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Research paper

Tribological Examination of Surface-Modified Copper Sulfide Nanoparticles

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ABSTRACT

Today, advancements in technology across various fields are enhancing the quality, durability, and performance of machine components used in both machinery and vehicles. This evolution necessitates that parts in contact for extended periods can withstand high pressures and temperatures. Lubrication is one of the solutions. Thus, the demand for nano-lubricant additives is rising to improve the performance of lubricants and oils. In this study, bare CuS and Tween 80 (TW)-capped CuS nanoparticles (NPs) were synthesized as sulfur-containing lubricant additives using a solvothermal method. The particle sizes of both bare CuS and TW-capped CuS NPs were determined to be spherical, with sizes less than 15 nm. The addition of bare CuS NPs to 5W-30 base oil at a concentration of 0.05 (wt.%) reduced the wear rate by 50.2%, while TW-coated CuS NPs reduced the wear by 32.4%, compared to the base oil under high load level.

Keywords: *Copper Sulfide (CuS), Extreme Pressure Nano Additives, Tween 80, Anti-wear*

I. INTRODUCTION

Today, rapidly advancing technology in various sectors significantly impacts the quality, service life, and performance of machine components used in machines and vehicles, altering their usage parameters. This evolution necessitates that parts working in contact for extended periods exhibit resistance to high pressures and temperatures. Consequently, as a solution, there is an increasing demand for the development of lubricants that can minimize energy consumption and reduce cost losses caused by wear and friction. Nanoparticles are commonly used as oil additives to enhance lubrication effectiveness (Wang et al., 2012; Jason et al., 2020; Abud Ali et al., 2022; Özakın et al., 2025b). Metals, metal oxides, sulfides, carbon compounds, rare earth compounds, and nanocomposites have been used as lubricant additives (Uflyand et al., 2019; Raina et al., 2021; Li et al., 2022; Pawar et al., 2022; J. Wang et al., 2022; B. Wang et al., 2022; Nyholm & Espallargas, 2023; Wang et al., 2023). Nanoparticles improve the tribological properties of lubricants in the classification of extreme pressure (EP), antiwear (AW), and friction modifier (FM) additives (Tang & Li, 2014; Shahnazar et al., 2016; Minami, 2017; Thampi et al., 2021; Li et al., 2022). Nanoparticle characteristics can be enhanced through surface modifications to fulfill specific functions. Various materials, including molecules and polymers, can modify the surfaces of nanoparticles (NPs). Surfactants, in particular, play a crucial role in preparing nanoparticle-doped oils, improving their persistence by binding oil molecules to nanoparticles through their solubilization and adsorption properties (Özakın et al., 2025a). In the field of tribology, surfactants are vital for controlling lubricant

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properties such as oxidation resistance, emulsification, bio-resistance, and corrosion prevention, as well as reducing friction and wear (Adetunla et al., 2023). Sulfur-containing additives, including FeS (Zhang et al., 2000), MoS_2 (Nagarajan et al., 2024), WS_2 (Liu et al., 2025), ZnS (Uğur & Avan, 2023), Ag_2S (Ma et al., 2020), and Ce_2S_3 (Jiang et al., 2025) are commonly used additives for lubrication.

Copper sulfide (CuS) is a compound that contains sulfur and has a hexagonal structure. It is used in various fields, including optics (Rabkin et al., 2015), biology (Ain et al., 2022), photocatalysis, solar cells, sensors, and batteries, due to its excellent physical, chemical, electrical, optical, structural, and surface properties (Roy & Srivastava, 2015; Shamraiz et al., 2016; Yadav et al., 2019). Despite its wide range of applications, there are limited studies on the tribological effects of CuS nanoparticles (NPs). Some research suggests that CuS functions as a solid lubricant (Kovacı et al., 2018; Ishikawa et al., 2019; Kumar et al., 2023), which helps improve surface smoothness and reduce wear. Furthermore, CuS has been used in several studies to enhance the antiwear properties of oils. For instance, Kang et al. showed that adding oleic acid-modified CuS NPs improved the antiwear properties of paraffin oil (Kang et al., 2008). Chen and Zhu synthesized unmodified and oleic acid-modified CuS nanorods as oil additives, both demonstrating high antiwear properties (Chen & Zhu, 2017). Gu found that a 10% wear rate reduction of oleic acid-modified CuS nanorod additives compared to the pure CuS nanorod additives (Gu, 2018). Liu et al. highlighted that octadecylamine-coated CuS NPs additives enhanced the diethyl succinate ($C_8H_{14}O_4$) ester lubricant performance to 25 % (Liu et al., 2018). Hua et al. prepared methoxylpolyethylene glycol xanthate ($MPEGOCS_2K$) capped CuS NPs as an antiwear additive for water-based lubrication (Hua et al., 2024). In this study, bare CuS and Tween 80 (TW)-capped CuS NPs were synthesized using a solvothermal method. Both CuS NPs were characterized by FTIR, DLS, and SEM-EDX analysis. Particle sizes were determined through SEM and DLS analysis. The rheological properties of the nanolubricants were assessed via viscosity measurements. Wear tests were performed under an 80 kg load using a specially designed device that featured a rotating pin on a cylindrical geometry.

