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Friction performance of palm oil-based trimethylolpropane ester containing bacterial cellulose and sorbitan monostearate: A Taguchi-based study

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Abstract: Growing environmental concerns have prompted researchers to investigate renewable alternatives to mineral oils. Palm oil is a promising vegetable oil for use as a lubricant base stock due to its renewability, biodegradability, low environmental toxicity, and high viscosity. In this study, palm oil was chemically modified by a two-step esterification/transesterification process with trimethylolpropane (TMP) to yield palm oil-based TMP ester (PTMPE) as a base oil. Bacterial cellulose (BC) from nata de coco and sorbitan monostearate (Span 60) were incorporated into the base oil at different concentrations as an additive and a surfactant, respectively. Friction analysis was carried out using a four-ball tribotester at an elevated temperature. The Taguchi method was applied to optimize the experiment, with the mass fractions of BC and Span 60, as well as ultrasonication time, as input factors, and the coefficient of friction (COF) as the response. The results showed that the lowest COF was obtained when 0.3 wt% bacterial cellulose and 0.9 wt% Span 60 were added to the PTMPE ester and ultrasonicated for 10 minutes. In contrast to other studies that concentrated on water- or synthetic-based carriers, the friction-reducing performance of bacterial cellulose in PTMPE was investigated, resulting in a 16.45% decrease in COF under a 392 N load. Analysis of variance (ANOVA) showed that none of the investigated parameters were statistically significant in explaining variations in the coefficient of friction. However, the ultrasonication time had the largest estimated contribution among the investigated factors, accounting for almost 50%.

Keywords: palm oil; biolubricant; bacterial cellulose; coefficient of friction

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1. Introduction

Friction and surface abrasion in mechanical contacts, such as piston ring-cylinder liner interfaces, result in significant material deterioration [1], [2]. A lubrication system is one of the most effective methods of reducing friction and wear in moving mechanical contacts [3], [4]. Although mineral oil-derived lubricants significantly reduce these losses, their non-renewable nature, limited biodegradability, and substantial risk of environmental harm render them ecologically unsustainable. Therefore, it is imperative to develop high-performance, sustainable biolubricants from renewable vegetable oils to reduce energy dissipation and petroleum-related environmental pollution.

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The lubricant's viscosity and relative motion of contacting surfaces contribute to the formation of a protective film. Fluid lubricants, the most commonly used in machinery applications, consist of base stocks (over 85%) and various additives, including anti-friction, anti-wear, and extreme-pressure (EP) additives, antioxidants, and friction modifiers. Additives are mixed with the base stocks to enhance the performance and efficiency of the lubricants [5]. Generally, commercial fluid lubricants used in automotive engines and the manufacturing industry are based on mineral oil due to its current availability and lower production costs. Between 85 and 90 % of industrial lubricants, including compressor, hydraulic, and transmission oils, are derived from mineral oils [6], [7]. However, the availability of mineral oil cannot be sustained indefinitely because its base stock is non-renewable. Furthermore, the disposal of mineral oil-based lubricants contributes to environmental pollution, as the petroleum-based base stock is non-biodegradable. Additionally, hydrocarbon-based lubricants may leak or spill into marine environments, resulting in ecological harm to mangrove biota, larvae, corals, and fish [8]. These points have forced researchers to develop sustainable, environmentally friendly lubricant formulations. Biolubricants, or bio-based lubricants, are emerging and gaining global acceptance as potential substitutes for mineral oil lubricants, as their feedstocks are derived from bio-based oils and animal fats. Vegetable oils are the most favorable choices for base stocks of biolubricants [9], [10], [11], [12]. They have been experimentally shown to exhibit high viscosity and viscosity index, as well as high flash and pour points [13]. Additionally, vegetable oils have high molecular weights in their triglyceride structures, resulting in very low volatility [14]. High lubricant volatility increases lubricant consumption, and the loss can change its effectiveness. Vegetable oils are also perceived as renewable, biodegradable, and less toxic than mineral oils [15].

However, vegetable oil-based lubricants are not yet widely adopted due to several factors, including their low oxidative stability and poor low-temperature performance [16], [17]. The tribological properties of biolubricants are also not as good as commercial mineral oil-based lubricants [18]. These shortcomings can be tackled by using chemical modification processes (e.g., esterification and transesterification, hydrolysis/estolide formation, epoxidation, and ring-opening after epoxidation) and the incorporation of additives. Esterification and transesterification involve rearranging the acyl moieties (C=O) to form new triesters from the triglycerides in vegetable oils [18], [19]. In transesterification, vegetable oil reacts with a polyol alcohol to produce a synthetic ester. Trimethylolpropane (TMP), neopentyl glycol (NPG), and pentaerythritol (PE) are the most common polyol alcohols used in making the base stock of biolubricants. Many researchers have studied the tribological performance of palm oil-based TMP ester, with promising results [18], [20].

Moreover, the additivation process improves the tribological properties of biolubricants, including the coefficient of friction (COF) and wear rate [7], [16], [21], [22]. The most commonly used additives in commercial lubricants contain heavy metals and other harmful components that adversely affect the environment [23], [24]. Therefore, biolubricants should be developed with eco-friendly additives.

Currently, nanomaterials have emerged as lubricant additives, replacing conventional ones [25]. Cellulose nanomaterials have been experimentally investigated as lubricant additives. The physicochemical properties of plant-based cellulose nanocrystals (CNCs) derived from Pennisetum purpureum fibers as a lubricant additive in a commercial SAE 40 engine oil have been examined [26]. It was reported that CNC slightly increased the viscosity index compared with SAE 40. Furthermore, the plant-based CNC from acetate-grade dissolving pulp has been investigated by adding this cellulose to SAE 40 engine oil [21]. The result revealed that SAE 40 containing 0.1% CNC improved the tribological properties of the nanolubricant, lowering the COF and increasing the wear resistance. However, nanocellulose is produced not only by plants but also by bacteria, commonly referred to as bacterial cellulose (BC), including from the production of nata de coco using the ultrasonication process [27]. Indeed, this cellulose has been investigated as a lubricant

additive by blending it with a lubricant base stock, such as polyol ester oil (POE), to improve the tribological properties of contacting surfaces [28].

