



## An investigation on the optimization of anti-wear performance of nano-Fe<sub>3</sub>O<sub>4</sub> based ferro-magnetic lubricant

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KEYWORDS	ABSTRACT
Fe <sub>3</sub> O <sub>4</sub> Lubricant Nano Four-ball Wear Magnetic	<p>In the current study, an optimized nano- Fe<sub>3</sub>O<sub>4</sub> based ferro-magnetic lubricant was developed by using mechanical mixing technique. Fe<sub>3</sub>O<sub>4</sub> nanoparticles in 1, 3, 5 and 7 weight percentages were dispersed in group-2 N500 base oil containing 5 wt % of oleic acid as surfactant. The stabilities of the lubricants were evaluated by calculating their sedimentation over of a period of 196 days. The mechanical mixing speed, time and the weight fraction of nano- Fe<sub>3</sub>O<sub>4</sub> particles were optimized by using Taguchi's L16 orthogonal array. The wear scar diameter obtained in the ASTM D4172-B four-ball test was considered as the output response in the optimization. The ANOVA test revealed the maximum influence of mixing speed on the wear scar diameter followed by the time and concentration. Finally, the optimum parameter setting of 1 wt. % nano- Fe<sub>3</sub>O<sub>4</sub> particles, 3000 rpm and 15 mins of mixing time was obtained. Thereafter, the anti-wear performance of the optimized ferro-magnetic lubricant was studied under the variable magnetic field (0-54 mT) for 15 minutes. It was observed that at 26 mT magnetic field intensity the wear scar diameter was minimum and a reduction of approximately 35 % in the wear scar diameter was observed as compared to the base oil.</p>

Received 15 February 2020; received in revised form 16 March 2020; accepted 5 May 2020.

To cite this article: Chouhan et al. (2020). An investigation on the optimization of anti-wear performance of nano-Fe<sub>3</sub>O<sub>4</sub> based ferro-magnetic lubricant. Jurnal Tribologi 25, pp.119-135.

## 1.0 INTRODUCTION

Many significant technological advancements have occurred in the field of tribology. However, the quest is never ending and efforts are still being made to improve the tribological performance of the materials. Reducing the friction helps in minimizing the energy losses while preventing the wear results in the increased service life of machines (Shoeab and Mishra 2014; Singh, Vimal, and Chaturvedi 2012). Several attempts have been made to reduce the friction and wear by using conventional oils or by adding the oil-soluble additives in the base oils. However, these existing lubricants have reached the maximum limits of their performance characteristics (Weijiu Huang et al. 2002; Li et al. 2000). Therefore, the search for novel and high-performance lubricants has led many researchers to explore the use of nanoparticles (magnetic and non-magnetic) as additives in the existing lubricants (Dai et al. 2016; Gulzar et al. 2016). These lubricants are called nano-lubricants and have gained popularity owing to their improved performance. There are three system components in nano-lubricants; base oil, nano-particles and a surfactant (Bakunin et al. 2005). The surfactant is used to create an interface between the nanoparticles and base oil. Magnetic nanoparticles are the most promising material among the various nanoparticles to reduce the friction and wear due to their unique magnetic properties. This is due to the fact that their flow characteristics in the base oils can be adjusted by using an external magnetic field (Wei Huang and Wang 2016). Furthermore, these particles are insoluble in nonpolar base oils. These particles have also lower chemical activity with other additives present in the base oil. Because of its nano-metric size, it allows them to enter the contact area between the frictional surfaces, thereby improving the lubricity. The uniform and stable dispersion of magnetic nanoparticles in a base oil constitutes to a magnetic lubricant. In tribology, the main applications of magnetic fluids as lubricants are in the fluid seals, bearings, gears, etc. (Odenbach 2003; Uhlmann et al. 2002).

