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RESEARCH

Vegetable Oil based Biodegradable Refrigeration Oil - An Optimization of Tribological Characteristics Using D-optimal Design

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A B S T R A C T

The present study investigates the tribological behaviour of vegetable oil-based polyolester refrigeration oil using the pin-on-disc tribometer. Experiments were carried out choosing the load and sliding velocity as a function of rotational speed as the numerical factor and refrigeration oil as the categorical factor. Response surface methodology (RSM) based D-optimal design was utilized for modelling and optimization of friction and wear characteristics. The tribological characteristics of a lubricant-material combination of hermetically sealed compressor was observed with synthetic refrigeration oil (POE68), vegetable oil based polyolester (BIO) and blended refrigeration oil i.e., (BIO50) 50% by vol. of vegetable oil based refrigeration oil & 50% by vol. of POE68. The results showed that the application of blended refrigeration oil (BIO50) leads to the improved tribological behaviour in terms of coefficient of friction of 0.02742 and specific wear rate of 1.98x10-4 mm³/Nm at a speed of 1160 rpm and load of 7.44 kg. The surface morphology analysis of worn pin surfaces showed that the compressor material lubricated with blended refrigeration oil was glossier than synthetic and vegetable oil based polyolester refrigeration oil.

1. INTRODUCTION

The rising demands for energy usage and environmental safety have been the problems the refrigeration industry has been facing for the past few decades. Therefore, the effective energy utilization of refrigerators has to be improved and petroleum based lubricants are gradually being replaced consequent on the effect of environmental protection [1]. Vegetable oils have excellent lubricant properties which include high viscosity index, low volatility, high miscibility and excellent lubricity compared to other kinds of synthetic refrigeration oil [2-4]. Regardless of these advantages, bio- lubricants possess poor low temperature performance and thermal-oxidation stability [5, 6]. In order to overcome these restrictions, several chemical alteration methods like transesterification, selective hydrogenation, and epoxidation of vegetable oil can be tried. Earlier research findings reported that the transesterification of vegetable oil has an increased possibility for the synthesis of lubricant with enhanced temperature stability and significant fluidity [7]. This modified method support is to efficiently replace the hydrogen atom on the β-carbon structure of the oil. Different types of polyolester like trimethylolpropane, pentaerythritol and neopentylglycol were mostly used in the synthesis of bio-lubricants [8].

Refrigerator compressors enclose highly contacted mechanically loaded components that have to be adequately lubricated. Refrigeration oil is an indispensable element of the refrigerator compressor. The perceptible task of refrigeration oil in the compressor is cooling the sliding parts, sealing, maintaining the system equalizing pressure, decreasing the noise level and protecting against corrosion [9]. Additionally, the refrigeration oils are mainly employed to reduce friction and wear and thereby to increase efficiency and longevity of the aforesaid components [10]. The performance of refrigeration oils is considered satisfactory if the oil mists entrained with high-pressure discharge gas in the compressor system are readily separated from the refrigerant and quickly returned from condenser or evaporator. Such oil behaviour largely depends upon its liquid-vapour equilibrium data and their intensive properties such as viscosity, density and thermal conductivity [11]. Practically, almost 80% of the power consumed by the refrigerator is utilized to run the compressor. On the whole, the compressor power expenditure, nearly 60-70% is due to frictional losses [12]. Conventional refrigeration oils for CFC refrigerants in the refrigeration system are naphthenic mineral oil, paraffinic mineral oil and heavy alkyl benzene. Freon refrigerants are replaced by the HFC, HFO and HC refrigerants on the consideration of environmental protection and energy economy. Synthetic lubricants are highly compatible with the commonly used HFC refrigerants. POE68 is a synthetic refrigeration oil of ISO 68 grade which is used as a refrigeration lubricant in the domestic refrigerators. Polyolester oil (POE) with the viscosities ISO 15-220 is suitable for industrial and household piston and screw compressors. Saravanan and Vijayan [13] analyzed the influence of Al_2O_3/TiO_2 nano composite particles in commonly used synthetic lubricant in domestic refrigeration system i.e., polyolester oil (POE68) in the mass fractions of 25%, 50%, and 75% in household refrigeration system and reported that the power consumption of 50% blend of Al_2O_3/TiO_2 nano in POE 68 was reduced by 12.29% evaluated with POE 68 oil.

Many investigators have reported the thermal enhancement and tribological improvement of mineral and synthetic refrigeration oil and bio lubricants with and without the addition of nanoparticles and concluded that the inclusion of nanoparticles to refrigeration oil leads to the reduction in power consumption, lower friction coefficients and enhancement in COP [14-18]. However, the inclusion of nanoparticles in lubricants, contribute to copious problems such as clogging, sedimentation and erosion and reasons for significant enhancement with nanoparticles at different concentrations are also unclear [19]. Refrigeration oil directly influences the tribological behaviour of interacting components of a compressor [20]. Piston rings in the compressor as to provide the lubrication to the cylinder liner surface, act as a sealing agent to avoid leakage of gasses and to relocate heat from the piston to the cylinder surface and the cylinder liner surface is more exposed to friction and wear [21]. Therefore compatibility of piston ring/cylinder liner tribological pair material of a refrigeration compressor is imperative while developing the refrigeration oil. De Mello et al., [22] evaluated the tribological performance of actual refrigerant environments (air, CO_2 and R600a) in hermetically sealed refrigeration compressor materials (1020 steel disc and 52100 steel pins) coated with Si-rich multifunctional DLC coating via high pressure reciprocating pin-on-disk tribometer and concluded that R600a refrigerant atmosphere showed excellent friction coefficient of 234% greater compared to the air atmosphere and wear rate enhanced by 33% and 52% CO_2 and air respectively. Akram et al., [23] analyzed the tribological behaviour of grey cast iron material in the presence of refrigerant HFO-1234yf with PAG/POE lubricants at two different temperatures (24°C and 110°C). Their experimental results showed that the PAG/HFO-1234yf exhibited superior tribological performance compared to POE/HFO-1234yf. Kowser et al., [24] studied the tribological performance of compressor material (carbon steel-MN 4.9) on dry, R22 and R134a refrigerant at lubrication conditions. According to their results, the sliding pair of piston ring/cylinder liner material showed the superior tribological properties in terms of wear and friction under R22 refrigerant owing to the formation of the protective oxide layer. Shahabuddin et al., [25] revealed the tribological property of bio-lubricant derived from Jatropha oil and concluded that 10% addition of Jatropha oil with base lubricant exhibited superior tribological properties in-terms of friction and wear.

RSM is a collection of statistical and mathematical techniques useful for developing, improving, and optimizing processes. The objectives of quality improvement, including reduction invariability and improved process and product performance, can often be accomplished directly using RSM [26]. D-optimal designs are straight optimization techniques mostly suitable when classical designs do not apply and successful method for mixed type mixed-level factorial experimentations. Yashvir Singh et al., [27] investigated the friction and wear properties of phyllanthus emblica seed oil based lubricants via pin on disc tribometer. The RSM was used to optimize the blend ratio, load and sliding speed to achieve the optimum specific wear rate and coefficient of friction and reported that 10% blend of phyllanthus emblica seed oil with commercial lubricant showed the superior tribological behaviour in terms of coefficient of friction and wear rate.