Bacterial cellulose is a hydrophilic substance. It will agglomerate upon mixing with the lubricant base oil, thereby reducing the biolubricant's dispersion stability. This can be addressed by dispersing 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) and a surfactant, such as sorbitan monostearate (Span 60), into the base oil. In this case, the surfactant bonds to the additive, reducing its surface tension and preventing aggregation [28].

Chemical modification processes for palm oil-based biolubricants have been reported by numerous authors, including double-step esterification, epoxidation, and ring-opening after epoxidation. On the other hand, research on the use of environmentally friendly nanomaterials as additives has been ongoing for the past decade. Previous studies investigating BC as a lubricant additive have primarily focused on simple water-based systems and on mineral or synthetic base oils [28], [29], [30]. Additionally, those prior studies used a full factorial design to prepare the biolubricant. Consequently, a research gap remains concerning the effects of BC addition on the tribological properties of chemically modified palm oil and the optimization of these formulations using the Taguchi method. In this research, BC from nata de coco was used as a lubricant additive, and Span 60 as a surfactant, both of which were applied to a palm oil-based TMP ester base oil to produce a biolubricant. This study employed the Taguchi method to investigate the effect of BC and Span 60 mass fractions, as well as ultrasonication time, on the tribological performance of the produced biolubricant. Optimization methods should be used to minimize the number of experiments required to prepare the biolubricant samples. The Taguchi method is a widely used experimental optimization technique, along with Response Surface Methodology (RSM) and artificial neural networks [31], [32], [33]. Besides, the Taguchi method can optimize combinations of control factors [34].

2. Material and methods

This study incorporated modified bacterial nanocellulose, treated with TEMPO and Span 60, into the palm oil-based TMP ester. The mass percentages of additives and surfactants added to the base oil were selected based on previous studies [28], [35]. The investigation of friction characteristics was conducted using a four-ball tribometer and a commercial lubricant for comparison.

2.1 Biobase oil and additives

Palm oil methyl ester (POME), acquired from a local company in Riau, Indonesia, was synthesized through a transesterification process with a reactant of polyol TMP [2-ethyl-2-(hydroxymethyl)-1, 3-propanediol] and sodium methoxide as a catalyst to produce palm oil-based TMP ester (PTMPE) as a base stock oil in this study. TMP and sodium methoxide were purchased from Sigma-Aldrich (Germany) and Glentham Life Sciences (UK), respectively. The PTMPE base oil was prepared by placing the TMP in a 1.5-L reactor and heating and stirring it with an overhead mechanical stirrer, a reflux condenser, and a temperature-controlled oil bath, following the procedure described by a previous study. Figure 1 shows the transesterification assembly apparatus, which is an original apparatus developed by the authors [35]. Briefly, the procedure for producing PTMPE as the base oil is as follows: First, 200 g of POME was heated and stirred in the reactor bath at 180 rpm and 70 °C for 15 minutes. Then, the temperature was increased to 120 °C, 20 g of TMP was added, and the suspension was stirred for 10 minutes to ensure complete dissolution of TMP in the POME. Furthermore, 0.88 g of sodium methoxide (CH_3NaO) was added to the POME/TMP suspension as a catalyst, and the mixture was continuously heated and stirred at 120 °C and 180 rpm for 2 hours. Finally, the suspension was allowed to cool to room temperature. Then, the liquid was then transferred from the second stage of transesterification into a separatory funnel to separate the

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PTMPE synthetic ester from the glycerol. To remove any remaining glycerol, the separated PTMPE ester was subsequently reheated to 70°C for 40 minutes. Furthermore, the base oil of PTMPE was measured using a ViscoQC 300-L from Anton Paar in accordance with ASTM D-2770, yielding viscosity values of 9.804 and 6.239 cSt at 40 °C and 100 °C, respectively.

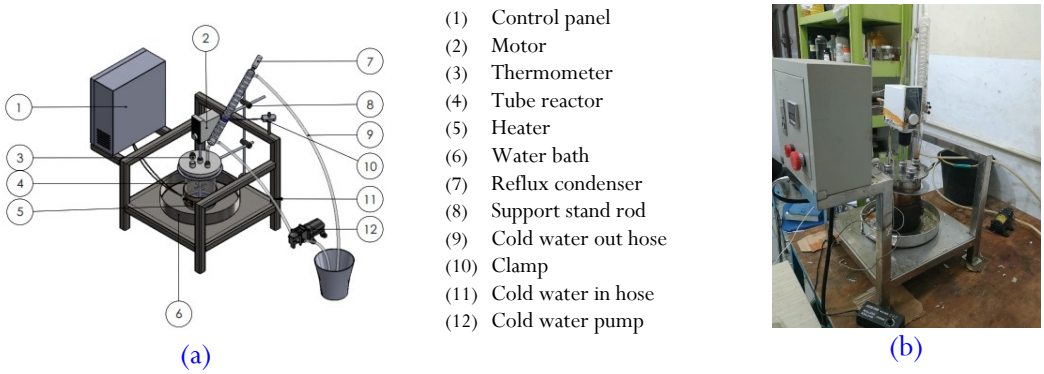

Figure 1. (a) A 3-D schematic diagram and (b) A photograph of the esterification and transesterification process machine

