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A Study On Preparation And Evaluation Of The Thread Greases from Renewable Resources Part1: Tribological Performance of Prepared Polymerized Jojoba Grades Including Jojoba Oil And Their Optimization

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Abstract

The scope of this study is investigating the tribological characteristics of prepared bio-based thread grease from renewable sources with polymerization products from jojoba oil as additives. Accordingly, the first part in this study aims to prepare three molecular weights grades from polymerized jojoba oil using microwave technique. The physicochemical and tribological properties of jojoba oil and its polymerizedwere determined .their tribological behaviour studied in term of coefficient of friction and wear scar diameter using four ball machines. The results revealed that the polymerized jojoba with low molecular weight shows the lowest friction coefficient and wear scar diameter compared with high molecular weight polymerized jojobagrades and jojoba oil before polymerization. This has been discussed based on the unique properties of the chemical structure of jojoba.

Studying three independent variables that affecting to coefficient of friction and wear scare diameter. using regression analysis. The studied affecting independent variables are the load, the velocity (revolution per minute, RPM) and the polymerization time., Optimization of these variables showed that minimum FCO and WSD could be achieved after polymerization time of 1.2 hr. under load of 196 N with a speed of 600 rpm.

It was concluded that the polymerized jojoba grade with low molecular weight has superior tribological performance and could be used as a significant potential additive for bio-based thread grease in the next part of this study.

Key words: jojoba oil, tribology, polymerized jojoba oil, friction, wear scare diameter

1. Introduction

Vegetable oils are used as bio lubricants and are considered to be environmentally friendly, biodegradable, and non-toxic because they are made from renewable resources.

The majority of biolubricant products currently on the market are manufactured entirely or partially from vegetable oils, as long as these oils meet international requirements for renewability, biodegradability and technical performance. A certified bio-based lubricant must have a carbon content in its chains of at least 25% and biodegradability of at least 60%, according to European standards [1, 2].In addition, the

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biolubricant must be non-toxic to the environment and suitable for the application. As a result, vegetable oils have a wide range of uses in the lubricant industry. Markets in European countries focus on vegetable oil-based lubricants for the automotive machines [1]. The use of vegetable oils and animal fats for lubrication purposes has been practiced for many years as an alternative to mineral oil as lubricant base stocks [2].

Non-edible vegetable oils are better lubricants because they have advantages such as better lubricity, biodegradability, non-toxicity, and a higher viscosity index, which are all requirements for lubricants for their application. They also have several limitations that can be handled by the modification of their properties using different processes like trans esterification [3, 4]. In general, lubricant technology dealing with jojoba oil and its derivatives in the 70's concentrated on its replacement of sulfurized sperm oil products in such applications as industrial and automotive gear oils, hydraulic oils and metal working lubricants[5, 6]. In the 80's, the lubrication industry has been developed and research on jojoba has been shifting towards new derivatives with potential application to new technologies and newer areas of lubricant use. A monograph by Wisniak et al, [7] summarized the chemistry and technology of jojoba oil and jojoba meal. Chung-Hung Chan et al [8] have studied in-depth discussion and comprehensive evidence of the tribological behaviors of biolubricant base stocks and additives. The influences of biolubricant base stocks and additives on tribological performance are summarized. Comparative tribological investigation on EN31 with non-edible oils as lubricant additives using a pin-on-disc tribometer at various loads and sliding distances were discussed [9].

Jojoba oil is a non-edible vegetable oil obtained from seeds of jojoba tree found in many parts of Egypt. Jojoba oil and its meal have proved excellent sources of prepared lubricating greases [10]. Previous research has been done on epoxidized jojoba and castor oil which are used as fluid for producing bio lubricating greases [3]. Bio grease additives are gaining popularity and acceptance globally due to their environmentally friendly and sustainable properties which are derived from nonedible vegetable oils.