Many of the researchers have focused on studying the effects of different process parameters (speed, loading factors, and mixing ratio, etc.) on the tribological characteristics (coefficient of friction, wear scar area, wear rate, and wear scar diameter etc.) of the ferro-fluids with the help of four-ball tester (FBT). (Trivedi, Parekh, and Upadhyay 2017) have discussed the performance of  $\text{Fe}_3\text{O}_4$  magnetic nanoparticles (MNPs) (synthesized by using a chemical co-precipitation method) dispersed in a gear oil containing alpha-olefin hydrocarbon. Their results showed that the coefficient of friction (COF) and wear scar diameter (WSD) were reduced by 45 % and 30 %, respectively, at 4 % by weight optimum concentration of magnetic nanoparticles when compared to the base oil. (Wei Huang et al. 2009) examined the tribological behaviour of liquids containing  $\text{Fe}_3\text{O}_4$  nanoparticles in various concentrations with the help of a FBT. Their results revealed that the WSD was decreased by 28.30 % as compared to the carrier oil, and the relative ratio of the COF was reduced by 31.3 % respectively. The optimum content of MNPs in the base oil was about 4 % by weight. (Li-jun, Chu-wen, and Yamane 2008)(Li-jun et al. 2009) investigated the tribological properties of the nano-ferromagnetic  $\text{Mn}_{0.78}\text{Zn}_{0.22}\text{Fe}_2\text{O}_4$  as an additive in the turbine oil 46# with the help of FBT. The turbine oil 46# with 6 wt.%  $\text{Mn}_{0.78}\text{Zn}_{0.22}\text{Fe}_2\text{O}_4$  nanoparticles showed the best tribological properties among the tested oil samples, and a decrease of 25.45 % in the wear scar was observed as compared to the base oil. And under the effect of a magnetic

field, turbine oil 46# containing 6 wt.%  $Mn_{0.78}Zn_{0.22}Fe_2O_4$  showed much better friction and wear-resistant properties as compared to the non-magnetic lubricant. (Xiang et al. 2014) discussed the tribological and tribochemical properties of magnetite ( $Fe_3O_4$ ) nano-flakes used as additives in #40 base oil by using the FBT. The average COF for the friction pair under the lubricant containing  $Fe_3O_4$  nano-flakes of 1.5 wt. % was found to be decreased by 18.06 % as compared to that of the base oil. (Suhane et al. 2017) have performed the optimization of MNPs based lubricant by using Taguchi's approach, taking into account the influence of the significant variables such as loading factors, speed and mixing ratio. By using the Taguchi optimization technique, an optimal combination of these process factors was obtained. Tribological parameters such as COF and wear were examined on a FBT.

The Taguchi's design of experiments technique is a potential tool for designing and enhancing the quality of any process or product at a very low cost. Taguchi had introduced a term called 'loss function' to estimate the response characteristics migrating from the desired target. The term 'loss function' has now changed into the signal-to-noise ratio (S/N ratio) concept. The S/N ratios for all the output responses of the parametric settings are calculated. Finally, the optimum parametric setting is one which has superior S/N ratio. However, the Taguchi approach is used to optimize only single response because the high S/N ratio of one individual response may correspond to a smaller S/N ratio for different responses. (Sindhu, Thakur, and Chandna 2018).

From the current literature survey, it has been studied that only few studies have been conducted on the evaluation of tribological properties of nano- $Fe_3O_4$  based ferro-magnetic lubricants (FMLs) under externally applied magnetic field. There is also a scarcity of work on the optimization of synthesis parameters and the nano- $Fe_3O_4$  particle concentration in base oil for minimum wear scar diameter (WSD) by using the Taguchi approach. Therefore, in the current study, the efforts were made to optimize the concentration of nano- $Fe_3O_4$  particles in group-2 N500 base oil along with the synthesis parameters (stirrer mixing speed and mixing time) for obtaining the minimum WSD concurrently via Taguchi method. The effects of nano- $Fe_3O_4$  particle concentration by wt.% in base oil, mechanical mixing speed and mixing time on the WSD of steel ball tested in FBT were studied and the prediction of minimum WSD was also performed. The stability of developed nano- $Fe_3O_4$  based FMLs was measured over a period of 196 days by calculating their sedimentation percentages. Furthermore, the optimized nano- $Fe_3O_4$  based ferromagnetic lubricant was subjected to the varying external magnetic field for studying its effect on the WSD of the steel balls tested over FBT.