II. MATERIALS AND METHODS

A. Materials and Chemicals

The materials consist of pairs of cylinders made from 1.2379 steel alloy (62 HRC) and pins made from 1.2738 steel alloy (30 HRC) with dimensions of Ø21 x 15 mm. The following chemicals were sourced from reputable suppliers: ethanol from Merck (Germany), copper (II) acetate hydrate from Carlo Erba (Italy), Tween-80 (TW), and thioacetamide from Sigma Aldrich (USA). A fully synthetic 5W-30 A5 multi-grade engine oil was used to prepare nanolubricants.

B. Preparation of CuS NPS

The preparation of bare and TW-modified CuS NPs involved a solution-phase reaction carried out in both water and ethanol. First, 1.0 g of $Cu(OAc)_2{\cdot}H_2O$ (5 mmol Cu^{2+}) was dissolved in 100 mL of water and 50 mL of ethanol by heating the mixture to 60 °C while stirring vigorously at 1200 rpm for 30 minutes. A surfactant, TW (1.3 g), was dissolved in 20 mL of ethanol. This surfactant solution was then added to the Cu^{2+} solution. The mixture was stirred vigorously for 1 hour at 60 °C at 1200 rpm and then cooled to room temperature. Next, 0.5 g (6.65 mmol) of thioacetamide (C_2H_5NS) was added to the same beaker. The reaction was stirred for 24 hours at 80 °C. Afterward, the mixture was cooled to room temperature and transferred to 50 mL centrifuge tubes. The tubes were centrifuged at 9000 rpm for 20 minutes to remove excess reactants and salts by decantation. The residue was washed with minimal water and ethanol, and any remaining supernatant was removed by centrifugation. Finally, the residue was dried in a vacuum oven to produce a black powder of TW-modified CuS NPs. The bare CuS NPs were prepared using the same procedure but without a surfactant.

C. Preparation of Nanolubricants

Nanolubricants were prepared by adding bare CuS and TW-CuS NPs with a concentration of 0.05 (wt.%) to the base oil. Nanolubricants were stirred for 12 h with a magnetic stirrer (Wisestir, MSH20A, South Korea).

D. Characterization of CuS NPs

FTIR analysis was performed using the PerkinElmer Spectrum 100 spectrometer. Dynamic Light Scattering (DLS) measurements were done with the Zetasizer Nano-ZS. The structural and morphological characteristics of CuS nanopowder were analyzed using a Carl Zeiss Ultra Plus FESEM with Energy Dispersive X-ray (EDX) spectroscopy. Worn surfaces were examined with the Zeiss Discovery V12 optical microscope, and viscosity was measured using a Brookfield RV DV3T rheometer with a type 21 spindle and 250 rpm at 25 °C.

E. Wear Test Conditions

The custom wear test device was used in tests (Uğur & Avan, 2024). Tests were performed in duplicate. Test parameters included an 80 kg load, a duration of 5 minutes, a rotating speed of 1000 rpm, a temperature of 22 °C, and 10 mL of nanolubricants. Wear rate (mass loss in grams) was measured using an OHAUS Adventurer analytical balance (AR2140) with 0.0001 g accuracy after tribometer tests under lubricated conditions.

III. RESULTS AND DISCUSSION

A. CuS Synthesis Results

A. 1. FTIR Analysis

FTIR spectroscopy was used to define functional groups of synthesized NPs (Figure 1). The characteristic absorption peaks of Cu-S and Cu=S were found at 621 cm^{-1} and 1240 cm^{-1}, as noted in previous studies by Xi et al. (Xi et al., 2021), who observed these peaks at 620 cm^{-1} and 1222 cm^{-1}, while Joy et al (Raj et al., 2020) assigned the Cu–S stretching modes to 668 cm^{-1}. Hua et al. indicated that the enhanced C=C and C=O bonds at 1626 cm^{-1} and 1750 cm-1 suggest some C–S bond breakage, providing a sulfur source (Hua et al., 2024). These peaks were detected at 1654 cm^{-1}, 1746 cm^{-1}, and 1727 cm^{-1} for the bare CuS and TW-CuS NPs. Yadav et al. found peaks between 600-623 cm^{-1} and 1072-1140 cm^{-1} cm^{-1} when using $Cu(CH_3COO)_2{\cdot}H_2O$ as a precursor for synthesizing copper sulfide nanoparticles, as our precursor (Yadav et al., 2019). These peaks were detected between 590-621 cm^{-1} and 1106-1144 cm^{-1}. The characteristic absorption peaks between 2865-2993 cm^{-1}, especially the difference peak at 1298 cm^{-1} from the bare CuS NPs spectrum corresponding to C-H bands (Xi et al., 2021), were detected for the TW-CuS NPs sample, depending on the surfactant. The binding of TW to CuS NP is confirmed by its characteristic C-O bands of TW appearing in the range of 1100-1120 cm^{-1} (Khan et al., 2010).