Accordingly, this study reports the synthesis and characterization of the different grades from polymerized jojoba oil and explored their tribological behavior in term of coefficient of friction (COF) and wear scar diameter (WSD) using four ball tester. Furthermore, the optimization of the obtained data is also studied to determine the lubricity efficiency of the jojoba oil compared with its polymerized grades.

2. Material and Methods

2.1. Homo polymerization of jojoba oil and their characterizations

Egyptian jojoba oil sample, designated J_0, under investigation was supplied by Egyptian Oil Company. It undergoes radical polymerization, using microwave technique UWave-1000 - Sineo Microwave Chemistry Technology (China) Co., Ltd; the process of reaction and its changes in UWave-1000 could be observed using camera system. Before preparation the three different molecular weights from polymerized jojobagrades, under investigation, many trials were done to achieve theoptimal concentration of jojoba oil and initiator, sonication time, microwave power and reaction time. Accordingly, fifty grams of the jojoba oil monomer with 0.03 g benzoyl peroxide as initiator were mixed well using Ultrasonic homogenizer for 30 minutes to obtain one phase solution and then taken in quartz glass vials.The vials were subjected to microwave power 300 W for 1, 2 and 3 hours with stirring and bubbling with nitrogen which were subsequently quenched by quickly cooling to room temperature [11, 12]. The produced polymerized jojoba grades after the reaction time 1, 2 and 3 hours were designated PJ_1, PJ_2 and PJ_3, respectively.

The obtained polymerized jojoba grades in addition to jojoba oil were diluted with tetrahydrofuran (THF)to determine the average molecular weights using gel permeation chromatography (Water 600E) equipped with Styragel Column operated at 40^{O}C and flow rate 0.4 ml/min. The refractive index instrument model Water 4110 is used as a detector and THF (HPLC grade) is used as mobile phases.

Oxidation assessment in term of oxidation stability index (OSI) for jojoba and its polymerized grades were determined according to Rancimat method, [13]. Dynamic viscosity measurement was performed using a Brookfield programmable Rheometer LV DV-III UITRA used in conjunction with Brookfield software, RHEOCALC V.2. Also, iodine value and flash point were determined by applying the Wijs method as Ketaren reported[14] and ASTM D92, respectively. Experimental data of the physicochemical properties were presented in Table 1.

Table 1: Physicochemical properties of jojoba oil and its polymerized grades.

Items	Average molecular weight	Dynamic viscosity@ $50^{O}C$(rpm 30), cP	Oxidation stability index	Iodine value	Flash point, ^{0}C
J_0	560	40	51.0	82.1	290
PJ_1	1230	67	68.7	56.5	295
PJ_2	4523	132	78.0	41.0	310
PJ_3	17573	401	85.0	29.0	335

2.2. Tribological assessment

In order to explore the tribological assessment of the jojoba oil and its polymerized grades under investigation, a four ball test machine (MMW-1Acompouter control vertical universal friction wear test machine) has been used [15].

All steel balls were thoroughly cleaned and dried before and after the experiments with acetone. The four steel balls are made of chrome alloy steel with 12.7mm diameter, following AISI E-52100 standard, extra polished (EP Grade 25) and hardened to 64-66 HRc (Rockwell C Hardness).

The conditions of four balls tester machine such as load, engine speed, operating time and temperature were 196-782 N, 200-1200 rpm, 30 minutes per specimen and room temperature, respectively.
These conditions are presented in Table2. Wear scare diameter (WSD) of the balls were determined using optical microscope as averaged WSD values. The Coefficient of Friction was calculated using the following equation as per [21].

$$\mu_k = F_k/N$$

Where μ_k is coefficient of kinetic friction, F_k is applied force, and N is load

Table 2: Conditions of four ball tester machine

Parameters	Range
Applied load, N	196-782
Engine speed, rpm	200-1200
Temperature, ^{0}C	Ambient temperature
Operating time, min	30 per specimen

To achieve the optimum value for Independent variables that affecting on coefficient of friction and wear scare diameter the optimization program Multiple-objective optimization software (LINDO, version 17) is used in this work.