Hence, it is essential to perform a tribological study of compressor materials under the influence of vegetable oil based polyolester refrigeration oil to select the appropriate material/lubricant pair for a vapour compression refrigeration system. In the present study, vegetable oil based pentaerythritol ester is formulated and to be used as probable substitution for synthetic grade refrigeration oil. RSM-based D-optimal design was used for designing the tribological experiments and for modelling and analyzing the output responses of tribological tests such as coefficient of friction (COF) and specific wear rate (SWR).

2. EXPERIMENTAL APPROACH

2.1 Formulation of Vegetable Oil-based Polyolester Refrigeration Oil

This work focuses on the development of vegetable-based polyolester as biodegradable refrigeration oil that can be utilized in domestic refrigerator's compressors to replace petroleum-based synthetic refrigeration oil. Rapeseed oil is used for the formulation of polyolester-based bio-lubricant. The successive transesterification process was adopted for the synthesis of rapeseed oil into pentaerythritol ester. For 2 litre of rapeseed oil, methanol 880ml and NaOH of 18.5g were mixed well using a mechanical stirrer and heated up-to 55°C for one hour. The obtained methyl ester was then mixed with pentaerythritol of 95g and para-toluene sulphonic acid of 55g and xylene and heated to 150-160°C for 8 hour under nitrogen atmosphere. The obtained product mixture was allowed to cool to room temperature and purified with sodium carbonate solution to remove any un-reacted acid. Finally, the product of pentaerythritol ester of rapeseed oil is designated as biodegradable refrigeration oil (BIO). The detailed procedure for the formulation of Pentaerythritol ester was adopted from Hashem et al., [28]. Fig. 1 shows the chemical reaction of successive transesterification process.

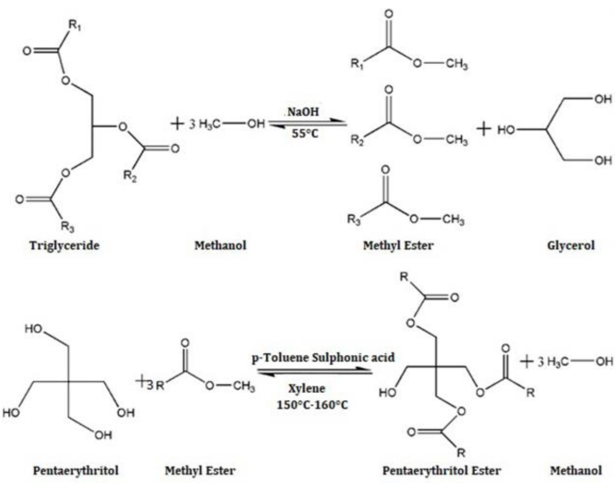


Fig. 1. Chemical reaction of successive transesterification process

2.2 Preparation of Vegetable Oil-based Polyolester Refrigeration Oil Blend

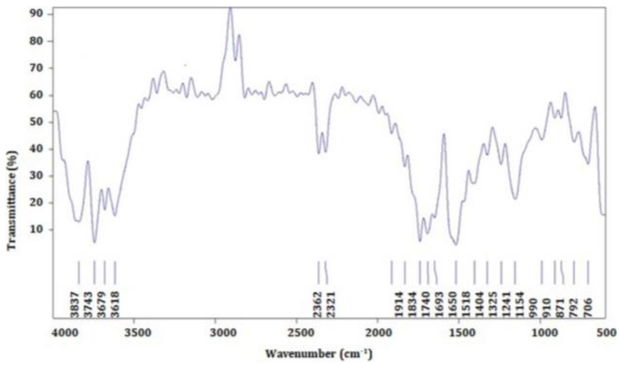
A commonly used synthetic lubricant in domestic refrigeration system i.e., polyolester (POE68) was chosen as a base fluid for comparison. The formulated pentaerythritol ester of rapeseed oil (BIO) was blended with POE68 grade synthetic refrigeration oil in 50% by volume fraction which is designated as BIO50. The blend preparation was carried out in a round-bottom glass reactor, and the mixture was heated up to 60°C by maintaining the stirring speed of 300 rpm for 1/2 hour [29]. Table 1 listed the physio-chemical properties of the refrigeration oil samples and Table 2 depicts the percentage composition of refrigeration oils. The functional groups and molecular bonding of pentaerythritol ester was identified using the FTIR (Fourier Transform Infrared Spectrometer Bruker Alpha T, Germany) spectroscopy technique. Fig. 2 (a) and (b) shows the FTIR spectra of pentaerythritol ester-based refrigeration oil and POE68. It can be seen from Fig. 2(a), the esterification of rapeseed oil methyl ester with pentaerythritol was confirmed by the ester peak disappearance of C=O stretching vibration at 1740 cm^{-1}.

Table 1. Physio-chemical properties of refrigeration oils

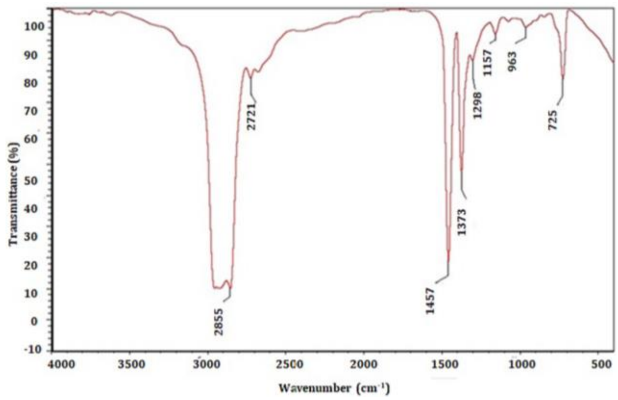
Properties	Standard	POE68	BIO	BIO50
Kinematic viscosity @40°C (cSt)	ASTMD445	68	67	56.4
Kinematic viscosity @100°C (cSt)	ASTMD445	9.3	9.02	10.54
Viscosity index	ASTMD2270	114	156	244
Flash point(°C)	ASTM D92	270	242	232
Pour point(°C)	ASTM D97	-39	-25	-28
Total Acid Number (mg KOH/g)	ASTMD974	0.3	1.2	0.79
Copper Corrosion @100°C	ASTMD130	1A	1A	1A

Table 2. Percentage composition of refrigeration oils

Lubricant	Pentaerythritol ester of rapeseed oil	Synthetic refrigeration oil
POE68	0	100
BIO	100	0
BIO50	50	50



(a)



(b)

Fig. 2. (a) FTIR spectra of Vegetable oil based pentaerythritol ester oil, (b) FTIR spectra of POE68

The existence of band at 3743 cm^{-1} correlated to the O-H stretching vibration of carboxylic acid. The appearance of stretching peak at 1154 cm^{-1} shows the evidence of the presence of C-O-C. The completion process of esterification reactions was identified by the hydroxyl group peak that appeared at 3743 cm^{-1}. The absorption bands at 2362-2321 cm^{-1} and 1518-1404 cm^{-1} of C-H stretching and at 706 cm^{-1} peak shows the existence of alkane functional group and the long chains of alkyls respectively. Fig. 2(b) shows the FTIR spectra of synthetic refrigeration oil (POE68). It can be seen that the absorption band appeared between 3600-3200 cm^{-1} corresponding to O-H groups in the polyolester oil. The existence of peak at 1457 cm^{-1} correlated to the N-H stretching vibration band. The absorption bands observed in the region 1800-1500 cm^{-1} corresponding to oxidation products containing carbonyl groups and antioxidants (1373 cm^{-1}) present in the polyolester oil. The appearance of peaks at 963 cm^{-1} and 1157 cm^{-1} correlated to the O-H bending of antioxidants and presence of anti-wear phosphate additives respectively.