On the other hand, the BC additive used in this research was prepared from a nata de coco pellicle, which was oxidized with TEMPO (98% purity), sodium bromide (NaBr), and sodium hypochlorite (NaClO). The nata de coco pellicle was purchased from a local market in Indonesia; TEMPO and sodium hypochlorite were supplied by Sigma-Aldrich, and sodium bromide was acquired from Merck. The process of additive preparation was similar to the one reported in previous studies [28], [30], [35]. The illustrations of the preparation of the lubricant base oil and BC additive were presented in Figures 2 and 3, respectively [35]. In brief, there were two processes to modify the pellicle BC with TEMPO: (i) purification and powder preparation, and (ii) chemical treatment and filtration. First, wet pellicle BC was soaked in 10% NaOH for 24 hours to remove impurities, then washed with distilled water to neutral pH. Then, the pellicle was blended for 30 minutes, dried at 75 °C for 3 days, and finely sieved through a 100-mesh sieve to obtain a powder. In the second stage of chemical treatment, oxidation was performed by suspending the dried BC powder in water containing TEMPO, NaBr, and NaClO, and stirring the mixture at 350 rpm for 2 hours at room temperature. Finally, the suspension was sieved through a 500-mesh sieve, washed with ethanol to neutral pH, and fibrillated at 12,000 rpm for 30 minutes to form an additive.

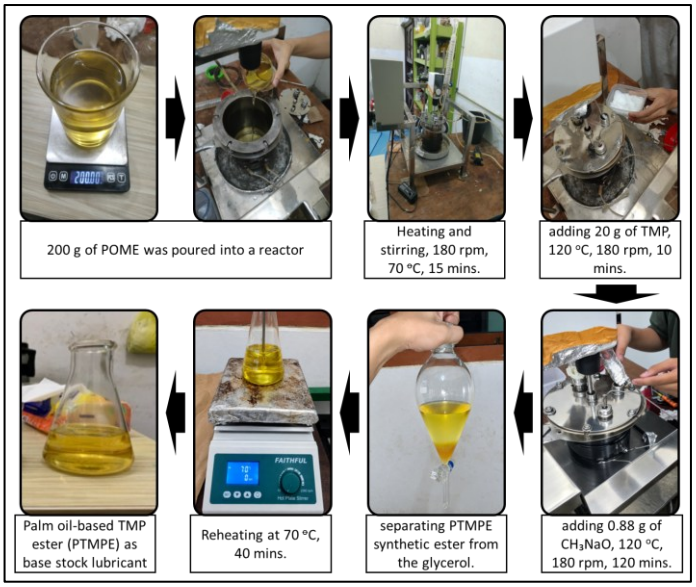

Figure 2. Schematic illustration of base oil preparation through the transesterification process

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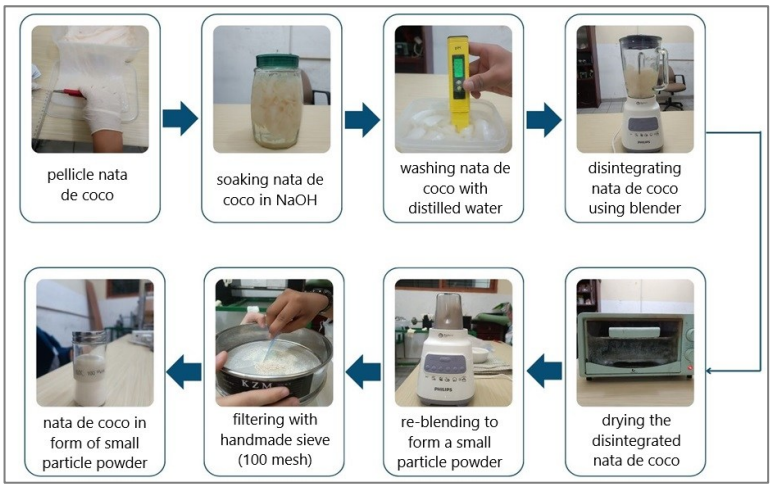

Figure 3. Illustration diagram of nano-additive preparation from wet pellicle nata de coco

2.2 Biolubricant preparation

Biolubricants were formulated by mixing the PTMPE base oil, BC additive, and Span 60 with different mass fractions, as tabulated in Table 1. Span 60 (sorbitan monostearate, CAS No. 1338-41-6, purity GC: 45 – 55 %) was purchased from Sigma-Aldrich as an off-white powder containing stearic acid (18:0), with the remainder consisting primarily of palmitic acid (16:0). To ensure good dispersion of the base oil-additive mixture, Span 60 was used as a surfactant during the manufacture of the biolubricants. Figure 4 illustrates the preparation of biolubricants. Briefly, the surfactant was dissolved in base oil and continuously stirred on a hot-plate magnetic stirrer at 75 °C for 30 minutes. Subsequently, the BC additive was gradually added to the PTMPE/Span 60 suspension, and the suspension was stirred at 110 °C for 120 minutes to evaporate the remaining ethanol until the weight remained constant and the composition reached the exact value shown in Table 1. Regarding dispersion homogenization, the appropriate mixing process and apparatus are also crucial for maintaining the biolubricant's dispersion stability. In this study, the mixing process for each lubricant sample was performed using a high-shear homogenizer (IKA T-25) at 12,000 rpm for 30 minutes, followed by ultrasonic processing with an ultrasonic probe (SX-Sonic Model: FS-1200N, 1200-Watt, 220 V ± 5%, 20 kHz) for 5, 10, and 15 minutes. During the dispersion process, ultrasonication was initiated at room temperature and interrupted when the temperature reached 60 °C. To maintain the device's stability, the probe was set to stop for 10 minutes every 2.5 minutes. Hence, running the sample for 5, 10, and 15 minutes of ultrasonication took a total of 25, 50, and 75 minutes, respectively.