3. Results and discussions

3.1. Physicochemical properties

The results of physiochemical characterization of the Egyptian jojoba oil and its prepared polymerized gradesare summarized in Table1. The obtained polymerization products of jojoba oil are confirmed by molecular weight, iodine value, dynamic viscosity, oxidation stability index and flash point, which are the most important items for lubricity function of such environmentally friendly lubricant-based stocks. These results showed the successful oligomerization-polymerization of jojoba oil and the vital role of microwave radiation time (1, 2 and 3 hours) to produce different molecular weights 1230, 4523 and 17573 of polymerized jojoba grades; PJ_1, PJ_2 and PJ_3, respectively. Inspection data of obtained molecular weights of these grades reveal that the PJ_1 and PJ_2 are produced from oligomerization, while PJ_3 is obtained from polymerization. This indicates that the jojoba oil needs sufficient time (more than two hours) to polymerize under such condition. It was concluded that through microwave radiation time, under such condition, could be controlled and selective in molecular weight of polymerized jojoba obtained from jojoba oil.
On other hand, the iodine value is a measure for the degree of unsaturation of fats and oils. Table1, shows that the iodine value data are in order $J_0 > PJ_1 > PJ_2 > PJ_3$, which indicates low iodine values of polymerized jojoba compared with jojoba oil.This indicates that the iodine value is related to the average molecular weight of the studied samples. Therefore, these low iodine values of PJ_1, PJ_2 and PJ_3 are confirmed and considered as good indicators to the oligomerizationand polymerization products obtained. This finding is in agreement with earlier reports showing that the higher iodine values indicate higher unsaturation of fats and oils [16, 17].
The apparent viscosity of jojoba oil and its polymerizedgrades is an important basic characteristic. There is a wide variation in their viscosity ranging from 40 cP of J_0 to 401cP of PJ_3 as shown in Table 1. This indicates that the dynamic viscosity is related to the average molecular weight of the jojoba oil and its polymerized grades. Therefore, the increase in dynamic viscosity for PJ_3, confirm that PJ_3 has resistance to segment deformation under shearing condition. The obtained results agree with those earlier reports saying that viscosity increased with molecular weight but decreased with increasing

unsaturated level of fats . Flash point data presented in Table 1 revealed that lowest (290ºC) was recorded for J_0 and highest (335ºC) was recorded of PJ_3. This indicates that the J_0has greater tendency for volatilization than PJ_3 as temperature increases. Accordingly, high flash and high viscosity values of PJ_3 indicate that PJ_3 can be used as a pollution free additive for lubricating greases. On the other hand, iodine value, dynamic viscosity and flash point data as manifested in Table 1, were successfully consistent with the molecular weights data obtained of polymerized samples PJ_1, PJ_2 and PJ_3.

The mechanism of the autoxidation of vegetable oils was well studied[17]. Vegetable oil oxidation was initiated by the formation of free radicals; freeradicals could easily be formed from the removal of a hydrogen atom from the methylene group next to a double bond. Free radicals rapidly reacted with oxygen to form a peroxy radical. Comparative investigation between jojoba oil and its polymerized grades (PJ_1, PJ_2 and PJ_3) through the oxidation reaction were carried out using Rancimat method [13]; the effects of the variation in the oxidation time on the conductivity of obtained primary oxidation products and secondary oxidation compounds had been determined at 100^0C. Data in Table 1 showed oxidation stability index (induction time)51.0, 68.7, 78.0 and 85.0 hours for J0, PJ_1, PJ_2, and PJ_3, respectively. The results showed that the $PJ_3$was, in general, more effective in controlling the oxidative deterioration and more efficient in preventing the formation of primary oxidation products and secondary oxidation compounds.

