engineering proceedings

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Proceeding Paper

Investigating the Friction Coefficient of Titanium Bolts with Vegetable Oils as Lubricants †

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† Presented at the 53rd Conference of the Italian Scientific Society of Mechanical Engineering Design (AIAS 2024), Naples, Italy, 4–7 September 2024.

Abstract: Threaded fasteners are one of the most important joining techniques, especially due to their ease of mounting and dismounting. These are essentially friction joints; therefore, friction coefficients between the surfaces play a critical role in the correct mounting of the threaded fasteners. Materials and lubrication conditions are the major factors that can affect the correct preload of the threaded fasteners. Particularly, when shifting from steel to titanium fasteners to achieve a high strength-to-weight ratio, the friction shift is very significant. Researchers have studied varying levels of lubrication to achieve optimum friction conditions. VG46 has been used most in the literature; however, its non-renewable nature necessitates the use of another alternative that is good for the environment and can cause a reduction in pollution in the environment. For this reason, in the present study, castor oil and fractionated coconut oil have been used for Ti bolts to achieve low friction coefficients. Torque tension tests have been performed on the Ti bolts using the different lubricants mentioned above and friction coefficients at the underhead and the threaded portions are compared with the commercial VG46 lubricant. Castor oil shows good performance compared to the other lubricants tested in terms of underhead friction coefficients, whereas the thread friction coefficients remain almost the same in all the lubrication conditions.

Keywords: titanium bolts; friction coefficient; lubrication; vegetable oils; coconut oil; sustainability

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Academic Editors: Umberto Galietti, Gabriele Arcidiacono, Enrico Armentani, Davide Castagnetti, Vigilio Fontanari, Aurelio Somà and Nicola Bonora

Published: 18 April 2025

Citation: Croccolo, D.; De Agostinis, M.; Fini, S.; Khan, M.Y.; Mele, M.; Olmi, G.; Scapecchi, C.; Tariq, M.H.B. Investigating the Friction Coefficient of Titanium Bolts with Vegetable Oils as Lubricants. *Eng. Proc.* **2025**, *85*, 52. https://doi.org/10.3390/engproc2025085052

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1. Introduction

The importance of threaded fasteners in machines and industrial applications is inevitable. Their significant advantage over other joining mechanisms such as rivets and welding is the ease of mounting and dismounting without destroying the joint. For maintenance reasons, this is the most important characteristic through which joints can be mounted and dismounted multiple times. For this reason, many researchers are studying the strength of threaded fasteners during multiple retightenings [1].

Friction plays a crucial role in the joining of threaded fasteners, being even more critical when they are subjected to multiple tightenings, such as retightening after maintenance works of the machinery. Therefore, lubrication is generally used in threaded fasteners to achieve low friction coefficients and optimum performance. The clamp load that keeps together two parts in a bolted joint is achieved by applying a tightening torque (T) to

the head of the screw (or the nut). This is governed by the Motosh formula, as shown in Equation (1).

$$T = F\left(\frac{p}{2\pi} + \frac{\mu_{th} d_2}{2\cos\alpha} + \mu_b R_b\right) \quad (1)$$

where F and T represent the preload force and the applied tightening torque, and p and α represent the pitch of the screw and the half-angle of the thread profile, respectively. In Equation (1), the term μ_{th} denotes the coefficient of friction at the interface between the threads of the screw and those of the nut, while d_2 is the pitch diameter [2]. Similarly, R_b and μ_b correspond to the effective bearing radius and the friction coefficient between the surfaces under the head, respectively.

The general rule of lubrication is clearly visible in Equation (1), where a change in the friction coefficient will result in a change in the tightening torque T to achieve a specific preload F. The mating of two components is the major concern that often increases friction after multiple interactions. Due to this purpose, the stability of friction coefficients is a major concern that affects the amount of preload offered by the tightening torque, as discussed above. Researchers have studied the effect of different lubricants to achieve this goal, especially over multiple retightenings [3–6].

Moreover, to reduce pollution and global warming, efforts are being made to reduce the weight of the machinery by varying materials and design techniques. In this regard, threaded fasteners are also being designed to have a lower weight to replace the existing high-weight threaded fasteners [7,8]. Ti screws are one of the main alternatives to steel screws that provide this type of advantage. However, this change in materials results in a change in the friction coefficients, which can drastically change the amount of preload achieved for a certain tightening torque. Croccolo et al. have studied the change in friction coefficients due to the use of Ti screws with steel nuts and Al bushing. The total friction coefficient obtained during the testing of Ti screws without lubrication was 0.35 [9]. This is very high compared to steel screws with aluminum nuts, which can provide a friction coefficient at a maximum of 0.28 according to VDI 2230 [10].

An initial study regarding the dependence of the friction coefficient upon oil type was conducted by Kato et al. (1985) [11]. Numerous oils have been studied in the past, including anti-wear oils [9,12], engine oils [13], and machine oils [14,15]. A comparison of different mineral oils with varying viscosities was conducted by Zou et al., who concluded that the friction coefficients are inversely proportional to the viscosity of the mineral oils used. Researchers have widely used VG46 as a lubricant in the category of mineral oils. Croccolo et al. [16] have studied the effectiveness of VG46 in relation to various materials and coatings. They inferred that when VG46 is used on both steel and aluminum plates, it provides stability of the friction coefficients with multiple tightening cycles. Also, a ceramic paste suitable for high-temperature applications was tested by Croccolo et al., which gave low friction coefficients and high stability for a number of retightenings [9]. By using different types of coatings and surfac10 treatments, researchers have tried to reduce the friction coefficients and minimize the use of lubricants, specifically by introducing minimum quantity lubrication (MQL) techniques. However, a large increase in the financial and redesign terms is attained, which is sometimes not possible in practice [4]. In addition to the above-mentioned mineral oils being non-renewable and a source of pollution for air and soil [17], vegetable-derived alternatives have been developed and tested in the past [18–20]. Researchers have tried to replace mineral oils with these vegetable-derived alternatives in different scenarios, such as in the automotive industry [21,22], manufacturing [23,24], and hydraulics [25,26].

Castor oil extracted from the castor plant is inedible and has several industrial applications [27]. Specifically, it is used for gear lubrication [28]. In addition, Bongfa et al.

compared castor oil to high-quality crankcase oil for use in automotive and power plant engines. They concluded that the frictional characteristics of the castor oil were superior to that of the crankcase oil tested [29]. A salient feature of castor oil is its significantly higher viscosity compared to other vegetable oils [30,31]. Furthermore, Dwivedi and Sapre's study demonstrated the potential for producing environmentally friendly grease using castor seeds [32]. Additionally, Jayadas and Nair reported that coconut oil exhibited a lesser degree of weight gain under oxidative conditions when compared to sesame and sunflower oils. The addition of specific additives allows for the modification of its pour, thereby optimizing its performance [33]. Furthermore, the potential application of coconut oil in motor engines for lubrication has also been studied by Tulashie and Kotoka [31]. Additionally, C. Mizera et al. explored the potential of coconut oil as a substitute for motor oil. Their research utilized a specific extraction process to obtain yellow coconut oil, which exhibited comparable properties to motor oil [34].

To the best of the author's knowledge, no systematic study has been carried out to potentially replace the non-renewable lubricants with renewable oils for the bolted joints made of Ti. Kato et al. (1985) conducted a study on the lubrication properties of castor oil when used with steel bolts [11]. Croccolo et al. systematically evaluated the performance of four vegetable oils including sesame, sunflower, coconut, and castor oil, and compared it to VG46 mineral oil [35]. The study yielded favorable outcomes when vegetable oils were utilized. Notably, fractionated coconut oil, a subject of particular interest in this study exhibited friction coefficients comparable to those of MoS_2. Particularly, the lubricating properties of this oil remained unaffected by the variations in tightening speed. Furthermore, sunflower, sesame, and fractionated coconut oil exhibited more stable friction coefficients compared to the commercially available VG46 lubricant. Also, Castor oil exhibited a substantial sensitivity to variations in the tightening speed; however, it was the most viscous oil used in the study among the other lubricants employed, suggesting its potential for lubrication on slippery surfaces, such as titanium. The rheological characterization of the various oils utilized in the study is presented in Table 1 [35]. The characterization of the oils was performed using the Zhan cup, in accordance with the ASTM standard D4212 [36].

Table 1. Viscosity of the lubricants measured in cSt at room temperatures 23 °C, 40 °C, and 60 °C.

Lubricant	Oil Viscosity @ 23 °C (cSt)	Viscosity @ 40 °C (cSt)	Viscosity @ 60 °C (cSt)
VG46	84 ± 0.00	49.13 ± 0.52	19.43 ± 0.52
Fractionated Coconut oil	19.80 ± 0.00	9.90 ± 0.00	5.50 ± 0.00
Castor oil	878.13 ± 6.98	409.47 ± 6.98	89.83 ± 1.65

The results indicate that at 60 °C, FCO exhibited a 72% reduction in viscosity compared to room temperature, while VG46 exhibited a slightly higher reduction of 77%. For CSO, the viscosity at 60 °C decreased by 90% relative to the room temperature, demonstrating the significant temperature dependency of CSO viscosity.

In the present study, coconut oil (best performing [35]) and castor oil (most viscous [35]) were utilized to assess their performance on Ti bolts in comparison to VG46 mineral oil. The present study investigates the performance of tightening tests on the test rig to obtain the friction coefficients over multiple tightenings for the aforementioned lubrication conditions.