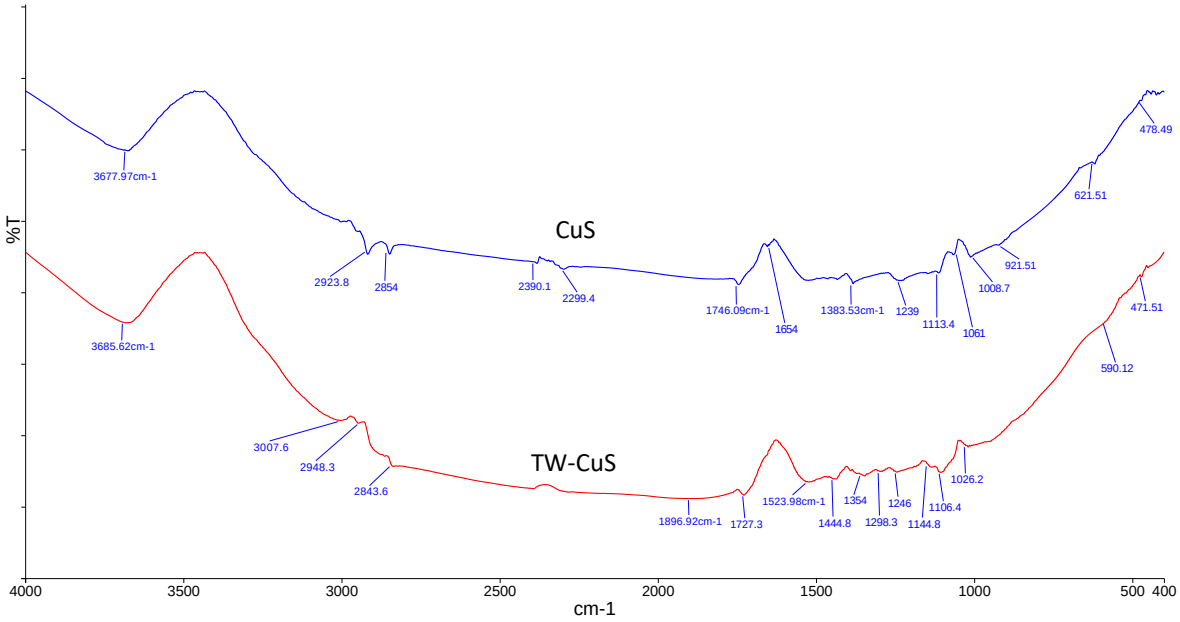


Figure 1. FTIR spectra of CuS and TW-CuS NPs.

A. 2. DLS Analysis for Measurement of Particle Sizes

DLS measurements revealed that the average sizes of the synthesized bare and TW-capped CuS NPs were approximately 291 nm and 394 nm, with a standard deviation values of 94.4, 109, respectively (Figure 2). Notably, Liu et al. reported that the average particle size of CuS nanoarrays treated with octadecylamine ($C_{18}H_{37}NH_2$) was about 1280 nm, which was higher than that of untreated CuS nanoarrays, which was around 825 nm (Liu et al., 2019). The scanning electron microscopy (SEM) micrographs of both synthesized CuS NPs, displayed in Figure 3, showed that the particle size was under 15 nm at a magnification of 100 kX. DLS measurements typically show higher z-averages than initial mean sizes from SEM (Perevyazko et al., 2010; Sentis et al., 2024). DLS captures the hydrodynamic size distribution of numerous particles in liquid, accounting for agglomerates and protective surfactants. In contrast, SEM provides high-resolution images for direct observation of particle shape and surface features.