It indicates that the content of oxidation products which results from unsaturation bonds content triggers the oxidation process. This finding was supported by the iodine value measured for investigated samples. This is attributed to the deceasing the unsaturation bonds in PJ_3 compared to PJ_2, $PJ_1$and J_0 which acts as initiating group to oxidation due to their absorbed oxygen. These results emphasize those of Gouveia-de-Soua et al.[18] and Erhan et al. [19]concerning monounsaturated and polyunsaturated chains in the vegetable oil. It was concluded that the lower unsaturation bonds in polymerized jojoba by increasing molecular weight could increase oxidation stability index. Generally, as indicated in Table 1, the physicochemical properties of polymerized jojoba grades have been improved compared to pure jojoba oil.

3.2. Friction and wear behavior

The results of the tribological test conducted on synthesized polymerized jojoba and jojoba oil, to evaluate their performance, are graphically presented in figures 1-4 to optimize the type of these synthesized samples for best reduction of wear and friction. These figures show the variation of friction coefficient and wear scar diameter with the lubricating conditions.

Figure 1 shows performance of synthesized polymerized jojobagrades and jojoba oil at different applied loads at engine speed 800rpm. It indicates that the PJ_1 with molecular weight 1230 showed positive effect on friction coefficient compared with jojoba oil alone, PJ_2 and PJ_3. This may be due to the influence of the movement of molecular backbone chains of PJ_1 oligomer results in the easier rearrangement of segment on the ball surface leading to formation of stable tribofilm during shearing, i.e. protective layer adhere to the metal ball surface, thus reducing friction. In this respect, PJ_1 oligomer with molecular weight 1230 has effective segment coil radius that make PJ_1 has effective adsorption on ball surface compared with J_0, PJ_2 and PJ_3.Figure1also indicates that the oligomerization products (PJ_1 and PJ_2) of jojoba oil demonstrated better lubricity. These results agree with those of Ossia et al. [20] on the effect of chain lengths on the coefficient of friction in boundary lubrications.

On the other hand, the high molecular weights of polymerized jojoba PJ_3 obtained negative effect on friction coefficient; overload and unstable friction occur in state of PJ_3 polymer as mentioned in Figure 1. This may be due to coagulation segment effect and lost the adsorption of the tribofilm on balls in state of PJ_3.

The same trend above observed concerning wear scar diameter against various loads and engine speed 800rpm as mentioned in Figure 2. It clearly indicates that the wear scar diameter of PJ_1 is decreased significantly compared with PJ_3, and moderately with J_0 and PJ_2. This may be attributed to decreased deformation of creating an effective tribofilm due to the strong interaction of PJ_1 on metal surface ball as mentioned above

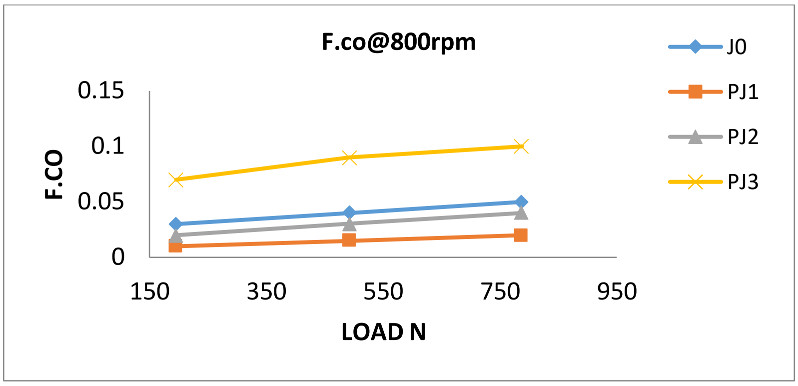

Figure 1: Coefficient of friction of jojoba oil and its polymerized grades at different loads and constant speed machine 800rpm