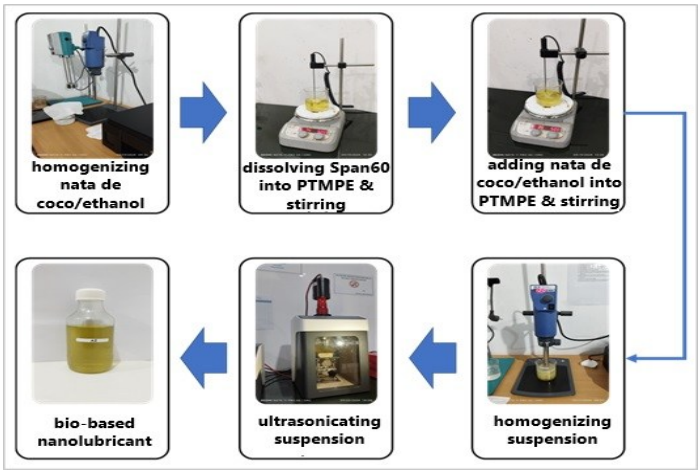

Figure 4. Illustration diagram of the final process for manufacturing the bio-based nanolubricant

Table 1. Mass fraction of PTMPE, BC, and Span 60

Sample No.	Mass fraction (%)		
	PTMPE	Bacterial cellulose	Span 60
A1	99.1	0.3	0.6
A2	98.8	0.3	0.9
A3	98.5	0.3	1.2
A4	98.8	0.6	0.6
A5	98.5	0.6	0.9
A6	98.2	0.6	1.2
A7	98.5	0.9	0.6
A8	98.2	0.9	0.9
A9	97.9	0.9	1.2

2.3 Tribology testing

In this study, the friction of all biolubricant samples was investigated using a four-ball tribometer, a device commonly used by lubricant manufacturers to evaluate the performance of oil lubricants and greases. The test rig consists of three stationary balls, a rotating ball, and the testing lubricant in a lubricant bath, as shown in Figure 5. It was equipped with a thermocouple to control the lubricant temperature and an electric panel to record all experimental data. This experiment used chrome-alloy steel balls, AISI E-52100, 12.7 mm in diameter, with extra-polished (EP) grade 25 and hardened to 64-66 HRC. The balls were cleaned with acetone to remove any residual material before each experiment and were wiped dry with a fresh, lint-free cloth afterward. Each experiment required 10 mL of lubricant to ensure the steel balls were fully immersed and to cover the contacting surfaces. It should be noted that, due to the high cost and limited availability of custom lubricants and instruments, the tribological test for each condition was conducted as a single screening run ($N = 1$). New steel balls were used for each biolubricant sample. The experiment followed ASTM D4172, using the experimental conditions tabulated in Table 2, and the tribotesting was conducted only once for each lubricant sample. The coefficient of friction (COF) value was calculated based on the frictional torque obtained directly from the test rig using Equation 1.

$$COF = \frac{T\sqrt{6}}{3Wr} \tag{1}$$

where T, W, and r represent the frictional torque, the applied load, and the distance from the center of the contact surfaces on the lower balls to the axis of rotation, respectively. In this experiment, the r value was 3.67 mm.

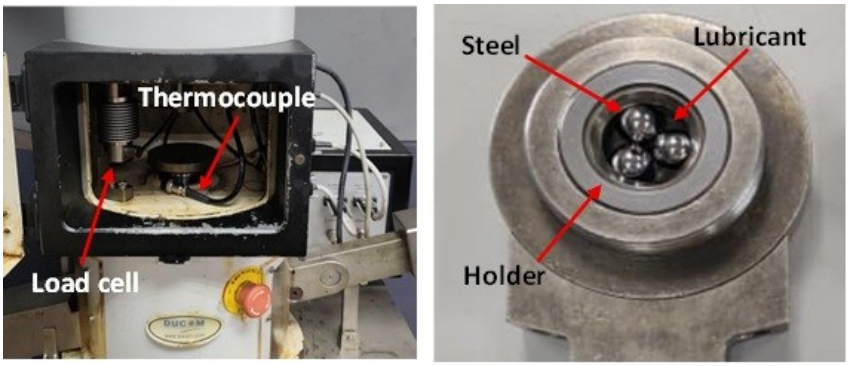

Figure 5. Four-ball Tribotester (left) and the oil rig (right)

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Table 2. Experimental conditions of the four-ball tribometer

Properties	Value
Load	392 N
Rotation Speed	1200 rpm
Duration	60 minutes
Temperature	75 °C

2.4 Design of experiments

The Taguchi method is a statistical tool that enables systematic experimental design while reducing the number of required tests, yielding optimal values. The mass fractions of the additive and surfactant, and the ultrasonication time, were the three input factors considered in this research. Each factor had three levels, as shown in Table 3. Based on three factors and three levels, an L9 orthogonal array was designed to produce biolubricants, resulting in up to 9 samples. The response considered in this study was the coefficient of friction (COF), with a focus on the S/N ratio for the 'smaller-is-better' option, using Equation 2 to determine the minimum COF values. Table 4 presents the Taguchi experimental design.

$$\frac{S}{N}ratio = -10 * log_{10}\left(\frac{1}{n} * y^2\right) \quad (2)$$

where y is the experimental data for COF, and n represents the number of experiments.

Table 3. Design factors with different levels of the biolubricant preparation

Parameters	Unit	Levels		
		1	2	3
wt% BC	%	0.3	0.6	0.9
wt% Span 60	%	0.6	0.9	1.2
Ultrasonication time	Minutes	5	10	15

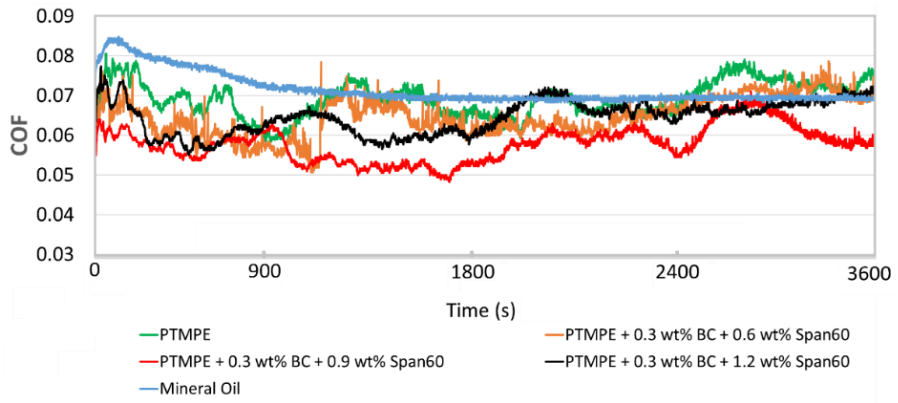
Table 4. Design of experiment L9 orthogonal array