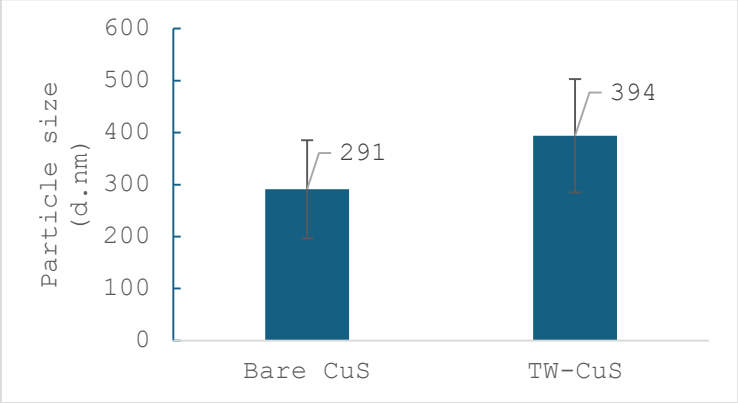
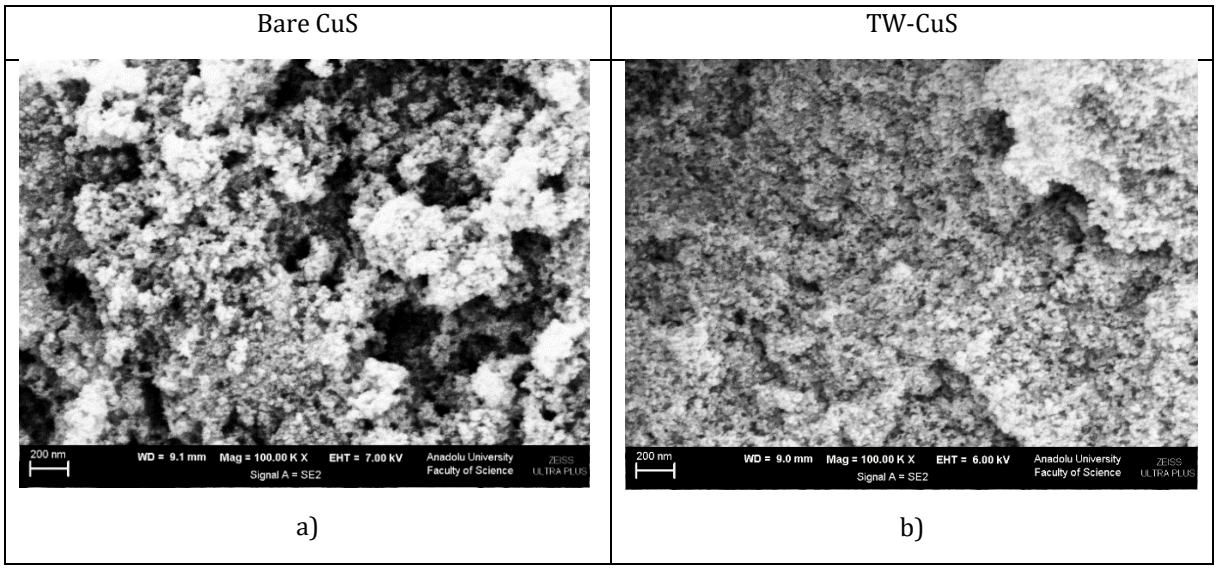
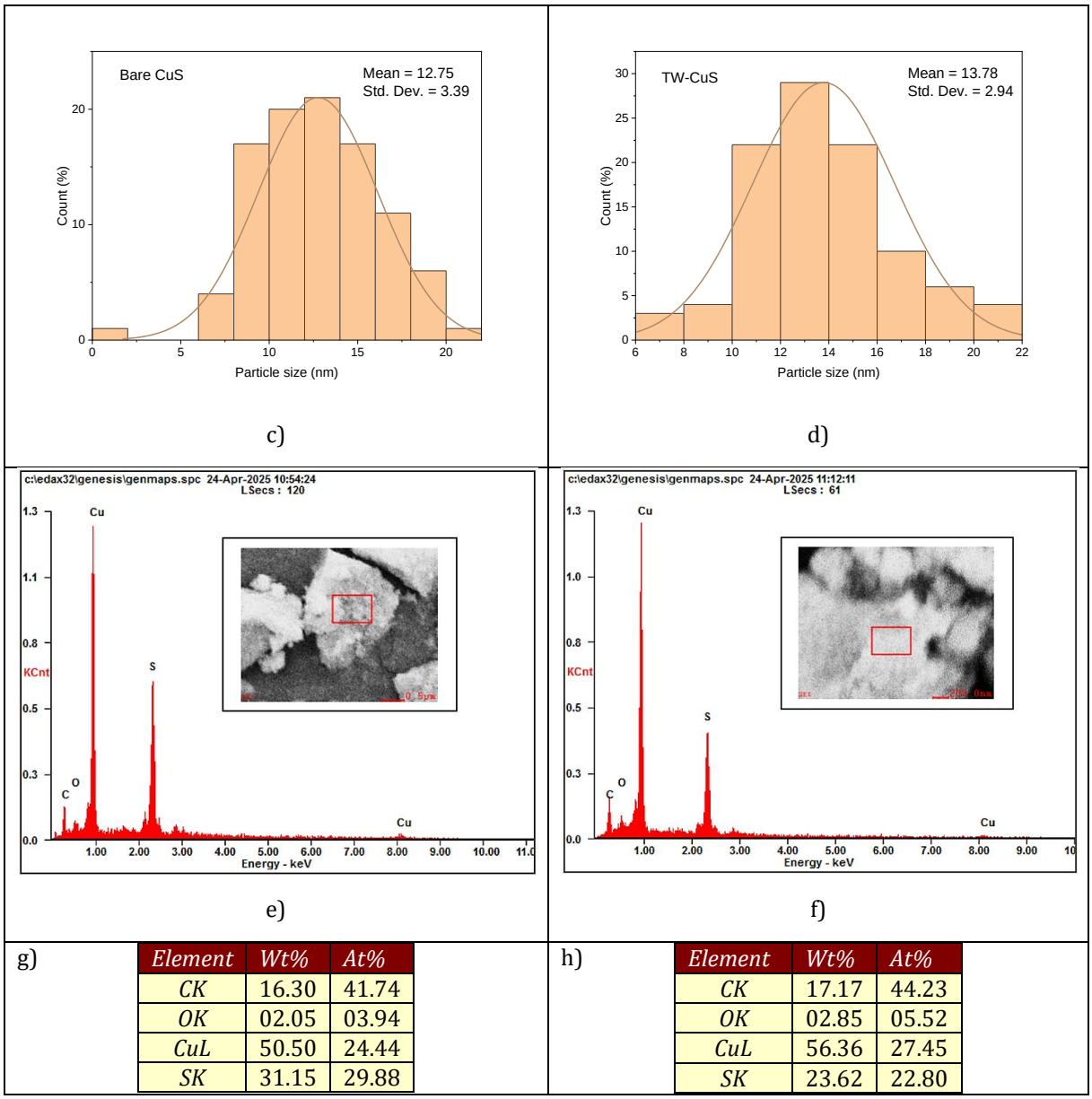