2. Methodology

2.1. Materials

The tests were conducted on hexagon socket head M10 × 1.25 × 55 Ti cap screws with dimensions according to ISO 4762 and ISO 4033 M10 × 1.5 class 10 steel nuts [37,38]. For the purpose of comparison, hexagon socket head M10 × 1.25 × 55 class 12.9 St cap screws

were utilized, having dimensions in accordance with ISO 4762 for both sets of bolts. The plates utilized for testing were fabricated from medium carbon steel (ISO 683-1, C45) [39].

The bolts and the plates were washed using ultrasonic cleaning in paraffin oil (Kemipol®), prior to the commencement of the testing to ensure the efficacy of cleaning. The washing cycle was 15 minutes at 40 °C and was performed on a benchtop ultrasonic cleaner (STS-090-T04H300 by Sonixtek®, Rochesterlaan 11, 8470 Gistel, Belgium).

The selection of lubricants was informed by a comprehensive review of the literature, with a focus on identifying the most efficacious lubricants in terms of friction coefficients and viscosity. The optimal lubricants identified in this analysis included coconut and castor oils [35]. The latter was selected as the surface of Ti is slippery, so highly viscous lubricant will adhere more effectively resulting in prolonged contact time. As illustrated in Figure 1, FCO and CSO exhibited the lowest friction coefficients among the liquid lubricants. Furthermore, CSO demonstrated the highest viscosity among the other lubricants, suggesting it has potential benefits for Ti bolts. To further assess the functionality of these lubricants in practical scenarios, a comparative analysis was conducted with a commercial VG46 oil.

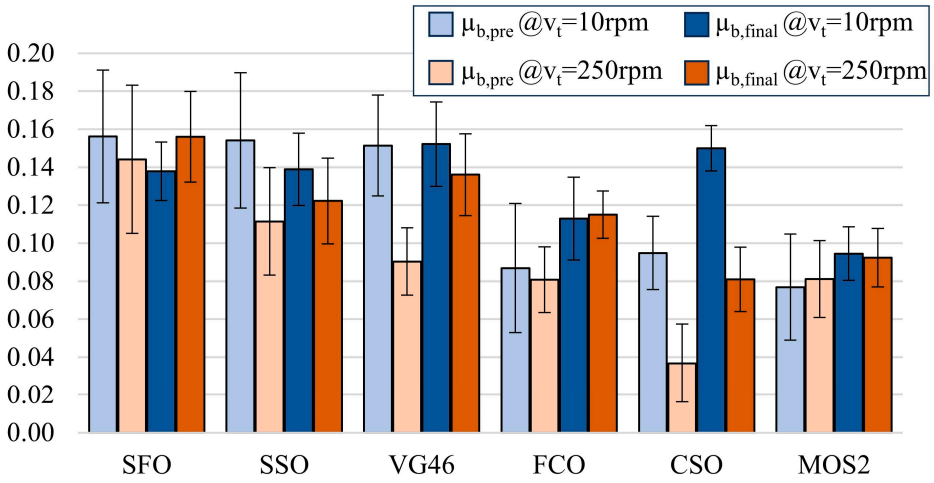


Figure 1. Average values of the bearing coefficients of friction (μ_b) [35].

VG46 oil, selected for this work, was Hydro 46 by Arexons®, Milan, Italy with a viscosity index of 100. Coconut and castor oils were procured from BENVOLIO 1938®, Treviso, Italy. The methodology of cold pressing was utilized to perform the biological extraction of these oils.

In accordance with a previous study by Croccolo et al., fractionated coconut oil was again utilized in this study for obtaining liquid lubrication at room temperature [35]. This oil is liquid at room temperature due to the removal of lauric acid during the fractionation process. Lauric acid is classified as an intermediate between long and medium-chain fatty acids [40].

During the process of lubrication, the bolts were inverted and the lubricant was applied to the bolt root and allowed to spread evenly up to the bolt head. Subsequent to reaching the bolt head, the bolt was subjected to a series of tightening tests. During the ten subsequent retightening, lubrication was only administered at the beginning, with no reapplication of lubricant to the bolts.

2.2. Tribological Tests

The test bench utilized for the tribological testing of the bolts was "Analyse System" by Kistler GmbH, Winterthur, Switzerland. The test setup is illustrated in Figure 2.

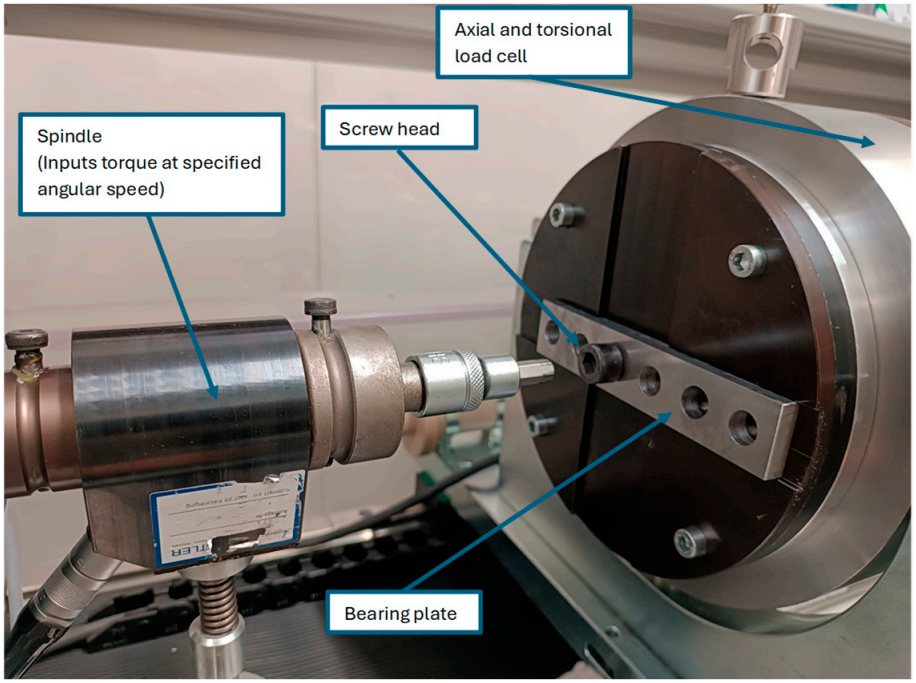


Figure 2. Kistler GmbH's apparatus utilized for the tribological testing of the threaded fasteners, encompassing the bolt head, plate, and tightening head.

During the course of this experimentation, torque was measured through the spindle. The torsional load cell, as depicted in Figure 2 is employed to assess thread friction torque. The axial force exerted by the load application unit is measured by the axial load cell. The measurement of the bearing friction torque is achieved by subtraction of the thread friction torque from the total torque, as indicated in Equation (2). Utilizing the acquired data, the friction coefficients μ_b and μ_t are subsequently calculated through the application of Equations (3) and (4) as outlined in ISO 16047 [41]. The equations are expressed as follows:

$$T_b = T - T_{th} \tag{2}$$

$$\mu_{th} = \frac{\frac{T_{th}}{F} - \frac{p}{2\pi}}{0.577 d_2} \tag{3}$$

$$\mu_b = \frac{T_b}{0.5 D_b F} \tag{4}$$

In the present study, a two-fold criterion was established as an extreme limit for the test, encompassing both torque and force control. The upper limits of torque and preload force were established, and the test was stopped once either of the criteria was met. Furthermore, the application of torque was executed in two distinct steps, as illustrated in the torque-angle generic graph in Figure 3.

Volkswagen standard was utilized to construct the testing cycle in the present study. Initially, the bolt is manually tightened to allow full contact of the head with the plate. Subsequently, the bolt is then loosened by three rotations, i.e., 1080°. A 0.5-second pause is then implemented to allow synchronization of the test rig. This is followed by the pretightening phase, in which the bolt is tightened up to 30% of the final force with the desired rotational speed v_t. Following this, a 2-second pause is allocated for the final tightening, which is conducted until the maximum load or maximum torque is reached (whichever occurs first). It is noteworthy that in all the scenarios, the final tightening is executed at a rate of 10 revolutions per minute (RPM). Following a subsequent 2-second

pause, the process of untightening is initiated at the rotational speed v_t. To mitigate the accumulation of heat due to friction, a 20-second pause is incorporated after each re-tightening cycle.

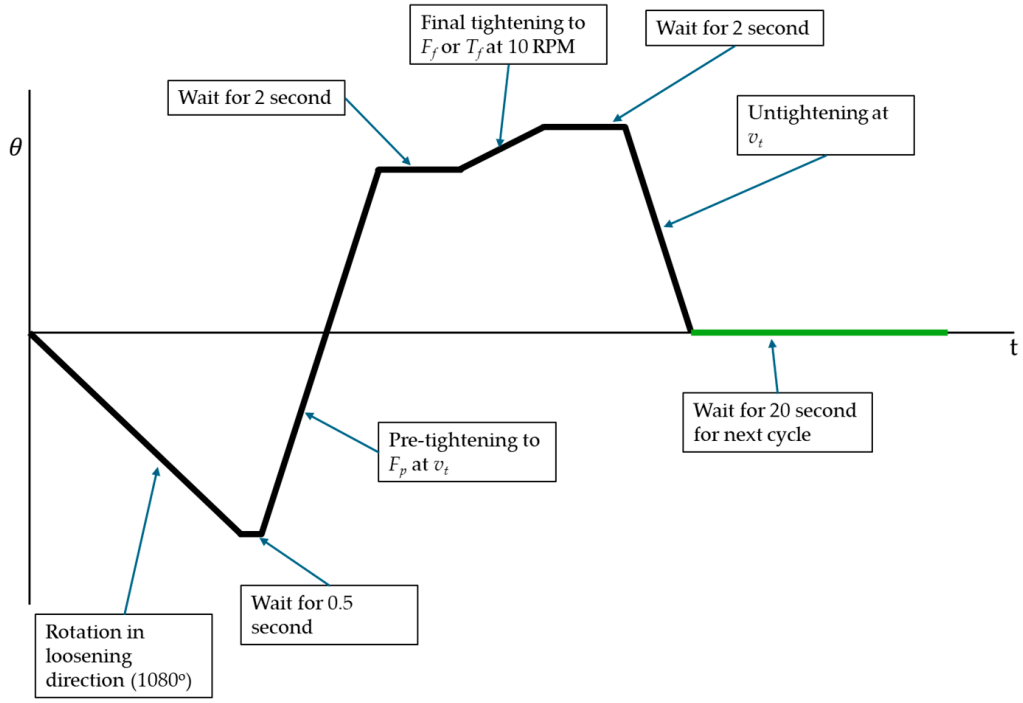


Figure 3. The torque-angle generic graph of the tribological tests demonstrating torque application in two distinct steps.