Sample No.	wt% BC (%)	wt% Span 60 (%)	Ultrasonication time (minutes)
A1	0.3	0.6	5
A2	0.3	0.9	10
A3	0.3	1.2	15
A4	0.6	0.6	10
A5	0.6	0.9	15
A6	0.6	1.2	5
A7	0.9	0.6	15
A8	0.9	0.9	5
A9	0.9	1.2	10

3. Results and discussion

3.1 Friction test results

Figure 6 shows the curves of COF values for the designed biolubricant samples of the pure PTMPE and PTMPE filled with the same mass concentration of 0.3 wt% BC at various weight percentages of surfactants (0.6, 0.9, and 1.2 wt% Span 60) and ultrasonication time (5, 10, and 15 minutes) during the whole test duration (a) and the final 600 seconds (b).



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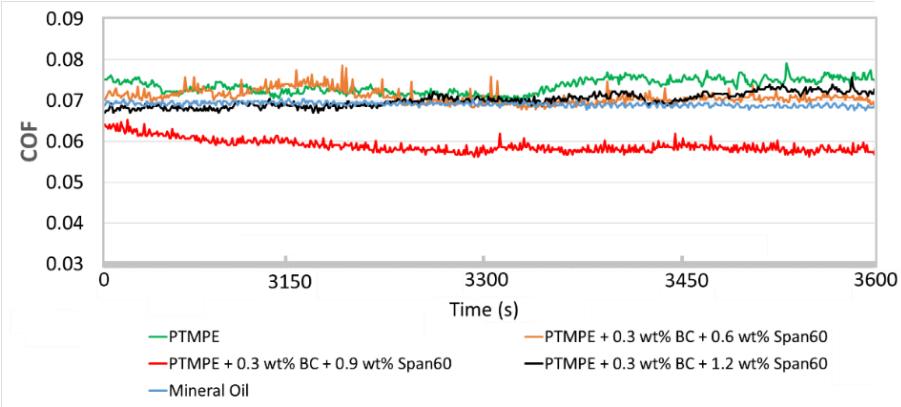

(b)

Figure 6. Graph of COF for pure PTMPE, PTMPE + 0.3 wt% BC + 0.6 wt% Span 60, PTMPE + 0.3 wt% BC + 0.9 wt% Span 60, and PTMPE + 0.3 wt% BC + 1.2 wt% Span 60 during (a) the one-hour test and (b) the final 600 seconds

Generally, the COF values for biolubricant samples initially fluctuated before approaching a steady-state region of each continuous friction trace, i.e., at the final 600 seconds of this experiment. The pressure drop during the test could cause this running-in phenomenon by increasing the contact area throughout the sliding process, as the COF is inversely proportional to the mean contact pressure [36]. Figure 6 also shows that the presence of BC as an additive and Span 60 as a surfactant lowered the COF compared to the PTMPE ester without additives or surfactants. Among the tested biolubricants, sample A2 (PTMPE + 0.3 wt% BC + 0.9 wt% Span 60) exhibited the lowest coefficient of friction. When the pure PTMPE was filled with 0.3 wt% BC and 0.6, 0.9, and 1.2 wt% Span 60, the COF decreased by 5.77, 16.45, and 5.34%, respectively, as seen in Figure 7, which shows the mean COF values for the steady-state region of friction with their standard deviation. This result indicates that sample A2 exhibited the most favorable friction-reducing performance among the tested formulations, consistent with the findings of another study [27], which reported that a suitable base oil, combined with BC and Span 60 as additives, effectively improved its anti-friction properties. Furthermore, bacterial cellulose combined with Span 60 may have contributed to a mending effect by filling surface irregularities. This resulted in a relatively smoother contact interface, thereby reducing friction caused by interlocking surface asperities [28], [30]. However, the COFs of all biolubricant samples with 0.3 wt% bacterial cellulose were lower than those of the commercial mineral oil (COF = 0.0690). This means that, tribologically, these biolubricants could be used as an alternative to mineral oil for reducing the coefficient of friction under the tested conditions. Further testing (e.g., wear resistance, oxidation stability, viscosity, and thermal behavior) is necessary to assess full commercial viability.

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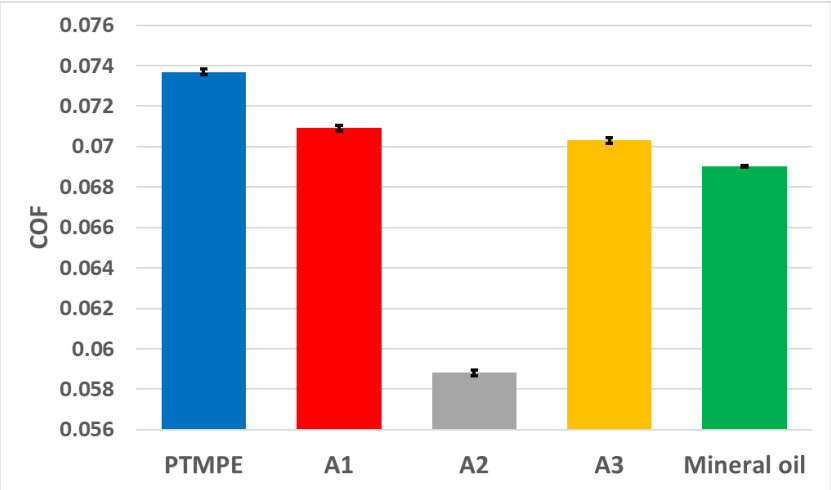