Figure 2. DLS measurement results of CuS and TW-CuS NPs in water.

A. 3. SEM-EDX Analysis

Figure 3. a, b. shows the SEM image of the CuS NPs. Figures 3c and 3d display the normal distribution function's typical fit to the particle size distribution histograms for both the bare CuS and TW-capped CuS nanoparticle (NP) samples. The particle size of bare CuS NPs is slightly smaller than that of TW-capped CuS NPs. EDX analysis of powdered CuS NPs shown in Figure 3. e, f, g, h confirmed the presence of Cu and S elements, with weight percentages of approximately 50% and 23%, respectively. The carbon (C) content increased from 16.3% to 17.1% for the TW-CuS NPs. The linear formula of the surfactant TW is $C_{64}H_{124}O_{26}$. EDX analysis confirmed the binding of carbon-rich TW to CuS NPs, showing increased carbon and oxygen percentages in the TW-CuS NPs.





g)

Element	*Wt%*	*At%*
CK	16.30	41.74
OK	02.05	03.94
CuL	50.50	24.44
SK	31.15	29.88

h)

Element	*Wt%*	*At%*
CK	17.17	44.23
OK	02.85	05.52
CuL	56.36	27.45
SK	23.62	22.80

Figure 3. SEM images of a) Bare CuS, b) TW-CuS NPs, and EDX analysis of e, g) Bare CuS and f, h) TW-CuS NPs, along with c, d) histogram analysis from FESEM images for both samples.

B. Rheological Analysis of the Nanolubricants

The viscosity values at the temperature of 25 °C were slightly decreased when the bare CuS and TW-CuS NPs were added to the 5W-30 base oil, as illustrated in Figure 4. Measurements were done in triplicate. The longer alkyl chain from TW decreased the viscosity of the base oil to 102.6 cP. Nanofluids generally have higher viscosities than their base fluids, which increase with nanoparticle concentration. However, the data in the literature are inconsistent (Murshed & Estellé, 2017). For instance, Qin et al. observed that stearic acid-modified CuS (60 nm) exhibits only a slight viscosity increase with higher volume fractions due to a small concentration gradient (Qin et al., 2017). In contrast, Jain et al. found that adding nanoparticles can reduce the viscosity of 5W40 engine oil (Jain et al., 2021). The reduction in viscosity is explained by a combination of percolation and NP structuring effects (Pakharukov et al., 2022). Sedimentation observations of the nanolubricants were conducted by comparing photographed images (Figure 5). After 30 days, a small amount of sediment was seen at the bottom of the nanolubricant test tubes. Bare CuS NPs, which were not treated with TW, exhibited better dispersion and stability in the base oil compared to TW-CuS NPs. The dark black color changed to brown for the nanolubricant containing TW-CuS NPs after 30 days. A concentration of 0.05 wt.% was identified as the optimal level based on sedimentation observations

among the various concentrations (0.05, 0.1, 0.2 wt.%) for both bare CuS and TW-CuS nanoparticles. Additionally, the wear results for the nanolubricants containing TW-CuS NPs were not superior to those containing bare CuS (Figure 6).