The final force was defined in accordance with the provisions stipulated in the VDI 2230 standard [10]. The preload force for the actual friction coefficients measured during the preliminary testing phase was designated as the final force. The friction coefficients μ_b and μ_t measured during the preliminary testing were found to be 0.16. The final force selected for this purpose was 44 kN and the preliminary force was 13.2 kN. The maximum torque criteria was set to 60 Nm, a topic that will be explained in Section 3.1.

Following the tribological experimentation, the bolt underhead surface and plates were observed using a ZEISS Stemi 508 stereo microscope manufactured by ZEISS Microscopy headquartered in Oberkochen, Germany.

2.3. Design of the Experiments

A full factorial design of experiments was employed in the present work in which three parameters were varied: lubrication, tightening speed, and the number of tightenings.

As previously mentioned, the lubricants were selected from the extant literature to test lightweight Ti bolts in conjunction with high strength steel bolts [35]. Three lubrication conditions were tested i.e., VG46 mineral oil, CaStor oil (CSO) and fractionated coconut oil (FCO).

The pre-tightening phase involved varying the tightening speed v_t between two levels: 10 RPM and 250 RPM. These rotational speeds are representative of the manual and automated fastening processes. However, the final tightening speed was maintained at 10 RPM, as higher rotational speeds lead to higher inertia, thereby hindering precise torque control, as per the Volkswagen VW 01131-1 standard [42].

Ten retightenings were performed on each bolt, to assess the variability of friction coefficients associated with mounting and dismounting processes for maintenance reasons. Each experimental point was repeated four times, with a new nut and screw utilized for

each repetition. These parameters are detailed in Table 2. Consequently, the full factorial design of the experiment resulted in 240 tightening cycles on 24 Ti bolts and similarly 240 tightening cycles on 24 St bolts. It is noteworthy that all the tests were conducted at a constant ambient temperature of 23 °C.

Table 2. Synopsis of the design of experiments.

Parameter	Number of Variables	Detail of Variables
Lubrication	3	VG46, Castor oil, Coconut oil
Tightening speed	2	10 rpm, 250 rpm
Number of tightenings	10	1… 10

Subsequent to the completion of the experimental phase, a statistical analysis, namely Analysis Of Variance (ANOVA), was performed on the experimental factors to ascertain the significance of the results. This analysis was carried out using StataSE 18 by StataCorp LLC, College Station, TX, USA.

3. Results and Discussion

The results and discussion section will begin with a discussion of the VW procedure according to VW 01131-1 with a maximum load of 44.5 kN, which was initially chosen. However, preliminary tests revealed critical issues with Ti screws, as outlined in Section 3.1.

3.1. Preliminary Tests

During the preliminary tests, the Ti screws exhibited anomalous failures on the head. Specifically, during the testing phase, failure occurred at the bolt head when the torque value exceeded the bolt head's strength. This phenomenon is likely attributable to the unconventional design of the bolt head, which enables diminished load capacity. The conical shape of the screw head, as depicted in Figure 4, renders it less stiff in comparison to the cylindrical shape of the steel screws.



Figure 4. M10 × 1.25 × 55 Ti bolt with special head design resulting in reduced loadability with respect to torque application.

When the torque reaches a sufficiently high value, the sides of the bolt head undergo plastic deformation due to very high stresses on the faces. In the subsequent cycle, when torque is applied, it fails the sides of the bolt that are already plastically deformed as illustrated in Figure 5a,b. This plasticized configuration of the screw head renders the tool incapable of fastening the screw, or it may become stuck inside the bolt head, as illustrated in Figure 5c.

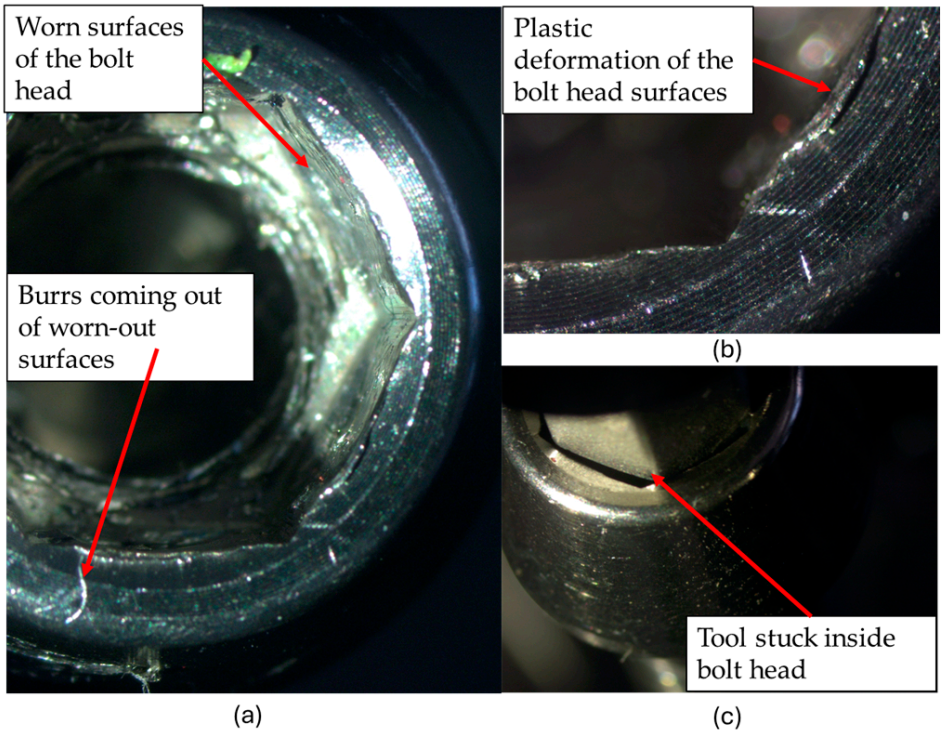


Figure 5. Worn-out bolt head showing (**a**) worn-out surfaces with burrs coming out of the head, (**b**) plastic deformation of surfaces before wear and tear of the surfaces, and (**c**) tool stuck inside bolt head when the surface of the bolt started to wear.

Furthermore, torque levels increase at higher rotational speeds due to the inertial effect, which can lead to higher torque levels and the failure of the bolt head. To address this issue, the utilization factor was increased, with the torque maintained at 60 Nm. It is noteworthy that the maximum torque required to reach the maximum preload limit for St screws (44.5 kN) was approximately equivalent to 60 Nm.

3.2. Tribological Tests

Subsequent to the preliminary testing, a two-fold criteria was established for the execution of the experimental characterization. The final preload was set at 44.5 kN and cut-off torque was kept at 60 Nm. The experiment was terminated as soon as either of the two criteria were met. The ensuing sections present the results obtained with each lubricant.

3.2.1. VG46

The evolution of the average friction coefficients of St screws using VG46 with the number of tightenings is shown in Figure 6. Coefficients of friction for tightening up to 30% of the final preload are shown in Figure 6a,b for tightening rotational speeds of 10 and 250 RPM. It is evident from the data that relatively high coefficients of friction are observed in the initial stage, which become stable after three to four tightenings. This outcome aligns with the findings reported in the literature [35]. This phenomenon can be attributed to the initial wear of the surfaces, which serves to remove the predominant peaks. Furthermore, it is observed that at 10 RPM, the friction coefficient is marginally lower during the initial tightening phase compared to 250 RPM. This observation can be attributed to the reduced centrifugal force at lower tightening speeds, leading to a higher effect of lubricant and, consequently, a lower friction coefficient.