## 2.0 EXPERIMENTAL DETAILS

### 2.1 Materials and Synthesis of Ferromagnetic Lubricant

$Fe_3O_4$  magnetic nanoparticles (MNPs) of size 10-30 nm were procured from *Nano Research lab, India*. Table 1 and Table 2 shows the physical characteristics and chemical composition as per the supplier's claim. Oleic acid (OA), acetone, and de-ionized water were procured from *LOBA Chemie, India*. The group-2 N500 base oil (BO) was obtained from *Fresh Lube, India*, for the preparation of

FMLs. Firstly, the group oil was tested in a FBT in order to determine its anti-wear performance, as per the ASTM D4172-B standard. Thereafter, oleic acid was added to the base oil in different proportions (0%, 1%, 3%, 5% and 7% weight percentages) and the solutions were tested again in FBT in order to determine their anti-wear performance. This was specially done to determine the optimum proportion of the oleic acid to be used as potential additive and a surfactant (Wei Huang et al. 2009) for the stable and uniform dispersion of the Fe<sub>3</sub>O<sub>4</sub> nanoparticles in the base oil.

Table 1: Physical characteristics of Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

Characteristics	Fe <sub>3</sub> O <sub>4</sub>
Colour	Black
Purity (%)	>99.5
Size (nm)	10-30
True density (g/cm <sup>3</sup> )	4.8-5.1
Morphology	Spherical
Specific surface area (SSA),(m <sup>2</sup> /g)	20-60

Table 2: Composition of Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

Fe <sub>3</sub> O <sub>4</sub>	S	Ni	Mg	Al
>99.5	<0.1	<0.02	<0.04	<0.20

Thereafter, Fe<sub>3</sub>O<sub>4</sub> MNPs in different weight percentages were mixed in the base oil having an optimum proportion of oleic acid exhibiting best anti-wear properties. Prior to their mixing in the base oil solution, these Fe<sub>3</sub>O<sub>4</sub> MNPs were properly coated with the oleic acid. The MNPs were then added in a small quantity of the oleic acid and the mixture was heated at 65 °C for 10 min followed by the ultrasonic for 20 minutes. Finally, these coated nanoparticles were extracted from the excessive amount of oleic acid by a strong neodymium magnet. The coated precipitate was cleaned with de-ionized water and acetone followed by drying in the atmosphere to remove the water contents. Lastly, the coated Fe<sub>3</sub>O<sub>4</sub> MNPs were dispersed in the solution containing the optimum proportion of oleic acid as a surfactant and base oil with the help of mechanical mixing procedure.

## 2.2 Characterization

The morphology and elemental composition of the uncoated and coated Fe<sub>3</sub>O<sub>4</sub> MNPs with surfactants were studied by using scanning electron microscope (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). The X-ray diffraction patterns of MNPs were obtained by using powder X-ray diffractometer (Rigaku, model Miniflex-ii Desktop X-ray) using CuKα (λ = 0.15414 nm). The 2θ angle was varied from 15° to 75° at a scan speed of 2° min<sup>-1</sup>. The average density of the various fluids used in the study was measured at 30 °C by using a pycnometer (make Thomas Scientific). The rheological properties of the base oil and the FMLs were measured by using the Anton Paar's MCR 302 Rheometer having a spindle attachment of 2° cone angle and 0.083 mm gap. Shear rate was varied from 1 to 10<sup>3</sup> s<sup>-1</sup> at the temperatures of 40 °C and 100 °C, respectively. The stability of the developed FMLs with dispersed MNPs in the base oil (having an optimum value of surfactant) was also determined by measuring the sedimentation of MNPs over a period of 196 days. The MNPs in different wt. % were mixed in the base oil in order to develop the FMLs with the help of a mechanical mixer rotating at 3000 rev/min for 60 minutes.