Figure 7. The mean COF for pure PTMPE ester and designed biolubricants with 0.3 wt% BC with 0.6 (A1), 0.9 (A2), and 1.2 (A3) wt% Span 60, calculated from the last 600 seconds of the experiment

It is noted that, due to cost and equipment access limitations, the friction test was conducted once per lubricant sample. However, a single friction run records several hundred continuous steady-state data points; hence, statistical metrics such as the mean and the 95% confidence interval for the standard deviation of the steady-state COF have been extracted from that single run. The computed 95% confidence intervals indicated the steady-state stability of the friction coefficient during each test run, rather than consistency across different runs. To calculate this confidence interval, the sample size, sample mean, and sample standard deviation were first extracted from the COF mean data, and then the standard error of the mean was computed. Then, the two-tailed critical value at the 95% confidence level ($\alpha = 0.05$) was calculated and multiplied by the standard error to get the margin of error. The statistical summary of this steady-state friction test is presented in Table 5. The COF value for sample A2 (0.0588) was substantially lower than those of the other samples, with no overlap in their confidence intervals. Furthermore, mineral oil yielded the tightest confidence interval (0.0000402) due to a significantly lower standard deviation.

Table 5. Statistical summary of the COF values

Metric	PTMPE	A1	A2	A3	Mineral oil
Mean	0.0737	0.0709	0.0588	0.0703	0.0690
95% CI Range	[0.07355, 0.07383]	[0.07078, 0.07104]	[0.05868, 0.05893]	[0.07017, 0.07045]	[0.06900, 0.06908]

For 0.6 and 0.9 wt% BC added to PMTPE, as seen in Figure 8, a similar trend to that of PTMPE filled with 0.3 wt% BC was observed. The COF values for PTMPE mixed with 0.6 wt% BC and 0.9 wt% Span 60 increased after about 2600 s and remained steady over the test. However, the COF values of these sample lubricants were slightly above the COF value of pure PTMPE. This was probably attributed to additive concentration and the additive-to-surfactant ratio in PTMPE [28].

The coefficient of friction for each sample during the last 600 seconds of the experiment is presented in Figure 9. It can be seen that, compared to pure PTMPE ester, the addition of bacterial cellulose as an additive and Span 60 as a surfactant altered the COF values. Generally, formulations containing lower BC concentrations exhibited lower COF values. As seen in Figure 9, the three biolubricant samples containing 0.3 wt% bacterial cellulose had lower COF values than those containing 0.6 wt% or 0.9 wt%. However, the COF value depended not only on the addition of additives and surfactants but also on the ultrasonication duration, which may influence dispersion

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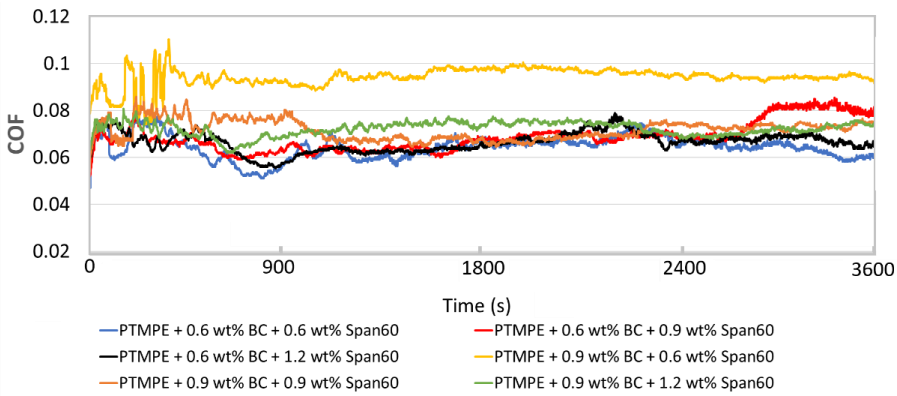
stability. Ultrasonication time significantly improved dispersion stability and maintained bacterial cellulose particle size, thereby preventing agglomeration [37]. The stability of well-dispersed particles between the base oil and additives, as well as the particle size of the additives, more effectively prevented direct metal-to-metal contact and reduced friction [38]. However, the duration of sonication to manufacture biolubricants should be considered. In this study, 10-minute ultrasonication resulted in lower COF values than the other durations. Excessive ultrasonication may promote cellulose fibril aggregation, increasing additive particle size, and causing agglomeration [38].



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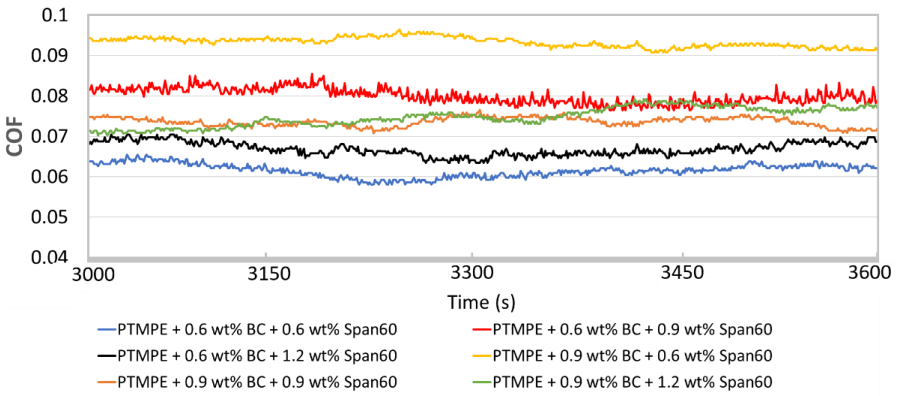