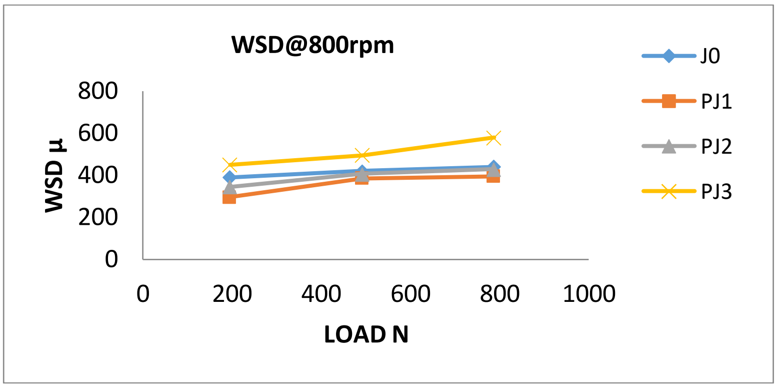

Figure 2: Antiwear behavior of jojoba oil and its polymerized under different loads and constant speed machine 800rpm

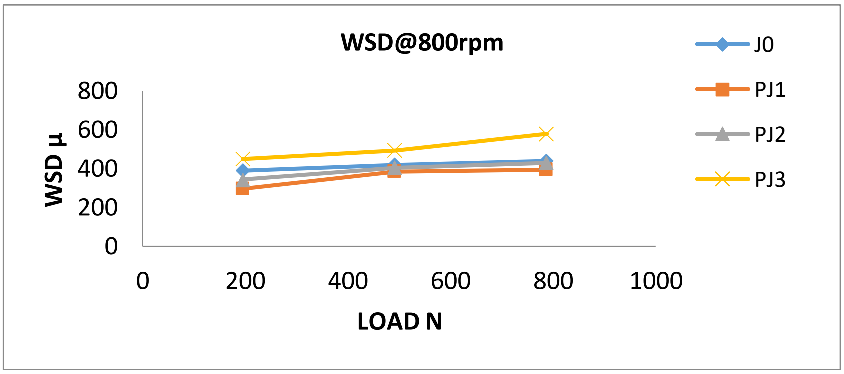

Figure 3: Coefficient of friction of jojoba oil and its polymerized at different engine speed and constant load 500N

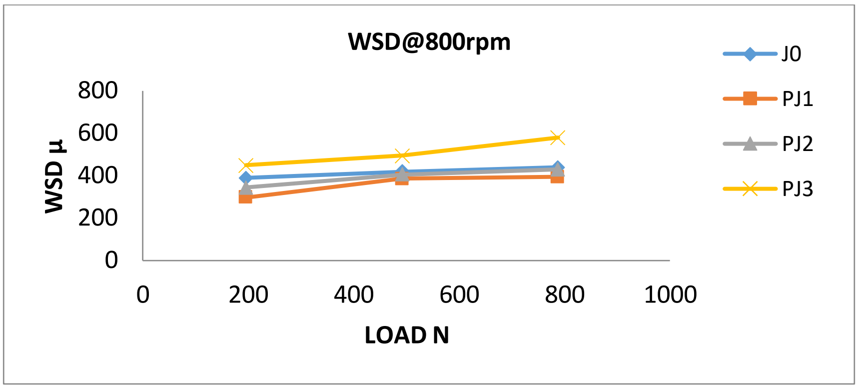

Figure 4: Antiwear behavior of jojoba oil and it's polymerized under differentengine speed and constant load 500N

Table 3: Experimental data for FCO and WSD under different conditions *P,hr is designated polymerization time,(hour)