### 2.3 Tribological Testing

Four-ball wear test was conducted on the base oil and modified base oils, as per the ASTM D4172-B standard on an Automatic Four-Ball Tester (DUCOM Instruments Pvt. Ltd., India). This setup uses four balls in a three down and one up configuration. The bottom three balls are held tightly in a pot containing a lubricant to be tested and are pressed against the top ball, as shown in Figure 1. At the end of each test, the average WSD formed on the three lower balls was calculated as per the method given in the test standard, with the help of an optical image analyser (having an accuracy of 0.01 mm) fitted with the FBT machine. Lastly, the average WSD was calculated for three identical tests and has been reported in the current study.

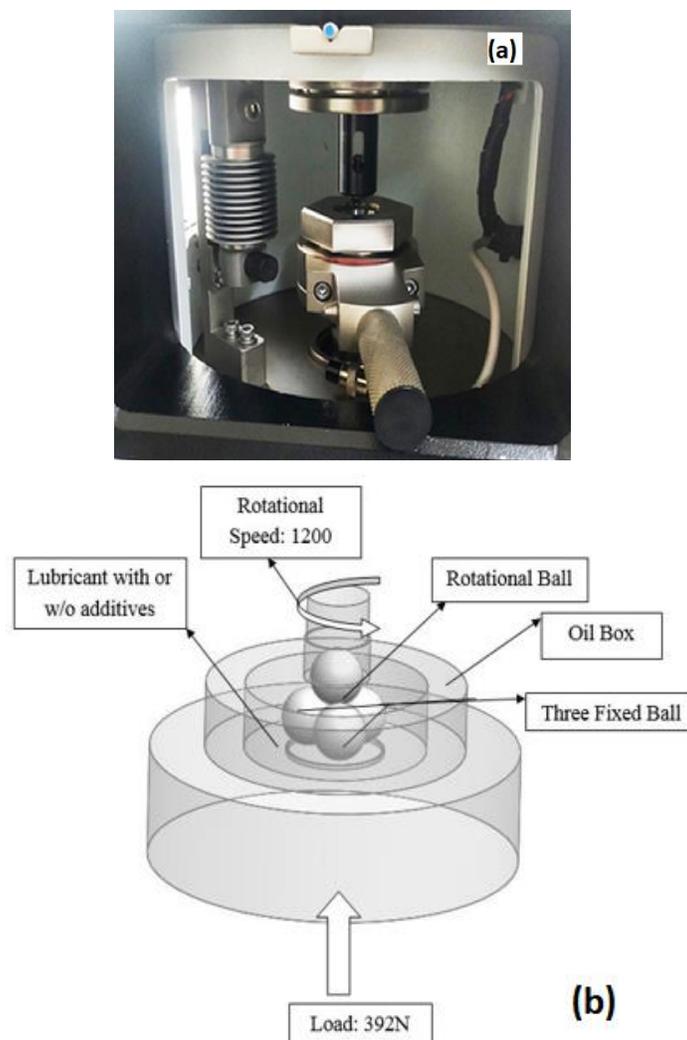


Figure 1: (a) Image showing actual four-ball tester, (b) configuration of four-ball wear test.

In order to test the developed ferro-magnetic lubricants the original four-ball tester was modified to generate a step-less adjustable magnetic field, as shown in Figure 2. The magnetic

field was generated with the help of a solenoid consisting of an enamel coated copper wire of diameter of 0.72 mm and 1350 number of turns. The coil resistance was found to be 12.9 ohms. The current and voltage flowing through the coil can be varied in the range of 0-5 A and 0-60 V. The coil was installed over the rotating spindle carrying the fourth ball. The direction of the relative velocity is perpendicular to the magnetic field at the point of contact (Figure 2). The magnetic field intensity can be adjusted by changing the value of current flowing in the coil. The rate of the magnetic field near the point of contact was measured using a Gauss-meter in a static state before starting the tribological test. The maximum magnetic field strength obtained was 166 mT. Due to the excessive heat generation in the magnetic coil during the experiment, the test time was limited to 15 minutes only.