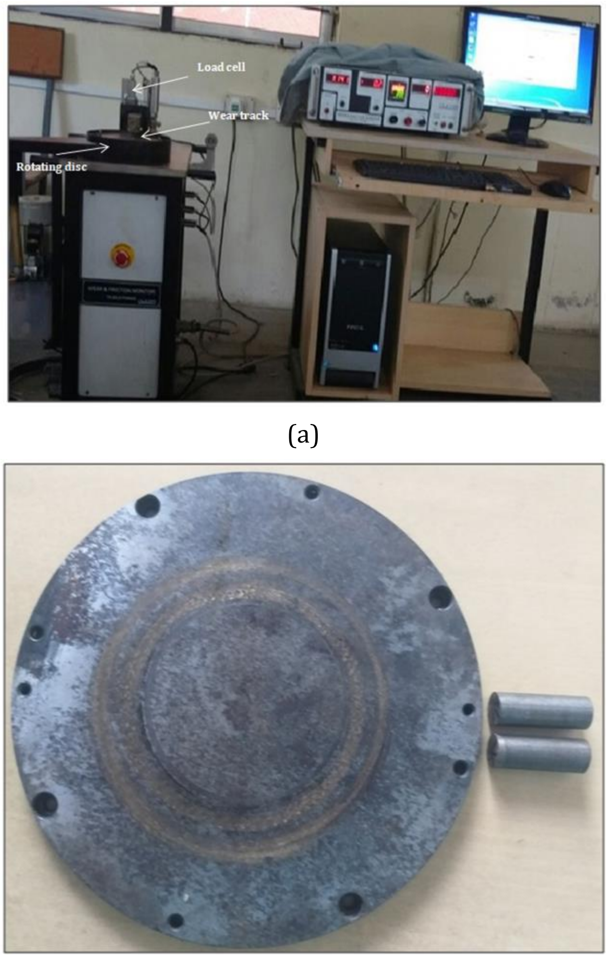
2.3 Test matrix and Procedure

Response surface methodology (RSM) is a method of statistical design and mathematical optimization technique used to develop and optimize the process parameters. RSM can be applied to determine the interaction of several affecting factors [30]. A D-optimal test matrix based on RSM is employed for experimental design for the tribological test and mathematical modelling and also for the optimization of the output responses-coefficient of friction (COF) and specific wear rate (SWR) [31]. The experiment was designed by selecting the load and sliding velocity as a function of rotational speed as numerical factor and refrigeration oil as categorical factor to examine the tribological behaviour. The numerical and categorical factors of the tribological experiments are depicted by Table 3.

Table 3. Coded and actual levels of variables for experiments

Parameters	Levels			
	Numerical	**-1**	**0**	**1**
	Categorical	**1**	**2**	**3**
Load (kg)	Numerical	5	10	15
Rotational speed (rpm)	Numerical	400	800	1200
Refrigeration oil	Categorical	BIO	BIO50	POE68

The tribological behaviour of refrigeration oil samples was investigated using a pin- on-disc tribometer (Ducom Instruments Pvt. Ltd., Bangalore, India, Model TR-20LE) by following ASTM G99, which is equipped with a data acquisition system. A schematic view of the pin-on-disc tribometer is shown in Fig. 3(a). A photographic view of disc and pin is shown in Fig. 3(b). The pins and disc material imitate the actual liner-ring contact of the hermetically sealed reciprocating compressor of domestic refrigerator. The pins are made of piston ring material-FG 150 grey lamellar graphite cast iron of hardness 210 BHN of 12mm diameter and the disc (165mm diameter, 8mm thick) are made up of cylinder liner material of compressor-SAE J431 grade cast iron with manganese 0.4-1.2 wt%, heat-treated and hardened up to 60 HRC. In actual practice, it is very complicated to precisely envisage the situation in the compressor as always having the varying functioning environment. However, the number of parameters selected which affects the tribological performance of contacting surfaces such as sliding speed and the contact pressure due to applied load led to frictional heating.



(a)

(b)

Fig. 3. (a) Schematic view of Pin on disc tribometer, (b) Photographic view of disc and pin

Lubricant flow velocity is maintained constant as 275ml/min in order to achieve flooded lubrication condition. However, the variation in speed doesn't affect the tribological characteristics in flooded lubrication. The maximum applied load and speed of pin on disc tribometer was limited to 200N and 2000rpm respectively. The test conditions for the tribological tests were chosen as load of 50N, 100N and 150N with the rotational speed of 400 rpm, 800 rpm and 1200 rpm for a constant sliding distance of 1000m. During the experiment, the refrigeration oil was introduced and circulated between the rotating disc and stationary pin by a lubricant pump. The rotational speed and average load acting on the disc generates the frictional force-the sensors used for the determination of the coefficient of friction. The pins are weighed before and after each test. The specific wear rate was computed using a weight-loss method calculation.

Table 4. Test matrix and Responses

Run	Numerical factor		Responses				
			Categorical factor	COF		Specific wear rate (mm^3/Nm)	
	Load (kg)	Rotational speed (rpm)	Refrigeration oil	Actual	Predicted	Actual	Predicted
14	10	1200	BIO	0.048	0.04742	0.00032	0.000040
12	15	800	BIO	0.057	0.05631	0.00035	0.000039
10	5	800	BIO	0.052	0.05497	0.000218	0.000029
6	5	400	BIO50	0.041	0.04218	0.00029	0.000028
4	10	400	BIO	0.029	0.02985	0.000212	0.000026
2	15	1200	BIO50	0.05	0.05059	0.000276	0.000029
5	15	400	BIO50	0.062	0.06093	0.000368	0.000029
8	5	400	POE68	0.049	0.04801	0.000316	0.000032
9	15	1200	POE68	0.043	0.04315	0.00043	0.000041
20	5	1200	POE68	0.046	0.04336	0.00038	0.000036
3	5	1200	BIO50	0.031	0.03046	0.000258	0.000026
11	10	800	POE68	0.039	0.03695	0.000269	0.000030
17	10	800	BIO50	0.038	0.03775	0.000216	0.000034
22	5	400	BIO	0.045	0.04217	0.000297	0.000030
15	15	400	POE68	0.047	0.04643	0.00039	0.000045
16	15	400	BIO	0.042	0.04282	0.000397	0.000031
18	10	400	POE68	0.032	0.03457	0.00022	0.000022
1	15	1200	BIO50	0.05	0.05059	0.000276	0.000029
7	10	1200	BIO	0.048	0.04742	0.00023	0.000040
13	5	1200	BIO50	0.031	0.03046	0.000258	0.000026
19	15	1200	POE68	0.043	0.04315	0.00043	0.000041
21	5	1200	POE68	0.04	0.04336	0.00038	0.000037

$$\text{Specific wear rate} = \frac{\text{Volume loss}}{\text{Applied load} \times \text{Sliding distance}} \text{ in } mm^3/Nm \tag{1}$$

$$\text{Sliding distance} = \frac{\pi DNT}{60000} \text{ in m} \tag{2}$$

$$\text{Volume loss} = \frac{\text{Weight loss} \times 1000}{\text{Density of the pin material}} \text{ in } mm^3 \tag{3}$$

Where D is the track diameter of the disc, N is the rotational speed of the disc and density of the pin material is 7.2 kg/m^3. The combined SEM/EDX analysis was done by using FEI Quanta 650 FEG microscope. Using the D-optimal design, 22 experiments were performed, and the outcomes are shown in Table 4. The whole procedure is repeated thrice at a similar experimental condition to ensure accurate outcomes of the test data and the average values are listed.