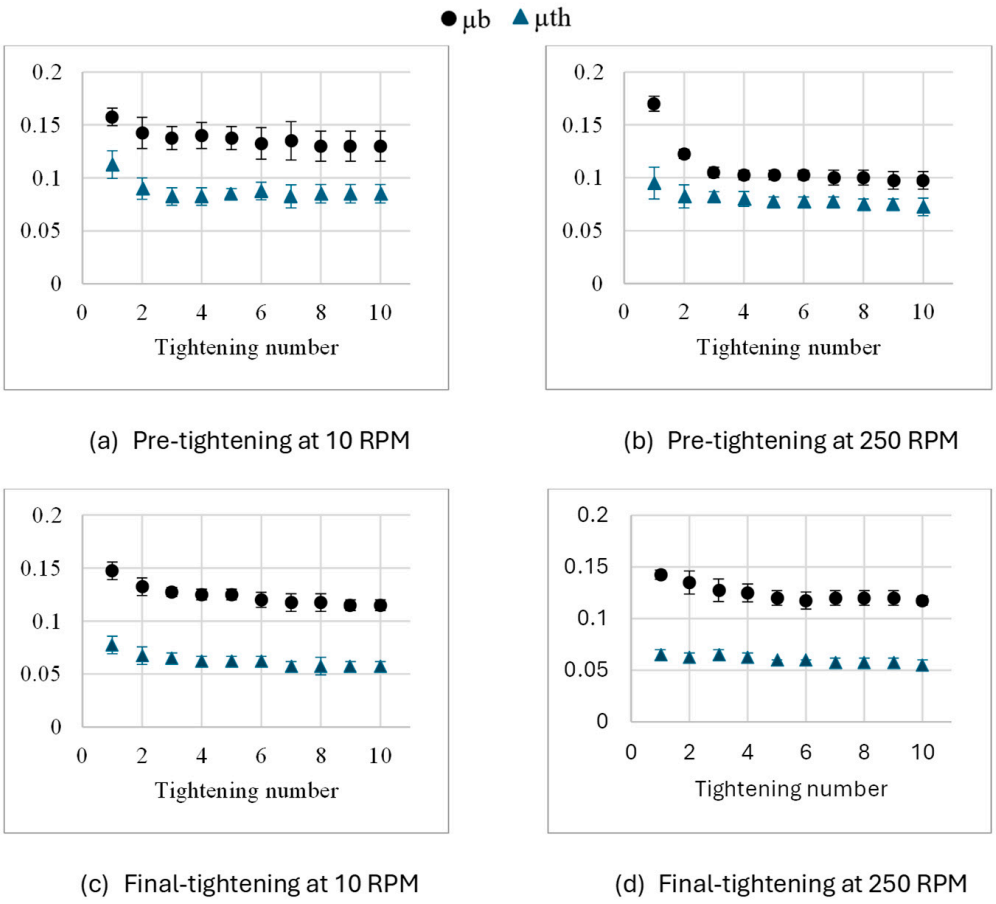


Figure 6. Friction coefficients during different tightenings for St VG46.

However, in the final stage of tightening, the trend of the friction coefficient is essentially the same. This observation may be attributed to the fact that the final tightening invariably is performed at the limited lubrication condition. As will be demonstrated during the forthcoming ANOVA statistical analysis, in the case of steel for final tightening, the underhead friction coefficient does not depend on the rotational speed, having a *p*-value greater than 0.05. This outcome lends further credence to the hypothesis that the impact of pre-tightening speed on temperature and dispersion of the lubricant does not exert a significant influence on the outcome of final tightening, which invariably is performed in limited lubrication conditions.

An examination of the Ti screws when VG46 is used as the lubricant, we have the friction coefficients given in Figure 7. The initial observation is that the underhead friction coefficients are considerably higher than the friction coefficients of the steel screws. Specifically, the average friction coefficients range from 0.45 to 0.54 during pre-tightening and from 0.38 to 0.48 during the final tightening. The elevated values of these friction coefficients may be attributed to the stripping away of the VG46 during the tightening cycle, resulting from the slippery surface of Ti. This phenomenon, known as the limit lubrication condition, arises due to direct contact between the asperities of the screw and the plate, resulting in elevated friction coefficients. However, the thread friction coefficients are approximately equal to that of steel, suggesting that the lubricant remains interlocked between the thread surfaces.

Moreover, the friction coefficients demonstrate notable repeatability, with the exception of pretightening at 250 RPM. Initial tightening at higher velocities (v_t) exhibits elevated friction coefficients, which may be attributed to the pronounced resistance exhibited by the asperities at the onset of the cycle. Furthermore, at the higher rotational speed, the

lubricant's removal is hindered due to the small time interval, leading to its adsorption on the surface. Consequently, the friction coefficients decrease for the final tightening phase in the high-speed tightening cycle.

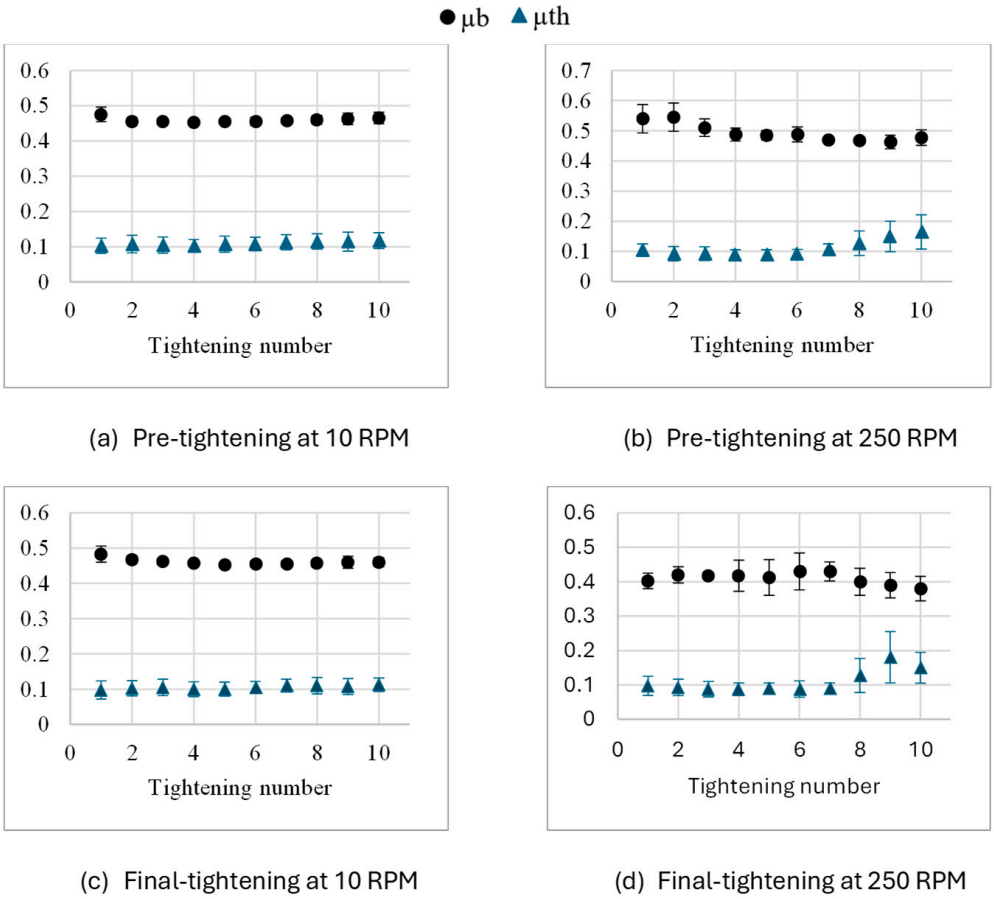


Figure 7. Friction coefficients during different tightenings for Ti VG46.

The microscopic observation of plates after the test is shown in Figure 8. It is evident that the plates' underhead surfaces for steel screws remain smooth attributed to the low friction coefficients. However, the surfaces of the plates in contact with the underhead surfaces of the Ti screws undergo a process of stripping due to the harder nature of the Ti surface making contact with the asperities of the steel plate at the micro level, resulting in a return to a rougher state at the macro level and elevated friction coefficients.

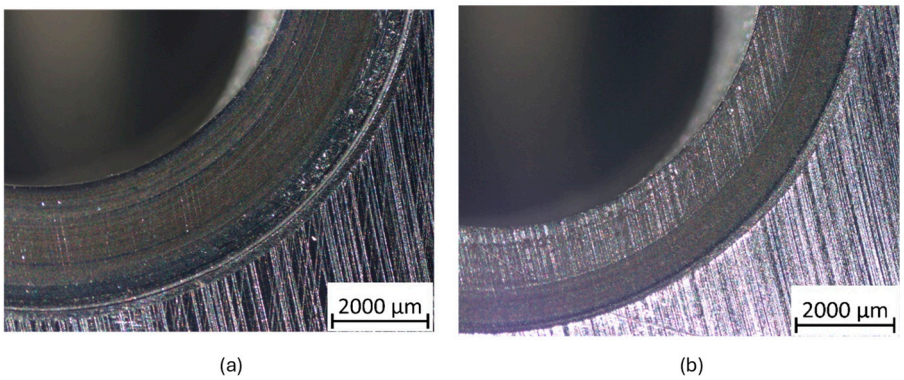


Figure 8. *Cont.*

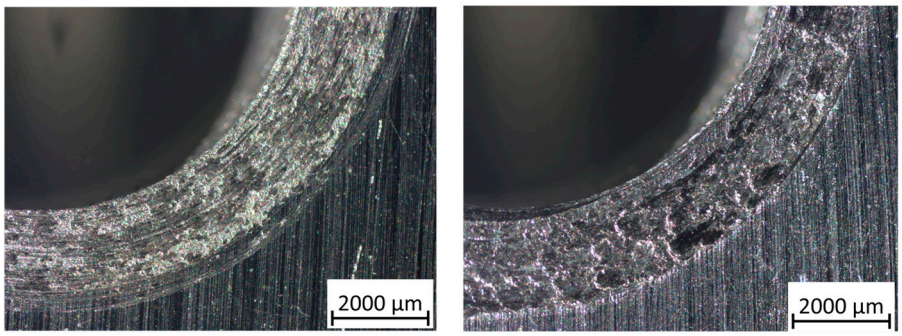


Figure 8. Microscopic observation of bearing plate surface after testing with VG46 against; St bolts at v_t of (**a**) 10 RPM and (**b**) at 250 RPM, and Ti bolts at v_t of (**c**) 10 RPM and (**d**) at 250 RPM.