FCO	WSD	Load N	RPM	*P.hr	FCO	WSD	Load N	RPM	*P.hr
0.032	402	196	800	0	0.025	350	196	800	2
0.035	413	196	1000	0	0.029	362	196	1000	2
0.04	422	492	800	0	0.03	417	492	800	2
0.052	450	492	1200	0	0.045	428	492	1200	2
0.047	433	492	1000	0	0.037	415	492	1000	2
0.05	460	788	1200	0	0.05	450	788	1200	2
0.034	406	492	600	0	0.027	392	492	600	2
0.032	410	788	200	0	0.029	400	788	200	2
0.041	421	788	600	0	0.032	417	788	600	2
0.028	391	196	600	0	0.021	355	196	600	2
0.047	435	788	1000	0	0.042	425	788	1000	2
0.025	398	492	200	0	0.02	380	492	200	2
0.038	420	196	1200	0	0.032	392	196	1200	2
0.02	380	196	200	0	0.04	350	196	200	2
0.045	427	788	800	0	0.037	421	788	800	2
0.012	331	196	800	1	0.068	420	196	800	3
0.018	345	196	1000	1	0.072	432	196	1000	3
0.015	385	492	800	1	0.09	495	492	800	3
0.024	400	492	1200	1	0.1	550	492	1200	3
0.019	392	492	1000	1	0.094	520	492	1000	3
0.035	420	788	1200	1	0.15	600	788	1200	3
0.012	370	492	600	1	0.08	475	492	600	3
0.02	380	788	200	1	0.082	489	788	200	3
0.027	392	788	600	1	0.092	490	788	600	3
0.008	345	196	600	1	0.061	399	196	600	3
0.032	410	788	1000	1	0.12	557	788	1000	3
0.009	363	492	200	1	0.071	459	492	200	3
0.021	382	196	1200	1	0.075	425	196	1200	3
0.005	341	196	200	1	0.053	390	196	200	3
0.029	401	788	800	1	0.095	530	788	800	3

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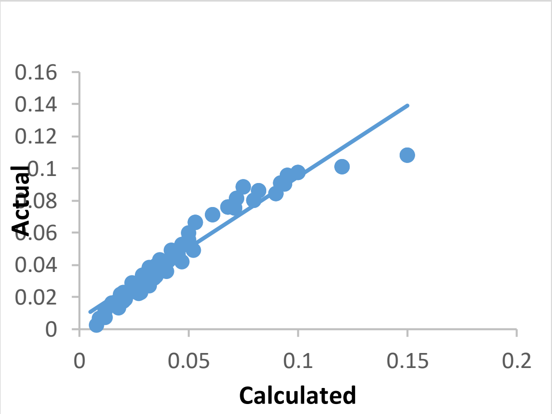

Figure 5 : Relation between actual and calculated results of FCO

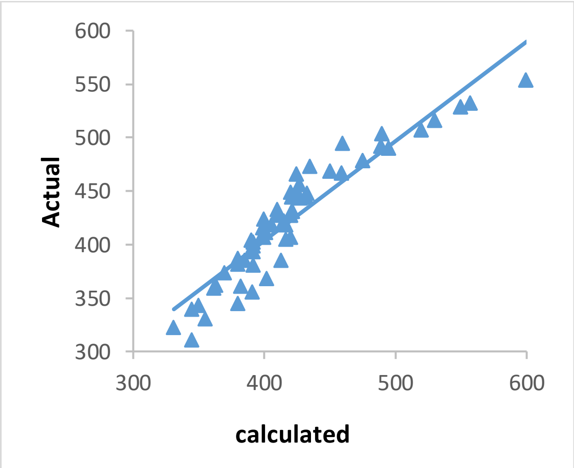

Figure 6 : Relation between actual and calculated results of WSD

Figure 3 and 4, shows the main effect of friction coefficient and wear scar diameter at different engine speeds and constant load 500N to evaluate the performance of synthesized polymerized jojoba grades and jojoba oil. Obtained results in these Figures showed superior protection ball surface against friction and wear in state of PJ_1 as lubricant compared with other samples