2.4 RSM based on a D-optimal Design

D-optimal design based on surface response based methodology was employed for performing the design of experiments for the tribological tests. Mathematical models generated for the output responses were such as coefficient of friction and specific wear rate. The independent variables effects on the tribological behaviour of refrigeration oils are investigated using a quadratic polynomial model, which was evaluated base on the experimental results with respective coefficients. A second-order polynomial equation (Eqn.4) was used to explicit the outcome of the tribological behaviour of refrigeration oils.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ij} X_i^2 + \sum_{ij>1}^{k} \sum_{i=1}^{k} \beta_{ij} X_i X_j + e \tag{4}$$

Where, Y is the output response (Y_1 is the COF; Y_2 is the specific wear rate); i and j denote linear and quadratic coefficients, respectively; β_0 is the intercept, β_i is the first-order model coefficient, k is the number of factors and e is a random number [32].

3. RESULT AND DISCUSSION

3.1 Model Evaluation Using ANOVA

The interaction effect between the individual variables such as the applied load, sliding velocity as a function of rotational speed and refrigeration oil has significant influence on the coefficient of friction and specific wear rate. The analysis of variance (ANOVA) was used to check the adequacy and validate the model statistically for obtaining the optimum experimental conditions [33].

Table 5. Validation test Results of COF.

Source	Sum of Squares	DOF	Mean Square	F Value	p-value Prob > F	R^2	Adj. - R^2	Adequate precision (AP)
COF								
Model	0.001444	11	0.0001	24.45	< 0.0001	0.9641	0.9247	18.16
A-Load	0.000218	1	0.0002	40.53	< 0.0001			
B-Speed	4.94E-08	1	4.94E-08	0.0092	0.9254			
C-Refrigeration oil	7.93E-05	2	3.96E-05	7.38	0.0107			
AB	1.37E-06	1	1.37E-06	0.2554	0.6242			
AC	0.00035	2	0.0001	32.61	< 0.0001			
BC	0.000499	2	0.0002	46.46	< 0.0001			
A2	0.000525	1	0.0005	97.78	< 0.0001			
B2	5.72E-05	1	5.71E-05	10.65	0.0085			
Residual	5.37E-05	10	5.36E-06					
Lack of Fit	3.57E-05	5	7.13E-06	1.98	0.2354		Not significant	
Pure Error	0.000018	5	3.60E-06					
Cor Total	0.001498	21						

Table 6. Validation test Results of SWR.

Source	Sum of Squares	DOF	Mean Square	F Value	p-value Prob > F	R^2	Adj. - R^2	Adequate precision (AP)
Specific wear rate (mm^3/Nm)								
Model	9.84E-08	11	8.95E-09	8.49	0.0010	0.9033	0.7969	10.12
A-Load	1.69E-08	1	1.69E-08	16.03	0.0025			
B-Speed	8.44E-10	1	8.44E-10	0.8013	0.3917			
C- Refrigeration oil	2.63E-08	2	1.31E-08	12.46	0.0019			
AB	7.9E-10	1	7.9E-10	0.7496	0.4069			
AC	2.18E-09	2	1.09E-09	1.03	0.3913			
BC	1.36E-08	2	6.81E-09	6.46	0.0158			
A2	2.33E-08	1	2.33E-08	22.15	0.0008			
B2	3.02E-09	1	3.02E-09	2.86	0.1211			
Residual	1.05E-08	10	1.05E-09					
Lack of Fit	6.49E-09	5	1.3E-09	1.60	0.3089		Not significant	
Pure Error	4.05E-09	5	8.1E-10					
Cor Total	1.09E-07	21						

Table 7. Quadratic model equations for COF and SWR

Responses	Model Equation
COF	
POE68	$0.076333-0.01034\cdot Load+3.69E\text{-}05\cdot Speed+1.71E\text{-}07\cdot Load\cdot Speed+0.000506\cdot Load^2-2.7E\text{-}08\cdot Speed^2$
BIO	$0.058603-0.01012\cdot Load+6.38E\text{-}05\cdot Speed+1.71E\text{-}07\cdot Load\cdot Speed+0.000506\cdot Load^2-2.7E\text{-}08\cdot Speed^2$
BIO50	$0.063869-0.00831\cdot Load+2.81E\text{-}05\cdot Speed+1.71E\text{-}07\cdot Load\cdot Speed+0.000506\cdot Load^2-2.7E\text{-}08\cdot Speed^2$
Specific wear rate (mm^3/Nm)	
POE68	$0.000557-5.7818E\text{-}05\cdot Load-1.9E\text{-}07\cdot Speed-4.1E\text{-}09\cdot Load\cdot Speed+3.37E\text{-}06\cdot Load^2+1.98E\text{-}10\cdot Speed^2$
BIO	$0.000518-5.339E\text{-}05\cdot Load-2.4E\text{-}07\cdot Speed-4.1E\text{-}09\cdot Load\cdot Speed+3.37E\text{-}06\cdot Load^2+1.98E\text{-}10\cdot Speed^2$
BIO50	$0.000641-5.9816E\text{-}05\cdot Load-3.6E\text{-}07\cdot Speed-4.1E\text{-}09\cdot Load\cdot Speed+3.37E\text{-}06\cdot Load^2+1.98E\text{-}10\cdot Speed^2$

The actual values of SWR and COF obtained from the experimental runs and the predicted values were calculated based on the model. Table 5 and 6 depict the sum of squares, degrees of freedom (DOF), F value, R^2, adjusted R^2 and Adequate Precision of COF and SWR. The adequacy of the model is ensured by the F value of 24.45 for COF and 8.49 for SWR. The value of "Probability > F" less than 0.0500 indicates that model terms are significant. The chance of 0.01% of "F-Value" of the model could be largely attributable to noise. The range of predicted value of the independent variables at the design points to the average prediction error evaluated the adequate precision ("Adeq Precision"). The signal to noise ratio (Adeq Precision), greater than 4 for both COF and SWR confirms the desirability of the model [26]. The "Lack of fit F-value" of 1.98 for COF and 1.60 for SWR implies the not significant relative to the pure error. Table 7 shows the actual terms of factors of the final quadratic models which correspond to COF and SWR.

3.2 Optimization of Tribological Parameters Using Desirability Analysis

Desirability analysis is a mathematical technique to acquire the optimum value. Desirability is based on the design that the process has multiple quality characteristics for several responses and factors. This technique determines operating conditions X that endow with the most desirable response values. For each response Y, a desirability function d(X) consigns numbers between zero and one to the possible values of Y. The objective of the desirability approach has to maximize the overall desirability D concerning the controllable factors [34]. Fig. 4(a-c) depicts the 2D contour plots for desirability, COF, and Specific wear rate.