3.2.2. Fractionated Coconut Oil

The evolution of the average friction coefficients of St screws using FCO as a function of the number of tightenings is shown in Figure 9. The coefficients of friction for tightening up to 30% of the final preload are displayed in Figure 9a,b for tightening rotational speeds of 10 and 250 RPM, respectively. A relatively high coefficient (0.15) is evident at the initial stage, which stabilizes at nearly 0.1 after six to seven number of tightenings at the lower tightening speed and four to five tightenings at the higher tightening speed. This phenomenon could be attributed to the removal of predominant peaks of the surface roughness following a specific number of tightenings. Therefore, the lubricant's effect becomes more pronounced after a specific number of tightenings, leading to a decrease in the friction coefficient and its subsequent stabilization. Moreover, at higher rotational speeds, stability is achieved earlier, which can be attributed to the accelerated removal of the asperities or the surface roughness peaks due to the increased forces at high speeds.

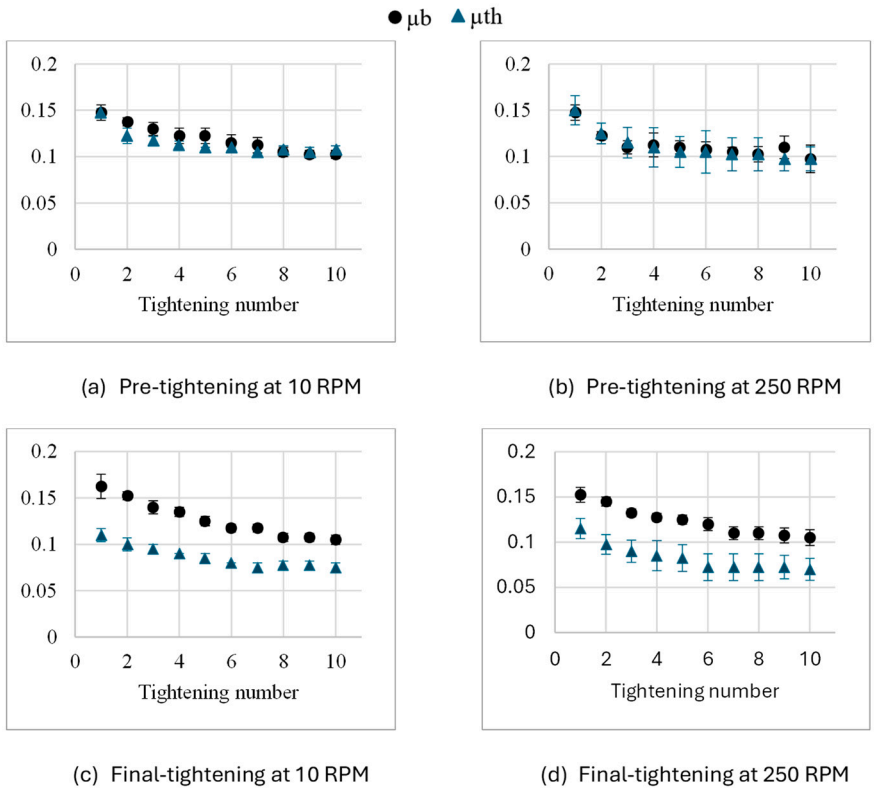


Figure 9. Friction coefficients during different tightenings for St FCO.

The findings of Croccolo et al. [35] on St bolts lubricated with FCO demonstrate enhanced stability in friction coefficients. This may be attributed to the higher underhead pressure attained in the present study, corresponding to the preload force of approximately 44.5 kN, as compared to the preload force of 24.7 kN employed by Croccolo et al. [35]. This elevated pressure may result in the abrupt expulsion of the lubricant from the bearing region. However, the friction coefficients remain minimal, thereby validating the chemical affinity of the FCO with the St screws.

Furthermore, it is observed that at both speeds, the friction coefficient at the initial tightening stage is approximately equal. At both tightening speeds, the lubricant sets on the screws and plates, suggesting a heightened lubricant effect on the friction coefficient.

In the final tightening, the trend of friction coefficient is approximately the same as observed in the case of VG46. This similarity can be attributed to the fact that the final tightening stage always takes place at a lower tightening speed.

The friction coefficients of Ti screws when lubricated with FCO are presented in Figure 10. The underhead friction coefficients are once again notably higher relative to the friction coefficients of the steel screws. The average friction coefficients range from 0.48 to 0.55 during pre-tightening and 0.40 to 0.60 in the final tightening. This phenomenon may be attributed to the removal of VG46 during the tightening cycle, resulting from the slippery surface of Ti. This phenomenon, known as the limit lubrication condition, gives elevated friction coefficients, especially due to the interaction of dissimilar materials. Analogous to VG46, the thread friction coefficients approximate those of steel, suggesting that the lubricant remains interlocked between the thread surfaces.

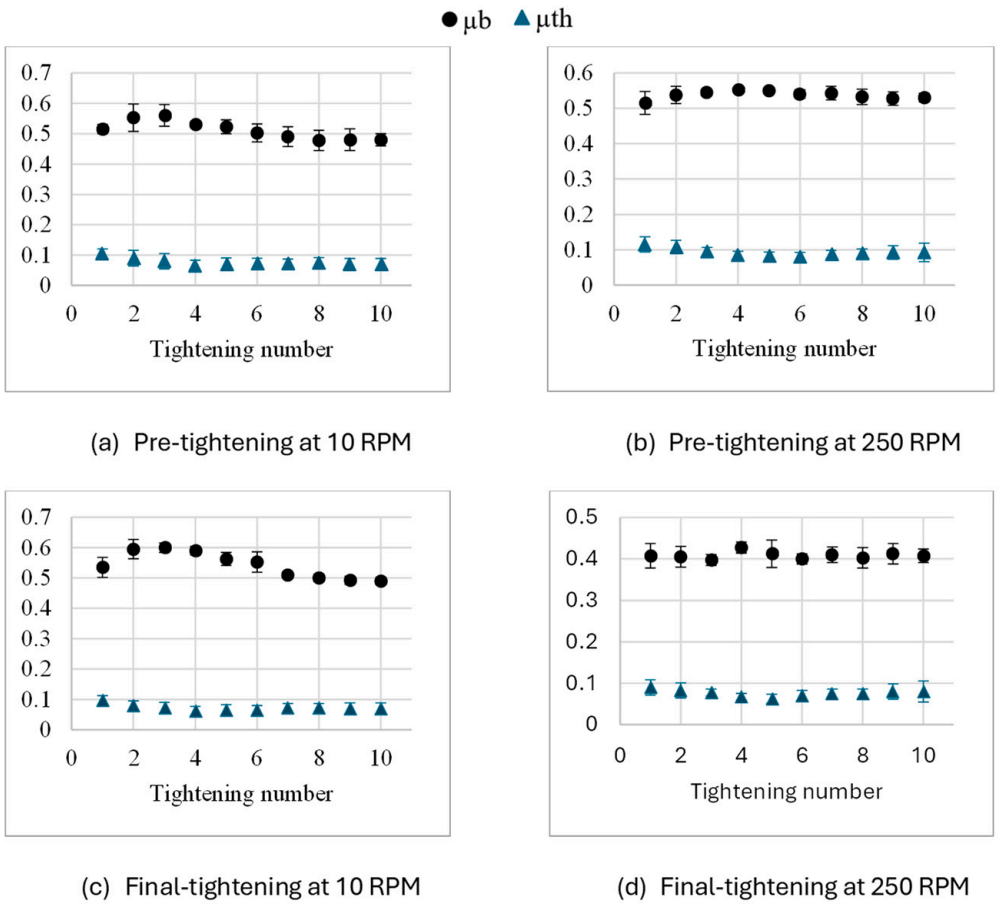


Figure 10. Friction coefficients during different tightenings for Ti FCO.

Moreover, the initial tightening stage exhibits elevated friction coefficients, which may be attributed to the pronounced resistance exhibited by the asperities cycle's commencement. Furthermore, at higher rotational speed, the lubricant's effectiveness is enhanced, as it does not have enough time to remove, similar to VG46, and gets adsorped on the surface, during the final stages of high-speed tightening.

The microscopic observation of plates after the test is shown in Figure 11. Evidently, the plates' surfaces beneath the underhead surfaces of steel screws remain smooth, attributable to the low friction coefficients. However, plates' surfaces beneath the underhead surfaces of Ti experience material loss due to the harder surface of Ti making contact with the asperities of the Steel plate at the micro-level, resulting in a rougher surface and higher friction coefficients at the macro scale.