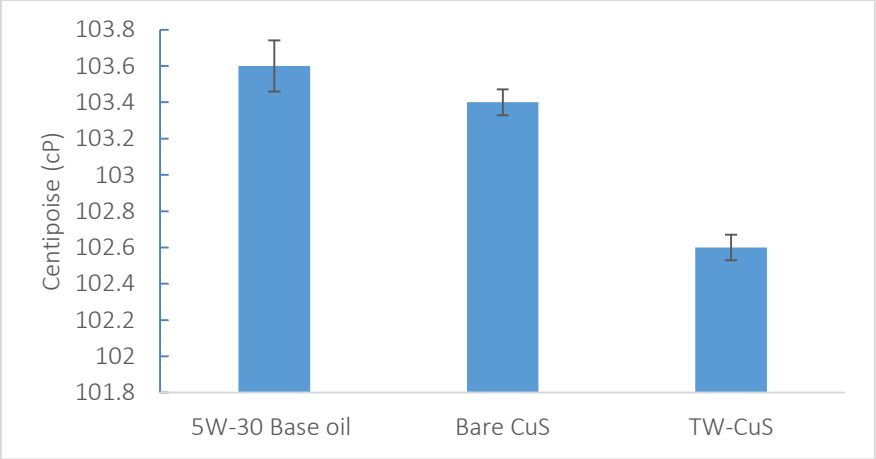


Figure 4. The results of the mean values and standard deviations (±0.14, ±0.07, and ±0.10, respectively) of the viscosity measurements.

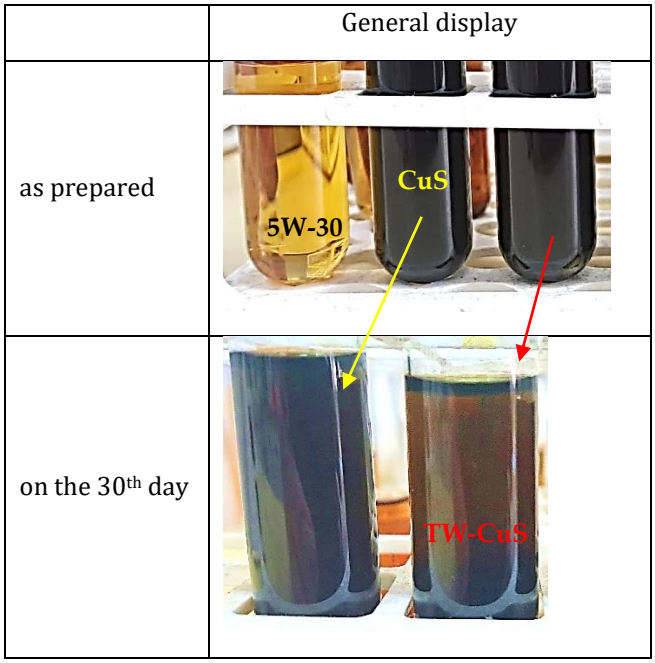


Figure 5. Dispersion properties of bare CuS and TW-CuS NPs in 5W-30 base oil.

C. Wear Test Results

The average wear reductions were calculated to be 50.2% for bare CuS NPs and 32.4% for TW-capped CuS NPs (Figure 6). Notably, the addition of functional groups to the NPs may change their structure and subsequently affect their tribological performance (Wang & Qu, 2025). However, in this case, bare CuS NPs demonstrated better anti-wear properties compared to the TW-CuS NPs. Additionally, a plausible explanation is that hard debris produced during rolling may get trapped in the hydrophobic tail groups of Tween 80 on TW-CuS NPs' surface. Under high load, this mixed debris could lead to agglomerate formation, which may undergo plastic deformation and become lodged between sliding surfaces. The dragging of these larger agglomerates could result in a more significant scar compared to clean bare CuO NPs. A similar situation was also previously observed while long alkyl chain modified CuO NPs (CuO-NP-C18) were used as a lubricant additive (Chimeno-Trinchet et al., 2019).



Figure 6. Wear test results of the mean values and standard deviations (±7.8x10^{-5}, ±14.1x10^{-5}, and ±7.1 x10^{-5}, respectively) of the nanolubricants.

C. 1. Optical Microscopy (OM) Analysis of Wear Scars

The OM and 2.5D profile images of worn surfaces from 5W-30 base oil, bare CuS, and TW-CuS are presented in Figure 7. The lowest wear losses were observed when the concentration of bare CuS NPs was 0.05 wt.% mixed with the base oil. OM images confirm these findings. The wear performance of the lubricant additive TW-CuS was better than that of the base oil but not as effective as bare CuS. Furthermore, the 2.5D images provided additional insights into surface roughness. The wear scar profiles indicated that the grooves caused by plough wear were diminished with the addition of CuS and TW-CuS NPs to the base oil, suggesting that the wear tracks were less pronounced with these nanoparticles. When TW-CuS NPs were used as a lubricant additive, some strips appeared on the worn surface. The black strips indicated minimal or no contact, while the white dashed strips showed moderate interaction between the metal pairs. Additionally, the dominant mechanisms are clearly indicated with arrows on the SEM images of the worn surfaces. The minimum wear rate was observed in lubricants formulated with bare CuS nanoparticles, where negligible adhesive wear and plastic deformations were noted. In contrast, the base oil 5W-30 without nano additives exhibited a higher wear rate due to delamination and abrasion wear. The use of TW-CuS nanoparticles resulted in a moderate improvement in wear rate; however, an increase in delamination wear was observed compared to the sample with CuS NPs. The lubrication regime was identified as boundary lubrication based on the Stribeck curve (Ludema, 1996; Totten, 2006; Hutchings & Shipway, 2017), with a lambda ratio calculated less than 1 ($\lambda \leq 1$) as 0.27 under an 80 kg load.