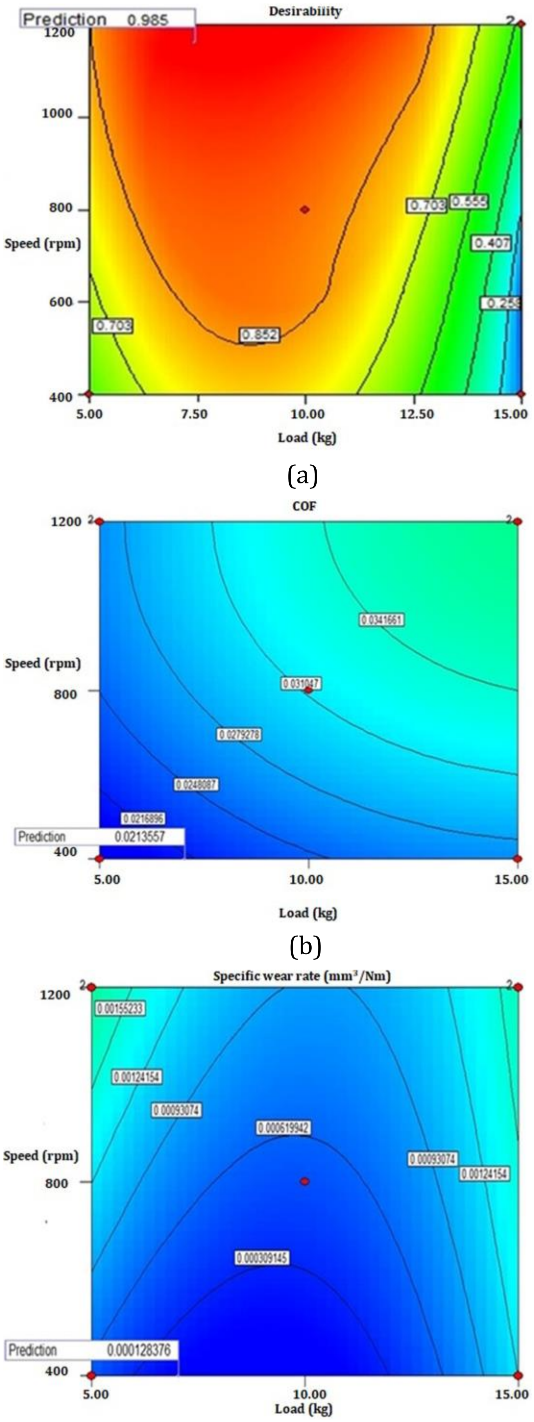


Fig. 4 (a) 2D contour plot of desirability, (b) 2D contour plot of COF, (c) 2D contour plot of specific wear rate

Table 8. Goals Set and Levels Used For Desirability Analysis

Parameters	Goal	Lower level	Upper level	Lower wgt	Upper wgt	Importance
Load in N	Is in range	50	150	1	1	3
Rotational speed in rpm	Is in range	400	1200	1	1	3
Refrigeration oil	Is in range	POE68	BIO50	1	1	3
COF	Minimize	0.029855	0.060937	1	1	3
Specific wear rate (mm^3/Nm)	Minimize	0.000022	0.000041	1	1	3

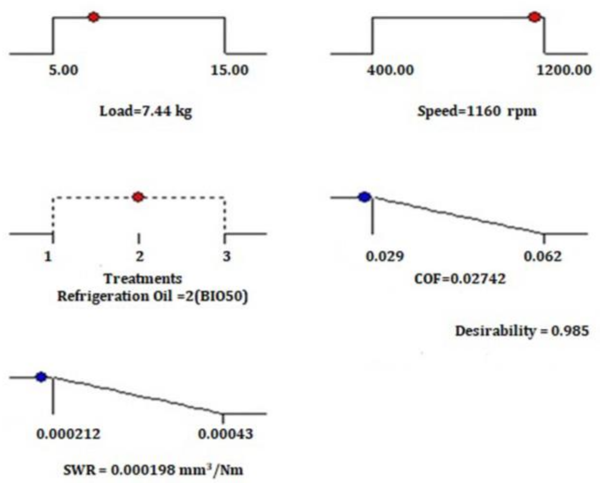


Fig. 5. Ramp function graph

The overall desirability for this optimization is 0.985. All responses predicted were within the desired limits. The overall results from the 2D contour plots concluded that the load of 7.44 kg, the rotational speed of 1160 rpm, refrigeration il of BIO50 are necessary to minimize the COF of 0.02742 and specific wear rate of 0.000198 mm^3/Nm. Fig. 5 shows the ramp function graph that represents the effect of the individual input variables such as load, rotational speed and refrigeration oil on output responses. The dot on the each graph specifies the desired input parameters consequent to the lower COF and specific wear rate. The goal set, lower limits used, upper limits used, weights used, and importance of the factors are shown in Table 8.

3.3 Confirmation Test

Results of the developed statistical models of D-optimal design were also validated by accomplishing confirmation tests. The validation of the confirmation tests was carried out triplicate and the average values were stated.

Table 9. Results of confirmation test

Optimum Setting level	Optimal output parameters	
	Predicted	Experimental
Load: 7.5 kg Rotational speed:1160 rpm Refrigeration oil :BIO50		
Coefficient of friction	0.02742	0.0279
Specific wear rate (mm^3/Nm)	0.000198	0.000216

The performed experimental results closely matched with the predicted values. This result provided the necessary support in the validation of the model and the developed statistical model was found suitable for predicting the tribological behaviour of biodegradable refrigeration oil. Table 9 represents the results of confirmation tests.

3.4 Effect of Tribological Characteristics on Output Responses

3.4.1 Friction Coefficient

A 3D response graph in Fig. 6(a-c) shows the coefficient of friction variation concerning the rotational speed and load applied for the different refrigeration oils. It can be noted from the Fig. 6(a-c) that the COF decreases with the load applied and rotational speed and starts to rise after reaching the minimum level with an increase in rotational speed and load applied for the all refrigeration oils. It can be seen from the Fig. 6(a-c) that the combination of cast iron compressor pin material/BIO50 shows off the lowest COF with all the experimented refrigeration oils endowed with a spread of the better protective layer on the surface. Fatty acids present in vegetable oil based refrigeration oil are long, straight-chain carboxylic acids which act as an active protecting layer against sliding surfaces and offer superior tribological properties [7, 35]. This superior frictional behaviour of cast iron compressor pin material/BIO50 is mainly attributed to lower frictional heating and lesser contact temperature.

Mostly the friction reduction behaviour of vegetable oil-based polyolester is principally persuaded by the existence of the saturated fatty acids as compared to POE68. However, as counterpart the presence of unsaturated fatty acid (oleic acid) in polyolester is unfavourable for its anti-friction ability and hence blended refrigeration oil, BIO50 is surpassed as compared to POE or BIO. The author assumed that, the reason for increased frictional behaviour at higher load and speed, the regime of lubrication changes from being hydrodynamic lubrication regime into boundary lubrication regime at increased load and speed, rather than under optimum conditions. The effectiveness of boundary lubrication is strongly dependent on contact temperature [4].

Additionally, cast iron compressor pin material / BIO50 shows the superior tribological behaviour in terms of lowest COF at higher sliding speed and load owing to lower temperature rise which leads to the lowest frictional heating. The lowest COF of 0.02742 is registered at optimal operating conditions, at a load of 7.44 kg, the speed of 1160 rpm and BIO50 refrigeration oil, whereas for the same optimal conditions, the COF is reported as 0.047428 and 0.043361 for the cast iron pin lubricated by POE68 and BIO type of refrigeration oil respectively.