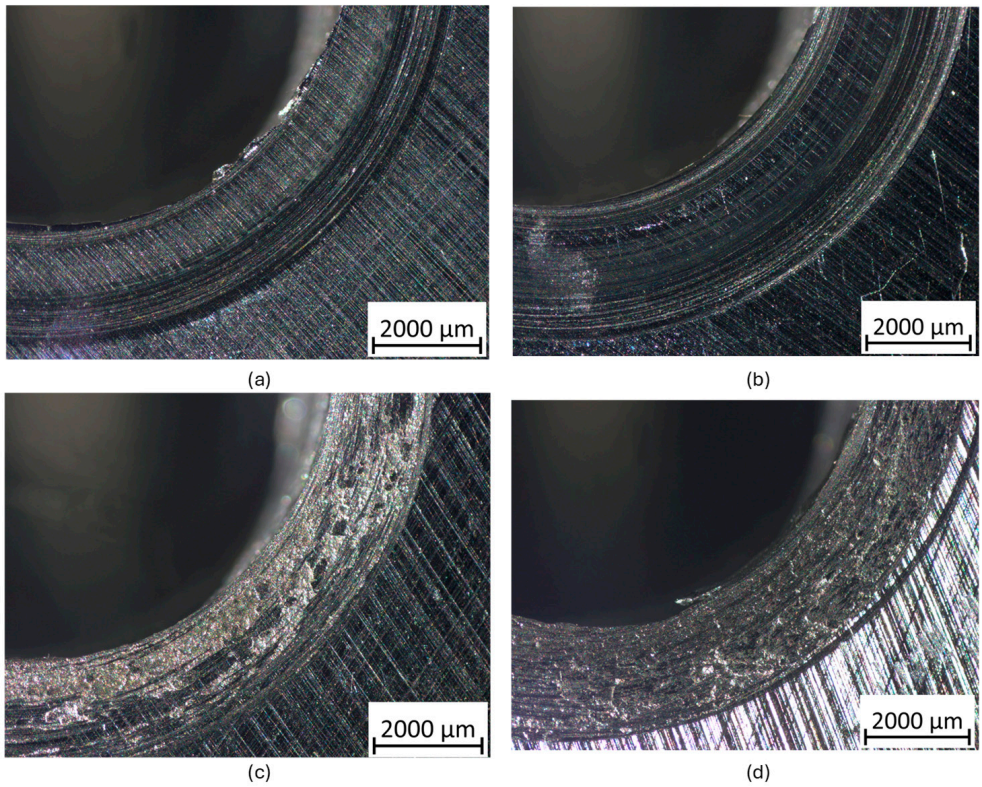


Figure 11. Microscopic observation of bearing plate surface after testing with Coconut oil against; St bolts at v_t of (**a**) 10 RPM and (**b**) at 250 RPM, and Ti bolts at v_t of (**c**) 10 RPM and (**d**) at 250 RPM.

It is noteworthy that, at 250 RPM, the applied torque is 60 Nm; however, due to the inertial effect, the actual torque output exceeds 80 Nm. This results in a preload force (~19.5 kN max) that exceeds the preload achieved by tightening at 10 RPM (~16.6 kN max). This phenomenon is demonstrated in the actual tightening cycle in Figure 12. This is a critical factor to be considered when mounting screws using automatic procedures in practical scenarios.

3.2.3. Castor Oil

The average friction coefficients of St screws using CSO lubricant with the number of tightenings are shown in Figure 13. Coefficients of friction for tightening up to 30% of the final preload are illustrated in Figures 13a and 13b, considering tightening rotational speeds of 10 and 250 RPM, respectively. This is evident from relatively high coefficients (0.15) observed in the initial stage, which attain stability after approximately five cycles of tightening at both lower and higher tightening speeds. This phenomenon can be attributed to the time-dependent nature of CSO adhesion, due to its high viscosity. Therefore, the

effect of lubricant became more pronounced after a specific number of tightenings, leading to a decrease in the friction coefficient and its subsequent stabilization. Moreover, at both rotational speeds, stability is achieved after a similar number of tightenings (n_t), indicating that the lubricant is retained during the tightening process. Instead, this phenomenon can be attributed to the presence of an inherent lubricant layer that is naturally formed between the underhead surface of the screw and the plate. However, as Croccolo et al. demonstrate, higher friction coefficients are exhibited at lower tightening speeds and lower ones at higher tightening speeds [35]. This phenomenon can again be attributed to the higher underhead pressure, which is developed due to the high final preload force in the present study. This results in pronounced compression of the lubricant. It has also been observed that, given its high viscosity, CSO exhibits enhanced performance in terms of friction coefficients.

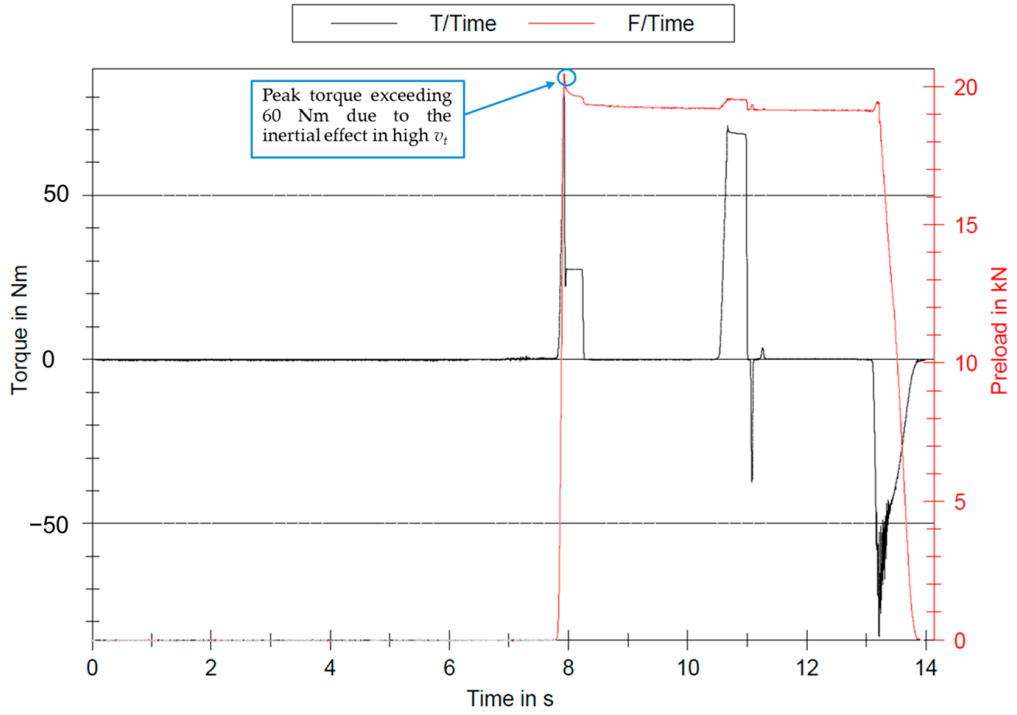


Figure 12. Test cycle for Ti 250 RPM with Final torque 60 Nm.

Furthermore, it is observed that at both speeds, the friction coefficient at the initial tightening is approximately equal. At both tightening speeds, the lubricant takes time to set on the screws and plates, so its effect on the friction coefficient is pronounced after a number of initial tightening cycles.

In the final tightening, the trends of friction coefficient for both tightening speeds are approximately the same as observed for VG46 and FCO. This observation underscores the notion that the final tightening stage invariably occurs at a lower tightening speed compared to the initial stages. Consequently, the friction coefficient at the final stage remains indistinguishable between the two tightening speeds.

Furthermore, the friction coefficients of the Ti screws using CSO as lubricants are presented in Figure 14. The underhead friction coefficients for CSO are notably higher initially ($\mu_{b,\,final} \approx 0.35$) than those for steel screws. However, it is observed that the friction coefficient undergoes a substantial reduction after a certain number of tightenings, attaining a level comparable to that of steel screws ($\mu_{b,\,final} \approx 0.20$). Furthermore, the trend of friction coefficient demonstrates stability and repeatability after four to five cycles of tightening. This trend can be attributed to the high viscosity of the CSO. Due to high viscosity, the

lubricant does not remove from the Ti screw, thereby maintaining its effectiveness in reducing the friction coefficient of the Ti screws. The elevated initial values of the friction coefficient indicate that the CSO requires a duration to set on the Ti screw. As it takes a prolonged duration for complete diffusion into the asperities of the Ti screw, the values are elevated initially. However, once it has been set, the friction coefficient values are reduced.

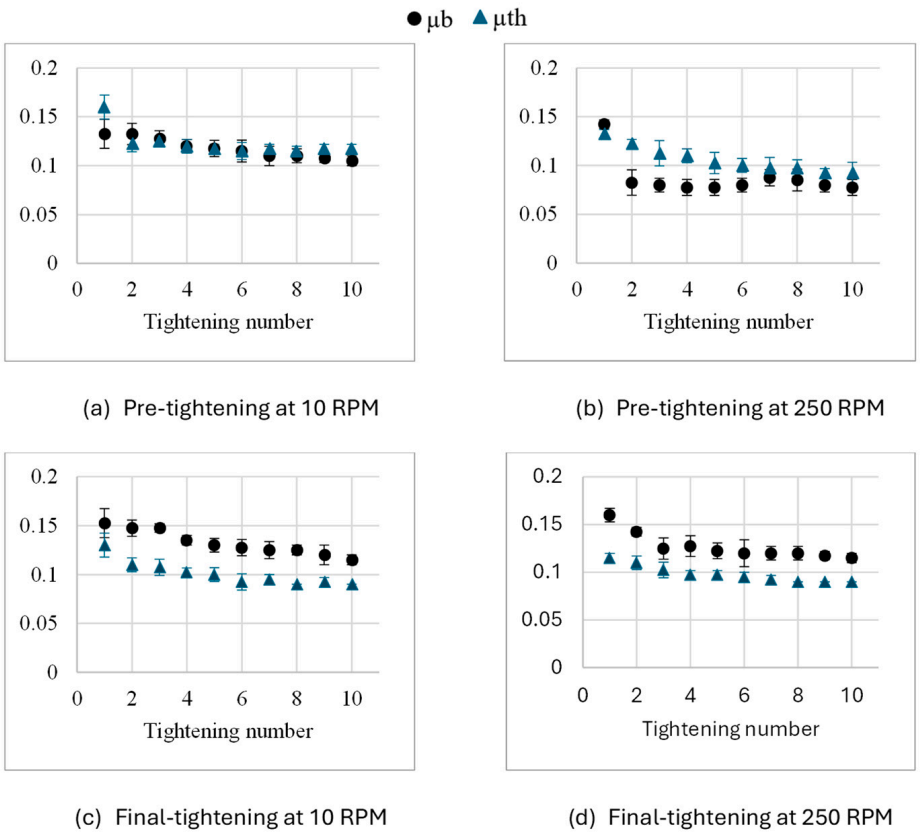


Figure 13. Friction coefficients during different tightenings for St CSO.