The Cu element was detected on the worn surfaces at 0.30 and 0.19 wt.% for the bare and TW-CuS samples in the base oil (BO), respectively, according to the SEM-EDX analysis (Figure 8 b,c). The S content was higher for the TW-CuS in the BO sample. No sulfide films were observed on the surfaces. The C content (wt.%) increased on the worn surfaces due to the hydrocarbon chain lengths of TW ($C_{64}H_{124}O_{26}$), rising from 10.6 to 13.5 (Figure 8c). EDX elemental composition (wt.%) results are shown in Table 1. Interestingly, plastic deformation-related scuffing regions were more prominent for TW-CuS than for bare CuS NPs. The best-worn surface belonged to the sample with bare CuS NPs, which had a lower wear loss ratio of around 50.2%.

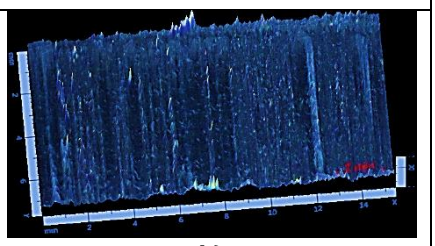
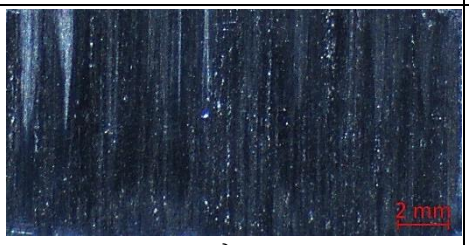
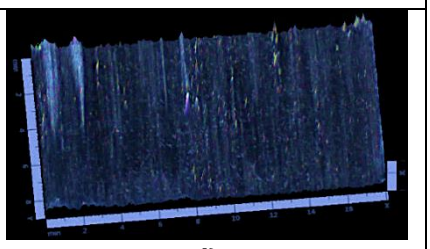
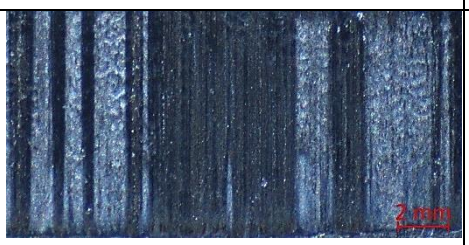
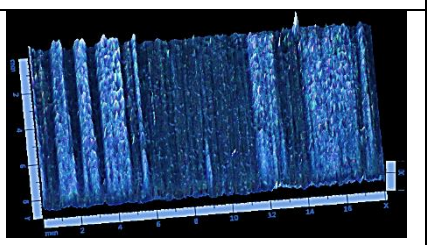
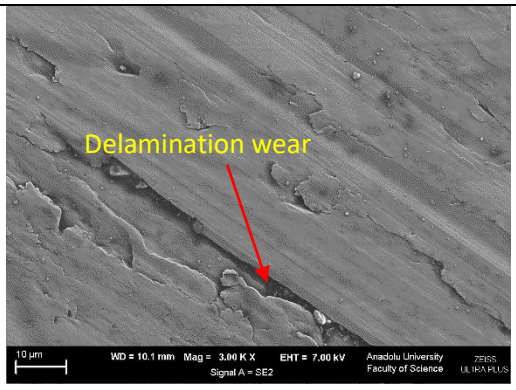
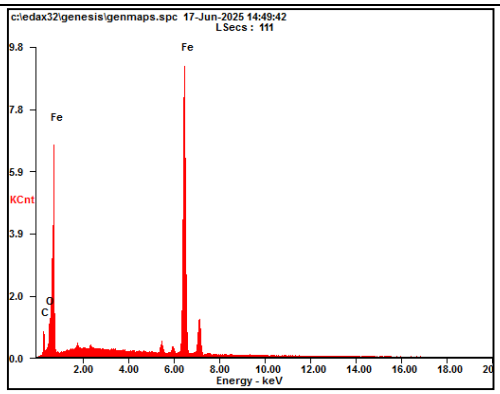
Samples	OM	2.5D
a) 5W-30 base oil	 a)	 b)
b) Bare CuS	 c)	 d)
c) TW-CuS	 e)	 f)

Figure 7. Optical images and 2.5D profiles of worn samples.