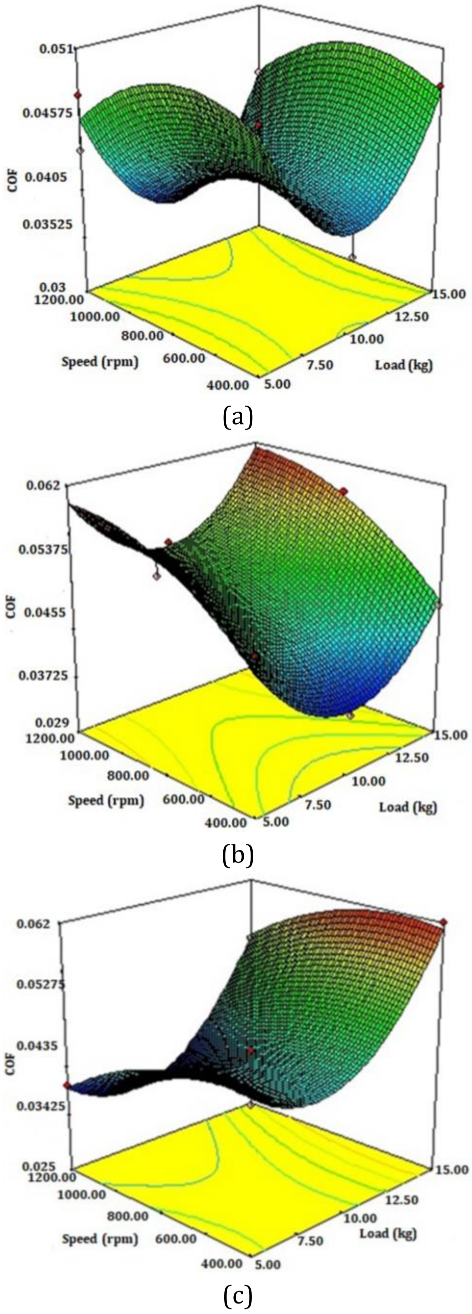
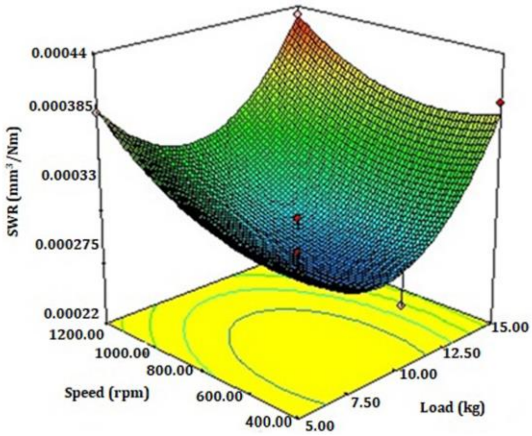


Fig. 6 (a) 3D response plots for COF of POE68, (b) 3D response plots for COF of BIO, (c) 3D response plots for COF of BIO50

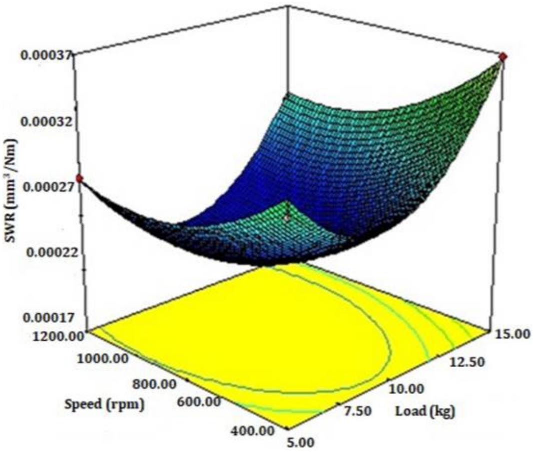
3.4.2 Specific Wear Rate

A 3D response graph shown in Fig. 7(a-c) portrays the specific wear rate variation concerning the rotational speed and applied load for the different refrigeration oils with the combinations of cast iron compressor pin material. It was noticed from Fig. 7(a-c) that the SWR decreases with the applied load and rotational speed. Further starts to increase after the optimal level with rise in applied load and rotational speed. The reason is due to wear, besides being a material intrinsic property, it is also a complex function of operating conditions involving load applied, speed, contact geometry, surface roughness, temperature and lubrication environment [36]. The combination of cast iron compressor pin material/BIO50 exhibits the preeminent anti-wear behaviour among all of the tested refrigeration oils. This preeminent anti-wear behaviour of cast iron compressor material/BIO50 has predominantly attributed to the steady-state conformity of the contacting surfacing under BIO50 lubrication environment, which leads the effective macro-contact conditions making the surface smoother in reducing wear than lubricated under POE68 or BIO.

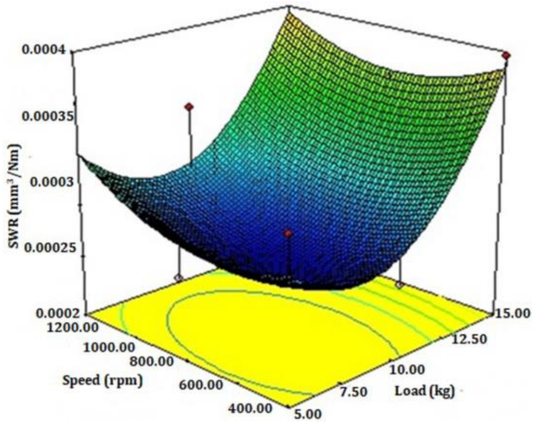
Additionally, cast iron pin/BIO50 shows the superior tribological behaviour in terms of lowest SWR at higher speed and load. The reason for this is because thicker lubricant film produced by blended refrigeration oil leads to improved wear-protection performance. Nevertheless, at the same load and speed, POE68 or BIO shows higher specific wear rate due to the lower kinematic viscosity compared to BIO which in turn generates higher thermal energy increase which leads to a thinner boundary layer formed between the pin and disc contact that eventually elevates wear. Aziz et al. [37] reported that the increase in applied load and higher sliding velocity, SWR increases owing to the breakdown of the oil-film. The lowest SWR of 0.000198mm^3/Nm is registered at optimal operating conditions, at a load of 7.44 kg, the speed of 1160 rpm and BIO50 refrigeration oil, whereas for the same optimal conditions, the specific wear rate is reported as 0.00038mm^3/Nm and 0.00032mm^3/Nm for the pin material lubricated by POE68 and BIO type of refrigeration oil respectively.