(b)

Figure 8. Graph of COF for PTMPE + 0.6 wt% BC + 0.6 wt% Span 60, PTMPE + 0.6 wt% BC + 0.9 wt% Span 60, and PTMPE + 0.6 wt% BC + 1.2 wt% Span 60 during (a) the one-hour test and (b) the final 600 seconds

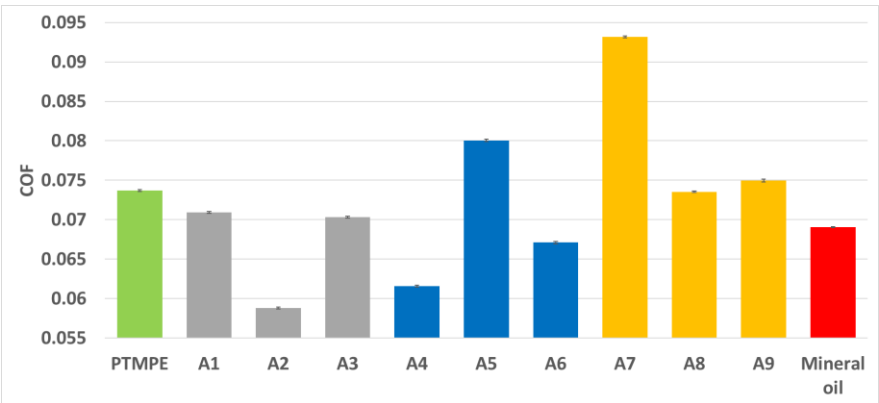

Figure 9. Mean COF for all designed lubricants, calculated from the last 600 seconds of the experiment

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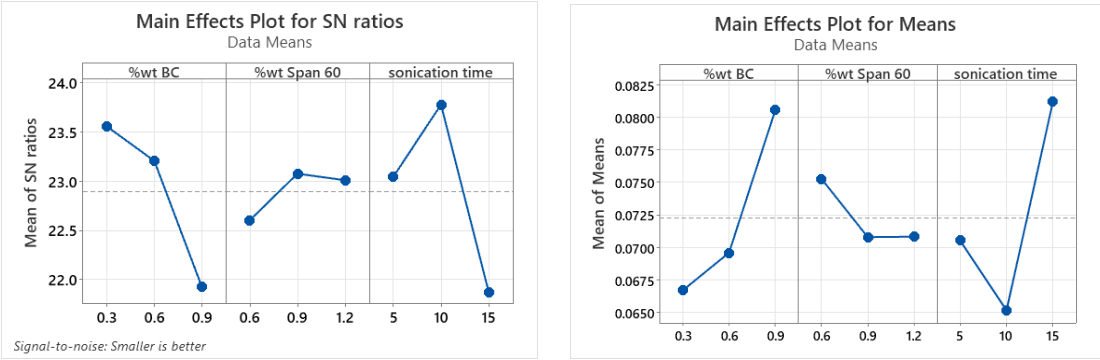
3.2 Signal-to-Noise (S/N) ratio and main effect plots for COF

Figure 10 shows the main-effect plots of signal-to-noise (S/N) ratios (a) and means for the coefficient of friction (b). The main effects plot of the S/N ratio is used to identify which factors have the strongest impact on the coefficient of friction, whereas the main effects plot for means is used to compare the relative strength of the effects of various factors on the COF. It is observed that the COF value generally increased with increasing bacterial cellulose addition and decreased with increasing Span 60 addition and sonication time. The lowest COF was achieved when 0.3 wt% bacterial cellulose and 0.9 wt% Span 60 were added to pure PTMPE ester via ultrasonication for 10 minutes. This was consistent with the experimental COF values tabulated in Table 6 and shown in Figure 9. The predicted COF obtained using the Taguchi method at the optimum values was 0.058033, which was close to the experimentally measured COF value. Table 7 shows the actual and predicted COF values for the optimum parameters. The error was still within the allowed range of 5% [34]. The main effect plot for means also shows a similar trend.



(a) (b)

Figure 10. Main effects plot of S/N ratios and means

Table 6. The value of the response (Coefficient of Friction)

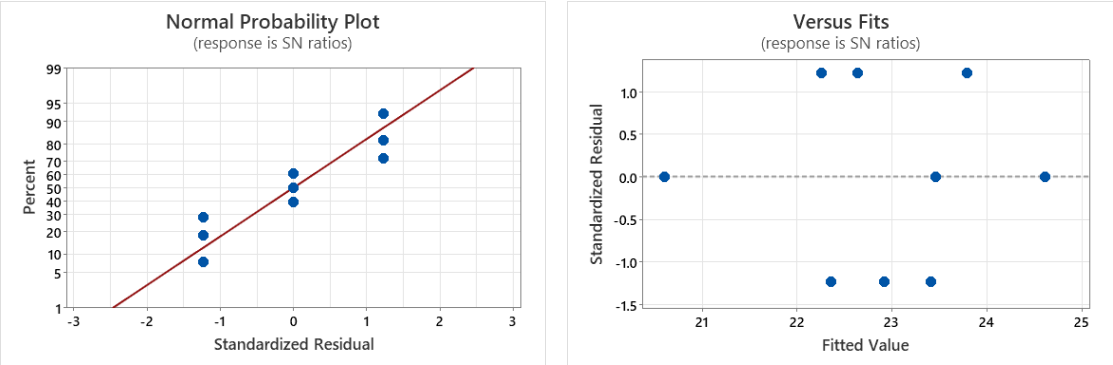
Sample No.	Coefficient of friction
A1	0.0709
A2	0.0588
A3	0.0703
A4	0.0616
A5	0.0800
A6	0.0671
A7	0.0932
A8	0.0735
A9	0.0750

Table 7. Experimental and Taguchi-predicted COF values under the optimal conditions