In this respect, the inspection of the tribological properties shown graphically in Figures 1-4, reveal that the optimum polymerized grades for minimum frictionco efficient and less wear scar diameter were PJ_1 and PJ_2. Consequently, the observed similarities in tribological properties forPJ_1 and PJ_2 oligomers obtained, mean that only a narrow range of molecular weight from 1230-4523 have significantly improved and tailored for their potential use as lubricants due to the high reactivity and functionality of their oligomer segments. It was concluded that the oligomerization products (PJ1 and PJ2) of jojoba oil are most suitable and appropriate lubricant, providing a durable lubricant film, compared with its polymerization product PJ3. Also, the unique structure of jojoba oil[7] , long chains with ester group, provides high strength lubricant films which it confirms the unique properties of polymerized jojoba grades (PJ1 and PJ2) obtained from jojoba oil oligomerization under such conditions studied in this part. This confirms the oligomerization is tailored process for improved jojoba oil products using microwave technique under such conditions.

3.3 Optimization of different affecting variables on FCO and WSD

The aim of this section is to get the optimum conditions lead to minimum FCO and WSD. It is found in this study that both of these items are widely affected by the load, the speed, in addition to the polymerization time. So, two correlations have been introduced to incorporate all these affecting variables to predict the values of FCO and WSD simultaneously. The experimental data given in Table 3 are used to extract correlations1and 2 using regression analysis.

Table 4: Statics for the proposed correlations

Correlation	R^2	Predicted R^2	Standard Error
Equation 1	0.92	0.96	0.008
Equation 2	0.95	0.97	0.005
Correlation	R^2	Predicted R^2	Standard Error

Table 5: Limitation for the studied variables used in optimization

Variable	Lower limit	Upper limit
N	196	788
RPM	200	1200
Time (hr)	0	3

$$FCO=0.012821+2.26E\text{-}05*N+\text{-}9E\text{-}07*rpm+\text{-}0.03821*t+1.08E\ 08*N^2+1.66E\text{-}08*rpm^2+0.018117*t^2 \quad \text{eq. 1}$$

$$WSD=283.8322+0.358638*N\text{-}0.01598*rpm\text{-}78.2*t\text{-}0.00021*N^2+5.54E\text{-}05*rpm^2+32.66667*t^2 \quad \text{eq. 2}$$

Where N is the load (N); rpm is revolution per minute and t is the polymerization time, hrs. Table 4 contains R-squared test results of the two introduced correlations. As shown in Table 4, R^2 and multiple R^2 are very close to 1, while the standard error approaches 0 for both correlations. This proves that the proposed correlations fit well the experimental results.

It is worth to mention that R^2 statistical test is used to evaluate how well the correlations results agree with the experimental data. R^2 is the fraction by which the variance of the errors is less than the variance of the dependent variable. The range of R^2 is between 0 and 1; it is the relative predictive power of a model. As R^2 value is close to 1, the correlation results are in a good agreement with the experimental results. Another quantity is predicted R^2, which indicates how well the model predicts responses for new observations.[21-23].Figures 5 and 6 illustrate the goodness of fit in experimental and calculated results. The introduced validated correlations are used to build up an optimization program aiming to find the optimum values for the affecting variables used in this research work. Multiple-objective optimization software (LINDO, version 17) is used in this work to perform the required optimization, aiming to minimize both FCO and WSD. The limits for the affecting variables are shown in Table (5). The introduced program, which is a non-linear program resulted that the minimum WSD and FCO are 309 and 0.003 respectively. This could be achieved at 196 N, 600 rpm and 1.2 hr.

Conclusions

Different Polymerized jojoba grades were synthesized from polymerization of jojoba oilusing microwave technique. The physicochemical and tribological properties were determined according to ASTM. The major conclusions can be summarized as follows:

1. Polymerization under such condition, using microwave technique, considers a novel synthetic approach for the chemical modification of jojoba oil to improve their Physicochemical and tribological properties.
2. Polymerized jojoba grades within molecular weight ranging 1230-4523 exhibit marked improvement in tribological performance, lowest values of both COF and wear scar diameter, due to functionality of their oligomer segments
3. Oligomerizationof jojoba oil is interesting and provides a promise insight into the field of utilization of polymerized jojoba as additives for bio-based thread grease in the next part of this study.

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