(a)



(b)

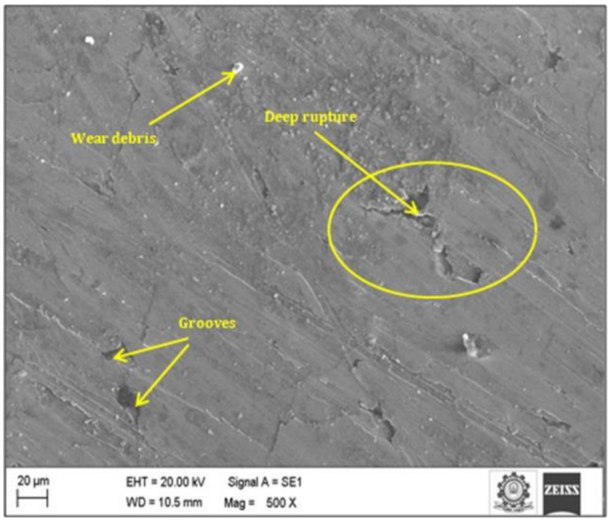


(c)

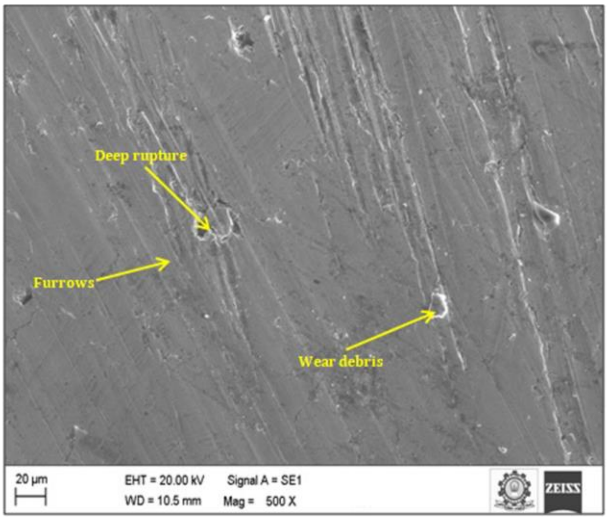
Fig. 7 (a) 3D response plots for SWR of POE68, (b) 3D response plots for SWR of BIO, (c).3D response plots for SWR of BIO50

3.4.3 SEM Analysis

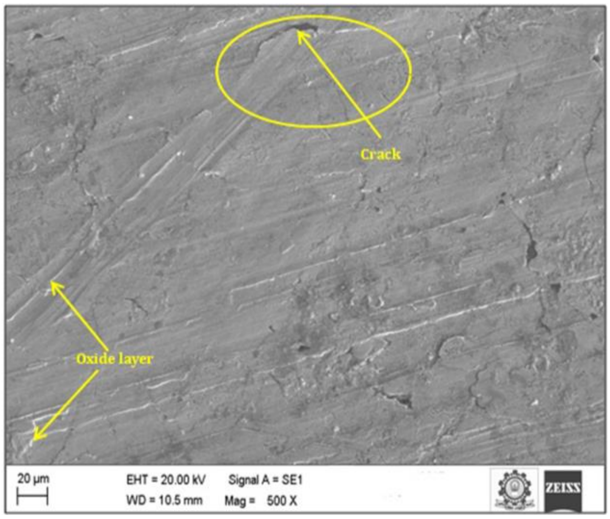
The combined SEM/EDX analysis was done by using FEI Quanta 650 FEG microscope. Fig. 9 shows the surface morphology analysis on the pin surfaces tested under optimized experimental conditions lubricated by POE68, BIO and BIO50.



(a)



(b)



(c)

Fig. 8 (a) SEM image of Compressor pin material lubricated by POE68, (b) SEM image of Compressor pin material lubricated by BIO, (c) SEM images of Compressor pin material lubricated by BIO50

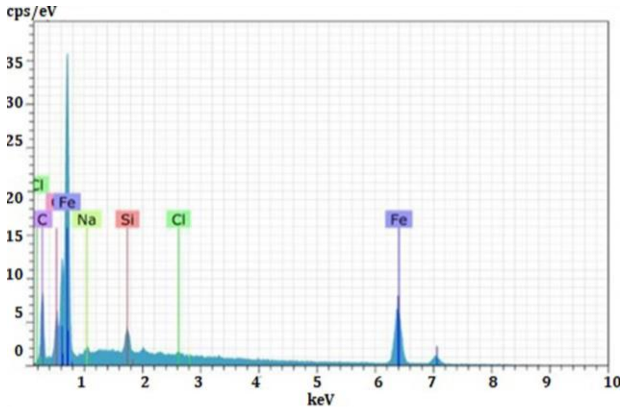
The surface deformation for all refrigeration oil samples indicates adhesive wear. The furrows, scratches, pits, cavities are severe, and had more deformation in the case of POE68, whereas in the compressor pin material under the influence of BIO50 showed lesser voids, wear debris with minor scratches on the wear track and indicated improved anti-friction and anti-wear behaviour. The reason for this was due to attachment of molecules to the engaged mating surface by the polar carboxyl group to form a protective layer reducing the asperity contact surface as well as reducing the friction and wear which resulted in the reduction of thermal energy and enhanced the lubricity [38].

3.4.4 Elemental Analysis of Pin Surfaces

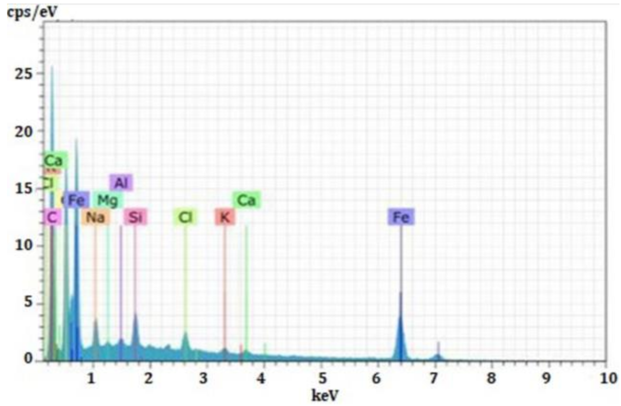
Fig. 9(a-c) illustrates the energy-dispersive X-ray spectra image of the pin surfaces lubricated with POE68, BIO and BIO50 refrigeration oils. Table 10 depicts the summary of the elements existed under the influence of refrigeration oils over the pin surfaces. The details of elemental weight percentages of the FG 150 grey lamellar graphite cast iron tested surfaces under the influence of refrigeration oil samples were obtained from EDX elemental spectra. From the findings of EDX, it is perceived that refrigeration oil samples contain an assortment of elements of base metal such as iron and alloying elements like carbon, oxygen, Sodium, Chlorine, Calcium, Silicon, Magnesium, Aluminium, Potassium and Phosphorus. The presence of oxygen in BIO50 is conducive to the development of oil-film on the interaction surface. The thick protective layer attributes the friction reduction on the engaging matting surface and also welded to enhanced adhesive wear.

Table 10. Elemental summary of EDX Spectra

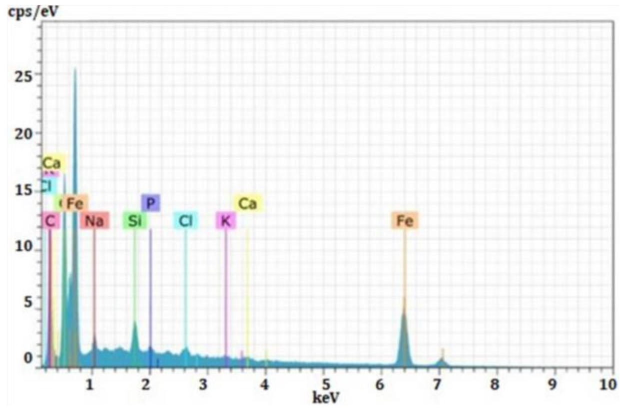
Wt. %	Refrigeration oil		
	POE 68	**BIO**	**BIO50**
Carbon	29.21	42.18	50.16
Oxygen	6.71	19.35	20.30
Sodium	0.96	1.34	1.18
Magnesium	-	0.03	-
Aluminium	-	0.08	-
Silicon	1.52	1.31	1.56
Chlorine	0.17	0.71	0.36
Potassium	-	0.21	0.11
Calcium	-	0.23	0.18
Ferrous	60.81	34.48	26.04
Phosphorus	-	-	0.12



(a)



(b)



(c)

Fig. 9 (a) EDX spectra of compressor pin material lubricated by POE68, (b). EDX spectra of compressor pin material lubricated by BIO, (c). EDX spectra of compressor pin material lubricated by BIO50

The reason for this is due to pentaerythritol ester composition consisting of triacylglycerols made up of esters and presence of long chains of polar fatty acids is responsible for adhering to metallic surfaces results in tightly packed protective layer [4, 37]. Further, in BIO50, the higher content of carbon increases the surface bonding, which covers the pin surface leading to the wear reduction.

