Fernandez Rico, A. Navas Arias, J. L. Viesca Rodriguez, R. Chou Rodriguez, and J. M. Diaz Fernandez, "The tribological behaviour of ZnO nanoparticles as an additive to PAO6," *Wear*, vol. 261, no. 3–4, pp. 256–263, Aug. 2006, doi: 10.1016/j.wear.2005.10.001.

[38] P. Gonzalez-Rodriguez, K. J. H. van den Nieuwenhuijzen, W. Lette, D. J. Schipper, and J. E. ten Elshof, "Tribochemistry of Bismuth and Bismuth Salts for Solid Lubrication," *ACS Appl Mater Interfaces*, vol. 8, no. 11, pp. 7601–7606, Mar. 2016, doi: 10.1021/acsami.6b02541.

[39] P. Murkute, J. Ramkumar, S. Choudhary, and K. Mondal, "Effect of alternate corrosion and wear on the overall degradation of a dual phase and a mild steel," *Wear*, vol. 368–369, pp. 368–378, Dec. 2016, doi: 10.1016/j.wear.2016.09.027.

[40] A. Arora, V. Jaswal, K. Singh, and R. Singh, "Applications of Metal/Mixed Metal Oxides as Photocatalyst: A Review," *Oriental Journal of Chemistry*, vol. 32, no. 4, pp. 2035–2042, Aug. 2016, doi: 10.13005/ojc/320430.

[41] Z.-W. Guo, C.-Q. Yuan, X.-Q. Bai, and X.-P. Yan, "Experimental Study on Wear Performance and Oil Film Characteristics of Surface Textured Cylinder Liner in Marine Diesel Engine," *Chinese Journal of Mechanical Engineering*, vol. 31, no. 1, p. 52, Dec. 2018, doi: 10.1186/s10033-018-0252-3.

[42] G. Ren, P. Zhang, X. Ye, W. Li, X. Fan, and M. Zhu, "Comparative study on corrosion resistance and lubrication function of lithium complex grease and polyurea grease," *Friction*, vol. 9, no. 1, pp. 75–91, Feb. 2021, doi: 10.1007/s40544-019-0325-z.

[43] G. Ren *et al.*, "Improving the lubrication and anti-corrosion performance of polyurea grease via ingredient optimization," *Friction*, vol. 9, no. 5, pp. 1077–1097, Oct. 2021, doi: 10.1007/s40544-020-0400-3.

[44] H. A. Abdullah, R. A. Anaee, A. A. Khadom, A. T. Abd Ali, A. H. Malik, and M. M. Kadhim, "Experimental and theoretical assessments of the chamomile flower extract as a green corrosion inhibitor for aluminum in artificial seawater," *Results Chem*, vol. 6, p. 101035, Dec. 2023, doi: 10.1016/j.rechem.2023.101035.

[45] A. A. Khadom, A. N. Abd, N. A. Ahmed, M. M. Kadhim, and A. A. Fadhil, "Combined influence of iodide ions and Xanthium Strumarium leaves extract as eco-friendly corrosion inhibitor for

low-carbon steel in hydrochloric acid," *Current Research in Green and Sustainable Chemistry*, vol. 5, p. 100278, 2022, doi: 10.1016/j.crgsc.2022.100278.

[46] S. A. Hussain, A. A. Khadom, and H. B. Mahood, "Yield Optimization for The Extraction of Organic Compounds from Okra Leaves Wastes," *Diyala Journal of Engineering Sciences*, pp. 19–26, Mar. 2024, doi: 10.24237/djes.2024.17102.

[47] "ASTM D4048-22 Standard Test Method for Detection of Copper Corrosion from Lubricating Grease," 2022, doi: 10.1520/D4048-22.

[48] "ASTM D 2266. Standard test method for wear preventative characteristics of lubricating grease (four-ball method). In: Annual Book of ASTM Standards. Philadelphia, PA, Section 5, Vol. 5.01, 1996.", doi: 10.1520/D2266-91R96.

[49] L. L. Radulovic and Z. W. Wojcinski, "PTFE (Polytetrafluoroethylene; Teflon®)," in *Encyclopedia of Toxicology*, Elsevier, 2014, pp. 1133–1136. doi: 10.1016/B978-0-12-386454-3.00970-2.

[50] X. Xu *et al.*, "An easy-processing organic-inorganic self-lubricating composite coating with high corrosion resistance," *Prog Org Coat*, vol. 137, p. 105377, Dec. 2019, doi: 10.1016/j.porgcoat.2019.105377.

[51] G. Li *et al.*, "Flake aluminum reinforced polyamideimide-polytetrafluoroethylene bonded solid lubricating composite coating for wear resistance and corrosion protection," *Eur Polym J*, vol. 152, p. 110485, Jun. 2021, doi: 10.1016/j.eurpolymj.2021.110485.

[52] Mohammed J. Abdul-Ghani, Bashir Y. Al-Zaidi, and Emad F. Mansour, "Preparation of water resistance greases from Iraqi Bentonite. 4th Int. Engineering conference – Mansoura University – Egypt, IEC 10-32, 51," 2004.

[53] H. Liu and L. Li, "Graphitic materials: Intrinsic hydrophilicity and its implications," *Extreme Mech Lett*, vol. 14, pp. 44–50, Jul. 2017, doi: 10.1016/j.eml.2017.01.010.

[54] X. Fan, Y. Xia, L. Wang, and W. Li, "Multilayer Graphene as a Lubricating Additive in Bentone Grease," *Tribol Lett*, vol. 55, no. 3, pp. 455–464, Sep. 2014, doi: 10.1007/s11249-014-0369-1.

[55] X. Fan, L. Wang, and Y. Xia, "Oil-soluble lithium salts as novel lubricant additives towards improving conductivity and tribological performance of bentone grease," *Lubrication Science*, vol. 27, no. 6, pp. 359–368, Oct. 2015, doi: 10.1002/ls.1286.

[56] Z. Wang, Y. Xia, and Z. Liu, "Comparative study of the tribological properties of ionic liquids as additives of the attapulgite and bentone greases," *Lubrication Science*, vol. 24, no. 4, pp. 174–187, Jun. 2012, doi: 10.1002/ls.1173.

[57] A. Saxena, D. Kumar, and N. Tandon, "Development of lubricious environmentally friendly greases using synergistic natural resources: A potential alternative to mineral oil-based greases," *J Clean Prod*, vol. 380, p. 135047, Dec. 2022, doi: 10.1016/j.jclepro.2022.135047.

[58] S. N. Gorbacheva, Y. M. Yarmush, and S. O. Ilyin, "Rheology and tribology of ester-based greases with microcrystalline cellulose and organomodified montmorillonite," *Tribol Int*, vol. 148, p. 106318, Aug. 2020, doi: 10.1016/j.triboint.2020.106318.

[59] J. E. Martín-Alfonso, M. J. Martín-Alfonso, C. Valencia, and M. T. Cuberes, "Rheological and tribological approaches as a tool for the development of sustainable lubricating greases based on nano-montmorillonite and castor oil," *Friction*, vol. 9, no. 2, pp. 415–428, Apr. 2021, doi: 10.1007/s40544-020-0407-y.