	SEM image	EDX analysis
a) 5W-30 base oil		

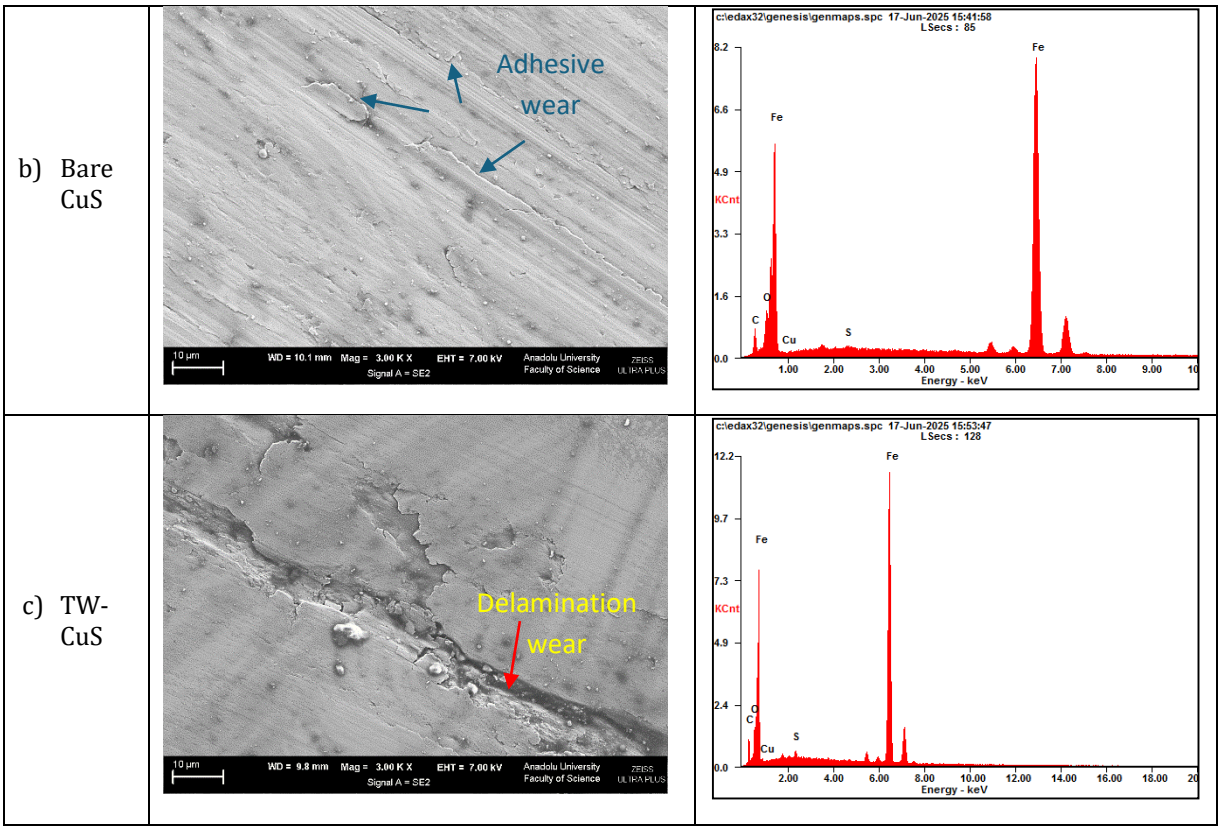


Figure 8. SEM micrographs and EDX analysis of a) 5W-30 Base oil, b) Bare CuS, and c) TW-CuS NPs 0.05 (wt.%) NPs concentrations on worn surfaces.

Table 1. EDX elemental composition (wt.%) results for some worn surfaces.

Sample No, NP	Element	C	O	Cu	S	Fe
5W-30 base oil (BO)	Composition (wt.%)	12.85	4.61	-	-	82.53
Bare CuS in BO		10.62	4.96	0.30	0.29	83.83
TW-CuS in BO		13.57	4.86	0.19	0.67	80.71

IV. CONCLUSION

The bare CuS and TW-CuS nanoparticles were synthesized in spherical shapes, measuring 12.7 nm and 13.7 nm, respectively. These nanoparticles were used as lubricating oil additives, as confirmed by SEM images. EDX analysis of the powdered CuS nanoparticles confirmed the presence of copper (Cu) and sulfur (S) elements, with weight percentages of approximately 50% and 23%, respectively. When both bare and TW-capped CuS nanoparticles were added to the 5W-30 base oil, a decrease in oil viscosity was observed. According to the Stribeck curve, the lubrication regime was identified as boundary lubrication, in which the adhesive wear mechanism was the predominant mode of wear for the sample CuS NPs. The wear test results indicated a reduction in the wear rate of 50.2% for bare CuS nanoparticles and 32.4% for TW-CuS nanoparticles compared to the base oil, at a concentration of 0.05 wt.% under an 80 kg load. These findings provide a new approach to developing nano-additive design for lubricants.

DECLARATIONS

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Conflict of Interest Disclosure: The author declares no conflict of interest.

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Ethical Approval and Participant Consent: This study does not involve human or animal participants. All procedures followed scientific and ethical principles, and all referenced studies are appropriately cited.

Plagiarism Statement: This article has been evaluated for plagiarism, and no instances of plagiarism were detected.

Availability of Data and Materials: Data sharing is not applicable to this study.

Use of AI Tools: The author declares that no Artificial Intelligence (AI) tools were used in the creation of this article.

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