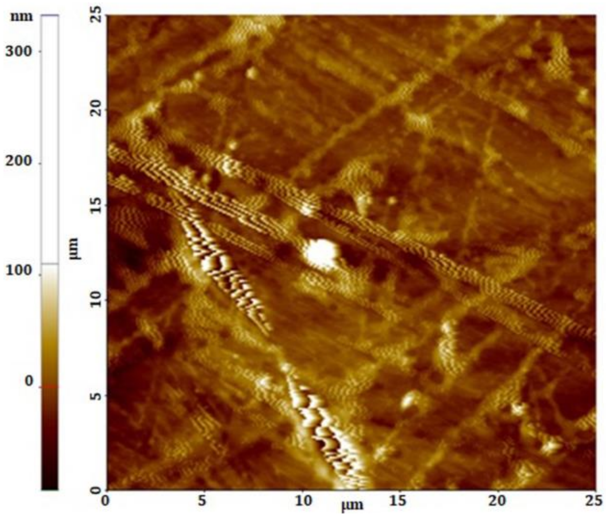
3.4.5 AFM Analysis of Pin Surfaces

The roughness parameters of post tested pin samples were measured at optimal test conditions as reported in Table 11 using atomic force microscopy (AFM) technique, Model: NTEGRA PRIMA, NTMDT, Ireland. At the same operating conditions, the tests were repeated for BIO and POE68 grade refrigeration oil in addition to BIO50 in order to analyse the influence of refrigeration oil on tribological behaviour. The corresponding two-dimensional AFM image and roughness parameters are depicted in Fig. 10(a-c) and Table 11 respectively. XEI program is used to translate the obtained roughness data into an image.

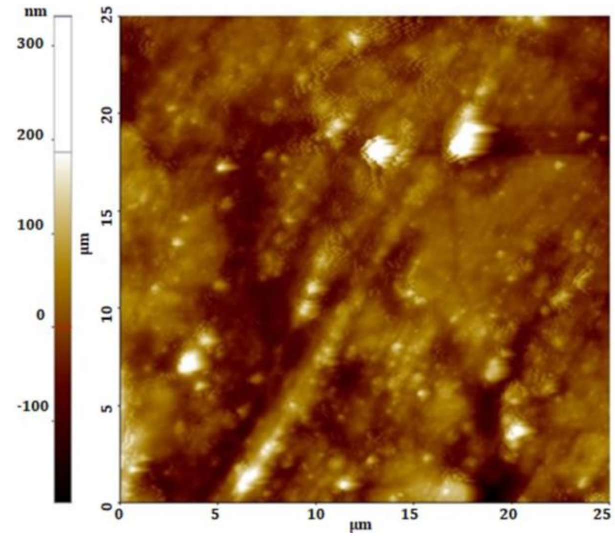
Table 11. Roughness parameters of post tested pin surface

Roughness Parameters (nm)	Surface lubricated with		
	POE 68	BIO	BIO50
Rq	23.002	17.818	12.863
Ra	14.833	13.772	9.968
Rz	344.532	154.544	146.469

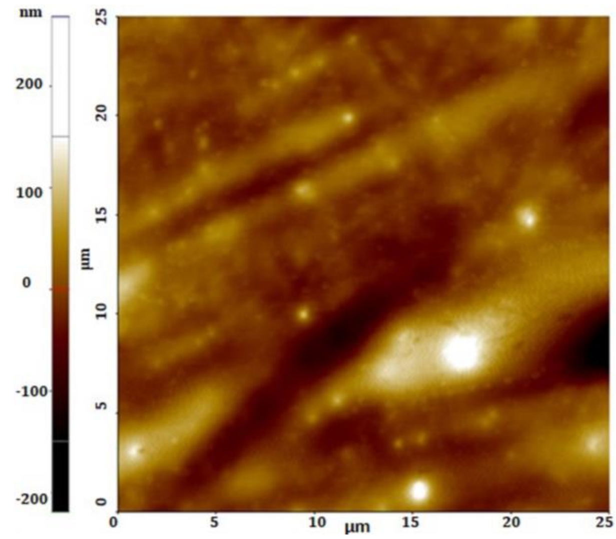
The surface roughness was characterized by the parameters of roughness: average roughness (Ra), root mean square roughness (Rq), ten-point average roughness (Rz). From 2-D AFM image, it is witnessed that the lower average roughness values are registered for tested pin surface lubricated by BIO50 of about 9.968nm as compared to BIO and POE68 grade refrigeration oils for which Ra is 13.772 nm and 14.883 nm respectively.



(a)



(b)



(c)

Fig. 10 (a) AFM images of Compressor pin material lubricated by POE68, (b) AFM images of Compressor pin material lubricated by BIO, (c) AFM images of Compressor pin material lubricated by BIO50

4. CONCLUSIONS

The tribological behaviour of vegetable oil based polyolester refrigeration oil in refrigeration compressor materials was examined using a pin-on-disc tribometer. Based on the experimental investigation, the findings are summarized as follows:

1. Response surface-based methodology with desirability approach is suitable to improve the tribological characteristics of rapeseed oil based polyolester refrigeration oil.

2. The ideal conditions to achieve the minimum COF and SWR are load of 7.44 kg, the rotational speed of 1160 rpm and blended refrigeration oil. At this ideal experimental condition, the COF and SWR of 0.02742 and 1.98x10-4mm^3/Nm are reported respectively.

3. From the pin-on-disc test, the BIO50 exhibited excellent tribological behaviour in terms of lower COFand low specific wear rate. The existence of fatty acids/anti-wear additive in BIO50 is jointly accountable for reducing the COF and SWR.

4. The SEM/EDX analysis supported the improved tribological behavior of the BIO50. The furrows, scratches, pits, cavities are severe, and more deformation in the case of POE68, whereas in the compressor pin material under the influence of BIO50 showed lesser voids, wear debris with minor scratches on the wear track and indicated improved anti-friction and anti-wear behaviour.

5. The average roughness parameter of the pin surfaces lubricated with BIO50 is reduced by 27.6% and 32.8% as that of BIO and POE68.

6. This outcome of the study delivered the authentication in the model validation in forecasting the tribological behaviour of vegetable oil based polyolester refrigeration oil.

On the whole, this finding recommends that rapeseed oil-based polyolester has a substantial prospect in becoming substitution refrigeration oil for the domestic refrigerator. In spite of this, the utilization of test rig in this study suggests a basic tribological test which attempts to replicate the vital factors in the refrigerator. Further, the customization of point contact does not replicate the real contact of compressor's ring/liner which limits the scope of the present study for real- time applications.

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