Furthermore, at higher rotational speed, even small amounts of lubricant do not have sufficient time to strip away and instead, it gets adsorbed on the surface, resulting in lower friction coefficients during the final tightening stage of the high-speed tightening cycle. The presence of lubricant, even in small quantities, can potentially strip off the screw, resulting in a slightly elevated friction coefficient at the lower tightening speeds.

The microscopic observation of plates after the test is shown in Figure 15. It is evident that the plates's surface beneath the underhead surface of the steel screws remain smooth, attributable to the low friction coefficients. In contrast to the other two lubricants, the plates' surface beneath the underhead surfaces of Ti screws are smooth, due to the presence of the lubricant film at the micro-level, which prevents asperities from coming into contact and damaging the surfaces at the macro level.

For a notable observation at 250 RPM, the final torque applied is 60 Nm, and it remains constant at the same torque unlike the other two lubricants tested. The phenomenon can be attributed to the high viscosity of CSO, which hinders the propagation of external forces due to inertia, thereby constraining the torque to the applied level. This phenomenon is demonstrated in the actual tightening cycle in Figure 16. This is a salient advantageous factor to be considered when mounting screws using automatic procedures in practical scenarios.

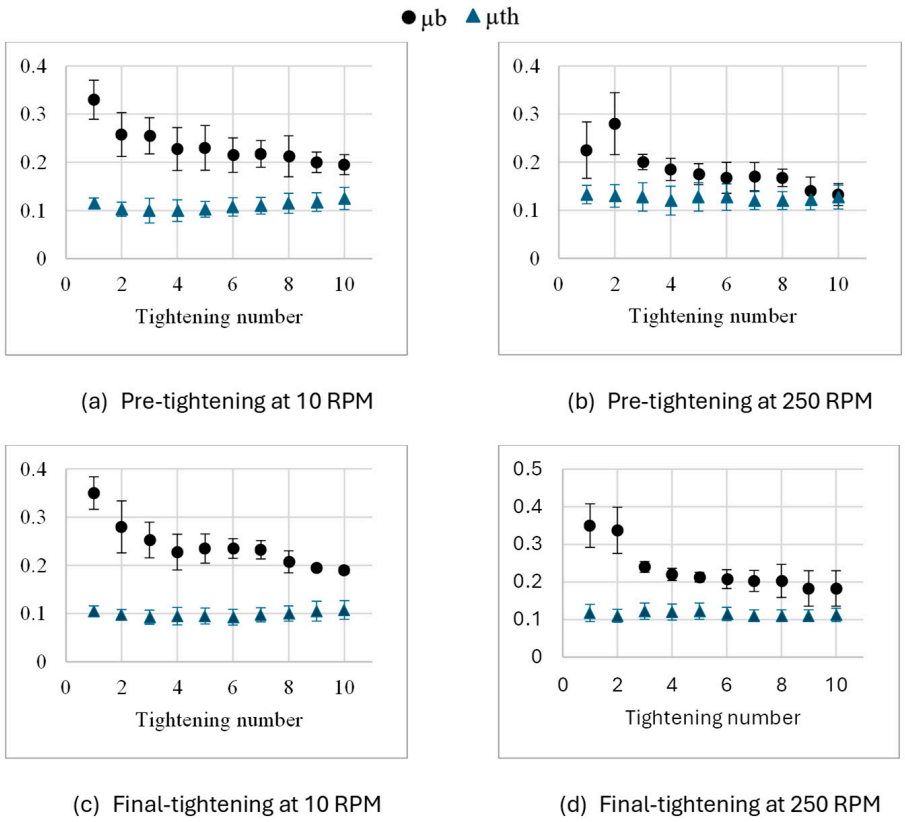


Figure 14. Friction coefficients during different tightenings for Ti CSO.

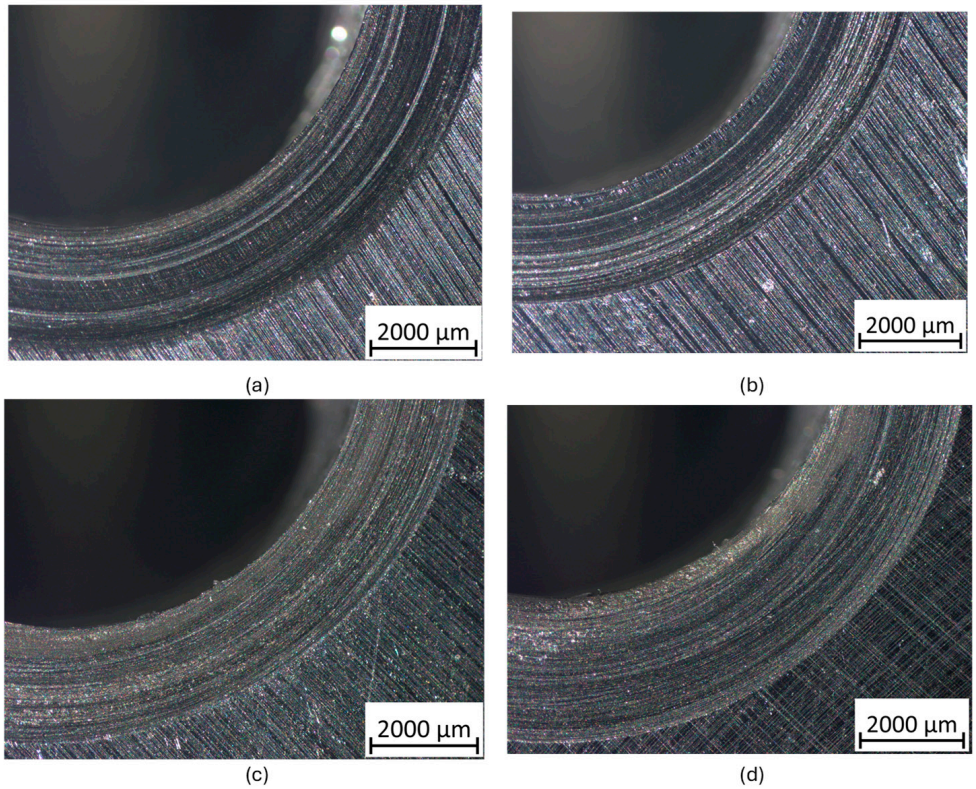


Figure 15. Microscopic observation of bearing plate surface after testing with Castor oil against; St bolts at v_t of (**a**) 10 RPM and (**b**) at 250 RPM, and Ti bolts at v_t of (**c**) 10 RPM and (**d**) at 250 RPM.

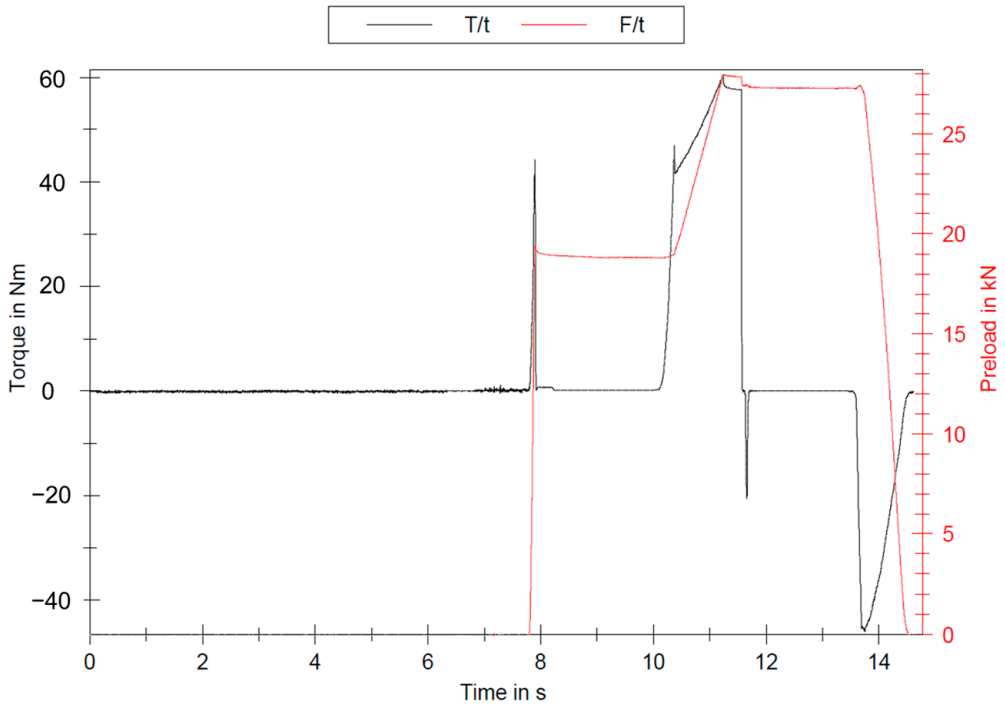
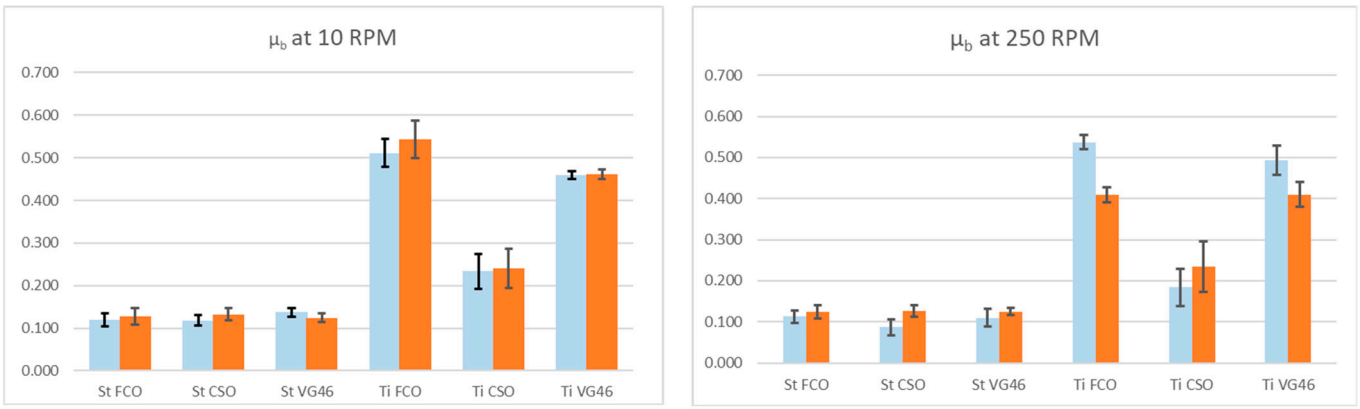