Optimum parameters		Actual COF	Predicted COF	Error, %
wt% BC	0.3			
wt% Span 60	0.9	0.0588	0.058033	1.31%
Sonication time (min.)	10			

The normal probability plot and residuals-versus-fitted-values plot for COF (response is Means) are shown in Figure 11 (a) and (b), respectively. The residuals were plotted on a normal probability plot (response is Means) to assess whether the model was adequate and the regression assumptions were met. The versus-fits plot, where the response is Means, is used to evaluate the validity of the

statistical model and its key assumptions, i.e., constant variance and linearity. The X-Axis in the versus-fit plot represents the estimated response (COF), while the Y-Axis represents the difference between the observed response and the fitted response. According to the normal probability plots (Figure 11), the residuals were well distributed, clustering closely around the straight regression line. The residuals appeared approximately normally distributed, providing preliminary support for the normality assumption. Furthermore, the difference between the observed and the fitted response values, as shown in Figure 11(b), was at most 0.004. The residual value below 0.05 can be considered significant [34]. Standardization of residuals on the Versus Fits plot shows that the points were randomly and symmetrically distributed around the zero line without forming any particular pattern. The absence of an obvious residual pattern provided preliminary support for the constant-variance assumption.



(a) (b)

Figure 11. Normal probability plots for COF and Versus Fits (response is means)

3.3 Analysis of variance (ANOVA)

ANOVA was used to determine the significance of each factor in this study. It was also used to estimate the relative contributions of the investigated factors. Table 8 shows the ANOVA of COF in this study. Based on the *P*-value, none of the investigated factors had a statistically significant effect on COF at the 0.05 significance level. However, the ultrasonication time showed the largest estimated contribution among the investigated factors, while none of the factors reached statistical significance based on the reported P-values; ultrasonication time accounted for almost 50% of the total contribution. After that, BC mass fraction accounted for approximately 36% of the estimated variation, while the addition of surfactant had a small impact on COF (4.8%). In this research, there were only two residual degrees of freedom ($df = 2$) in the ANOVA model, which naturally reduces the model's statistical power. This restriction could account for the finding that, despite ultrasonication time accounting for almost half of the total variation, no factor achieved formal statistical significance at $p<0.05$. Furthermore, it is noted that the tribological experiment was conducted once per sample due to the limited volumes of the custom lubricant and limited instrument availability.

Table 8. Analysis of variance for COF

Sources	DF	Seq SS	Adj SS	Adj MS	F	P-value	Contribution (%)
wt% BC	2	4.5636	4.5636	2.2818	3.93	0.203	36.2234
wt% Span 60	2	0.6054	0.6054	0.3027	0.52	0.657	4.8053
Sonication time (min.)	2	6.2678	6.2678	3.1339	5.4	0.156	49.7504
Residual Error	2	1.1617	1.1617	0.5808			
Total	8	12.5985					

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The coefficient of friction in this study was reported as the steady-state value measured over the last 600 seconds of operation, and the tribological evaluation for each lubricant formulation was conducted as a single screening test ($N = 1$). Standard deviations calculated over this period reflected within-run temporal variation and steady-state signal fluctuation rather than true between-run experimental uncertainty. Consequently, statistical parameters such as confidence intervals and inter-run error bounds could not be evaluated without independent test replications. To completely evaluate statistical significance and long-term repeatability, distinguish intrinsic test variability from material response, and verify statistical significance (including non-overlapping confidence intervals), future research will use independent test replications ($N \geq 3$) in compliance with conventional tribological methods (e.g., ASTM D4172).

4. Conclusion

This study evaluated the friction performance of palm oil-based TMP ester formulations containing bacterial cellulose and Span 60, considering the effects of the additive and surfactant mass fractions and the ultrasonication time. Based on the four-ball tribometer testing and the Taguchi analysis performed, it can be concluded that:

1. Ultrasonication time has the largest estimated contribution to COF variation among the investigated factors, followed by the percentage weight of bacterial cellulose and Span 60. The contribution of ultrasonication time, wt% BC, and wt% Span 60 values were approximately 49.75, 36.22, and 4.80%, respectively.
2. Based on the experimental value of COF, the optimum parameters were 0.3 wt% BC, 0.9 wt% Span 60, and an ultrasonication time of 10 minutes to obtain the lowest COF value of 0.0588. This was confirmed by statistical analysis using the Taguchi method.
3. The statistical model was adequate and reliable, in which the residual data of COF values were well distributed, clustering closely around the straight regression line, and residuals were randomly and symmetrically distributed around the zero line without forming any particular pattern. The error percentage between the actual COF during the test and the predicted COF by the Taguchi method was around 1.31%.

Author's declaration

Author contribution

Dedi Rosa Putra Cupu: Conceptualization, validation, statistical and formal analysis, investigation, writing – original draft. **Hairul Abral**: Supervision, funding acquisition, writing – review and editing. **Dedison Gasni**: Supervision, writing – review and editing. **Zahrul Fuadi**: Supervision, writing – review and editing. **Zulhanafi Paiman**: Experimental tribotesting supervision, writing – review and editing. **Chong Wei Quan**: Conducting tribological experiments, writing – review and editing.

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Data availability

The data obtained from tribometer testing and the Taguchi method, which support this study, are available from the corresponding author upon reasonable request.

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Conflict of interest

The authors declare no conflicts of interest in this work.

Ethical clearance

Not applicable.

AI statement

Grammarly and NotebookLM were used to assist with language editing, including grammatical accuracy, clarity, coherence, and readability. The manuscript also underwent professional proofreading by an English-language expert to ensure the quality and appropriateness of its academic English. All suggested revisions were reviewed and verified by the authors to ensure that the scientific content, experimental data, results, and interpretations remained accurate and unchanged. The authors take full responsibility for the final content of the manuscript.

Publisher's and Journal's note

Universitas Negeri Padang as the publisher, and Editor of Teknomekanik state that there is no conflict of interest towards this article publication.

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