Figure 16. Tightening test cycle of Ti screw with CSO as lubricant at v_t = 250 RPM.

3.2.4. Comparison

A comparison of average values of friction coefficients at the underhead is given in Figure 17a for a low tightening speed of 10 RPM and in Figure 17b for a fast tightening speed of 250 RPM for the different lubricants used.



(a) (b)

Figure 17. Average values of the bearing coefficients of friction (μb), where light blue bars illustrate friction coefficients at the initial tightening stage while orange bars illustrate friction coefficients at the final tightening stage.

The friction coefficients at low v_t demonstrate that Ti screws exhibit significantly high friction coefficients compared to steel screws. In particular, VG46 Ti screws exhibited approximately 400% higher μ_b, while FCO Ti screws displayed approximately 275% higher friction coefficients. However, using CSO as a lubricant results in only 108% higher friction coefficient of Ti compared to the FCO and VG46. Compared to the other lubricants used, CSO demonstrated notable compatibility with the Ti screws. Exhibiting approximately 130% and 110% lower average friction coefficients compared to FCO and VG46, respectively, CSO

demonstrated its optimal suitability to be used for Ti screws. A similar trend is observed for the high v_t, with the test lubricants demonstrating a comparable pattern to that of the low v_t. For the Ti screws, the highest friction coefficients are demonstrated by the VG46, followed by FCO, while the lowest friction coefficients are exhibited by CSO. In the case of St screws, the friction coefficients exhibit negligible differences among the three lubricants when compared with the differences observed in Ti screws.

The dependency of $\mu_{b,\,pre}$ on the tightening speed is observed to be significant for both Steel and Ti screws with CSO. With increasing tightening speed, the friction coefficients decrease due to the heightened dependency of CSO viscosity on the temperature between the interface, as evidenced by the viscosity results presented in Section 3.1. However, for $\mu_{b,\,final}$, the trend is similar for both the tightening speeds. As discussed previously, stability is achieved after approximately five retightening up to the final tightening in the present test. Furthermore, the discrepancy between the applied torque through the spindle and the actual torque measured (exceeded due to inertia) is negligible in the case of CSO unlike the other two lubricants tested. Consequently, it can be an excellent substitute for the commercial lubricants used in the industry.

To study the effects of DOE parameters, a statistical Analysis Of Variance (ANOVA) was performed on the test results, investigating whether tightening speed (v_t), tightening number (n_t), or their interaction ($v_t \times n_t$) has a significant influence on the coefficients of friction measured during the different retightenings. The *p*-values for $\mu_{b,\,final}$ for the aforementioned factors are provided in Table 3.

Table 3. ANOVA *p*-values showing the influence of tightening speed, number of tightenings, and their interaction for $\mu_{b,\,final}$.

Lubrication	*p*-Value of v_t	*p*-Value of n_t	*p*-Value of $n_t \times v_t$
Ti VG46	$\mathbf{<1 \times 10^{-5}}$	0.8181	0.5994
Ti FCO	$\mathbf{<1 \times 10^{-5}}$	$\mathbf{<1 \times 10^{-5}}$	$\mathbf{<1 \times 10^{-5}}$
Ti CSO	0.4588	$\mathbf{<1 \times 10^{-5}}$	0.6701
St VG46	0.8951	$\mathbf{<1 \times 10^{-5}}$	0.9619
St FCO	**0.0404**	$\mathbf{<1 \times 10^{-5}}$	0.5689
St CSO	**0.0135**	$\mathbf{<1 \times 10^{-5}}$	0.3035

Values in bold show the *p*-values less than 0.05.

The significance of the factors is indicated by the *p*-values less than 0.05 [43]. For the measured $\mu_{b,\,final}$, all the tested screws are significantly affected by the number of tightenings (n_t), with the exception of the Ti screws with VG46. This effect is further elucidated in Figure 7c,d, which demonstrate approximately stable friction coefficients in this instance. This outcome offers insight into the stability of Ti bolts with VG46. However, as illustrated in Figure 7, the values of friction coefficients are sufficiently high to preclude their practical application. The investigation further explores the impact of tightening speed (v_t) of Ti bolts with CSO and St bolts with VG46, revealing an insignificant dependency. For Ti bolts with CSO, the same friction coefficients can be attributed to the retention of the correct layer height of the lubricant due to the high pressure resulting from high preload force.

Interestingly, the friction coefficients of St bolts using FCO demonstrate a substantial dependency on both n_t and v_t, which is opposite to the trend reported in the literature using S235JR steel plates [35]. This can possibly be due to the increased pressure due to the higher applied preload which suggests that VG46 is forced out of the bearing region.

The interaction between tightening speed and the number of tightening cycles significantly impacts the FCO-lubricated Ti bolts, further hindering their practical application. Furthermore, Ti bolts with CSO demonstrate negligible dependence on v_t as well as on the

interaction $v_t \times n_t$. However, a significant dependency was observed only on n_t, which also attained stability after a certain number of tightenings, as depicted in Figure 14. This finding underscores the efficacy of CSO as a lubricant for Ti bolts, making it a suitable substitute compared to the commercially used VG46 lubricant.

A comparison of the friction coefficients in Figure 17 and the viscosity of oils presented in Table 1 suggests that the stability to different tightening speeds is inversely proportional to the viscosity of the oil in the case of Ti. However, it is imperative to note that further research is necessary to fully elucidate this relationship.

4. Conclusions

This study provided a novel analysis of the effect of bio-based lubricants on the tribological performances of Ti screws in the tightening phase. A comparison with steel screws of the same grade was also presented. Overall, the results indicate significant differences in the performances of vegetable oils, contingent on the composition of the screw material. Particularly, the main findings can be summarized as follows:

1. The performance of FCO is characterized by higher coefficients of friction in comparison to VG46. Moreover, the influence of the number of tightenings on Ti screws is also significant for FCO, unlike VG46, as demonstrated by ANOVA. This suggests that VG46 provides enhanced stability over the number of tightenings. Despite its enhanced stability, VG46 exhibits remarkably elevated friction coefficients, resulting in significant surface wear, thereby hindering the practical application of lubricants in real-world scenarios;
2. In contrast, CSO not only serves as an effective substitute but also demonstrates superior performance to the commercial lubricant, VG46. Particularly, it would be useful for high-loaded threaded joints, as it yields coefficients of friction that are considerably lower than those of VG46;
3. The repeatability of Castor oil was observed to stabilize after approximately five retightenings in the present study. This stability may be attributed to the settling of the lubricant within the material or a change in the chemical composition of the oil upon heating, which requires further investigation. In an industrial context, this behavior necessitates pre-tightening cycles in line before delivering the parts;
4. A comparison with previous studies indicates that the performances of screws lubricated with vegetable oil significantly vary with the final load and the hardness of the bearing plate.

Future work can focus on studying the change in properties of CSO during the retightenings due to high temperature and pressure. Moreover, efforts must be made to investigate other non-edible oils to prevent any conflict with the shortages of food.

Author Contributions: D.C.: writing—review and editing, supervision, formal analysis, funding acquisition, conceptualization. M.D.A.: writing—review and editing, validation, funding acquisition, data curation, conceptualization. S.F.: supervision, investigation, methodology, conceptualization, data curation, formal analysis. M.M.: writing—review and editing, supervision, validation, investigation, methodology, conceptualization, data curation, formal analysis, funding acquisition. G.O.: writing—review and editing, data curation, formal analysis. C.S.: investigation, methodology, conceptualization, data curation. M.Y.K.: writing—review and editing, investigation, data curation. M.H.B.T.: writing—original draft, review and editing, investigation, data curation. All authors have read and agreed to the published version of the manuscript.

Funding: Financed by the European Union—NextGenerationEU (National Sustainable Mobility Center CN00000023, Italian Ministry of University and Research Decree n. 1033—17/06/2022, Spoke 11—Innovative Materials and Lightweighting). The opinions expressed are those of the authors only

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Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data can be provided by the authors upon request.

Acknowledgments: The authors would like to thank Poggipolini S.p.A. for supporting the present work.

Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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