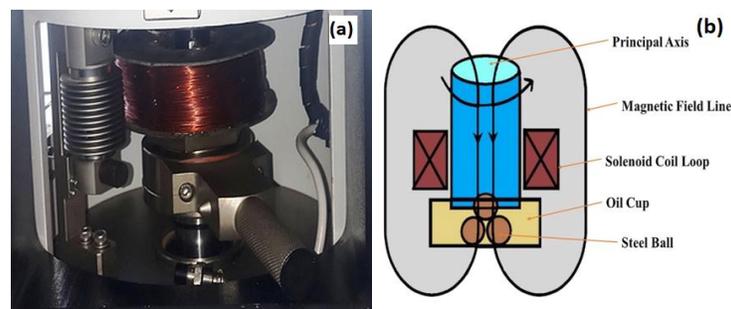


Figure 2: (a) Arrangement of solenoid in the modified four-ball tester (b) generation of magnetic field by solenoid in modified four-ball tester.

#### 2.4 Taguchi's Methodology

The proportion of MNPs in the base oil along with the parameters of mechanical mixing procedure were optimized by using Taguchi's design of experiments technique. The Taguchi's technique is a popular tool for design of experiment, which is used for collecting the data in a governed way and also to examine the influence of process parameters over the desired response (Taguchi and Rafanelli 1994; Wang, Zhao, and Yu 2005). In the present investigation, three process parameters; MNPs concentration by wt.% (with symbol A), mixing speed (with symbol B) and mixing time (with symbol C) were selected for the experimental design of optimized FML, as shown in Table 3. Three input parameters at four different levels were selected for the design of experiments. Taguchi's L16 orthogonal array was opted for the design of experiments. The WSD measured in the FBT for corresponding parametric settings was considered as the output response. The experimental results of WSD under different parametric settings were finally converted into the signal-to-noise (SN) ratios. Taguchi proposed the SN ratio for measuring the quality characteristics for different loss functions. The SN relations are mainly available in three forms, viz. smaller-is-better, larger-is-better and nominal-is-best (Blaza Stojanovic, Jasmina Blagojević, Miroslav Babic, Sandra Veličković 2017; Stojanović et al. 2018; Veličković et al. 2017). In present study, the smaller-is-better characteristic for minimizing the WSD was considered and the SN ratios for each experimental setting were calculated by using the following equation:

Table 3: Process parameters with their levels.

Symbol	Process parameters	Unit	Levels			
			1	2	3	4
A	Concentration	wt.%	1	3	5	7
B	Speed	rev/min	1200	1800	2400	3000
C	Time	min	15	30	45	60

$$\frac{S}{N} \text{ratio} (\eta) = -10 \log_{10} \left( \frac{1}{n} \sum_{i=1}^n y_{ij}^2 \right) \quad (1)$$

Where, n=number of replications  $y_{ij}$  =observed response value where, i=1, 2 ...n; j=1, 2...k

### 3.0 RESULTS AND DISCUSSION

Figure 3 (a-d) represent the SEM and EDX images of uncoated and oleic acid coated  $Fe_3O_4$  MNPs. It can be observed from Figure 3c that coating of oleic acid followed by the ultrasonication resulted in the de-agglomeration of the spherical shaped  $Fe_3O_4$  MNPs as compared to the uncoated particles (Figure 3a) which are present in the agglomerated form. The EDX report presented in Figure 3b exhibited the presence of Fe and O in the major proportions in uncoated MNPs while Mg, Ni, Al, and S are present in the form of impurities. Figure 3d represents the EDX report of coated MNPs which shows the presence of an extra carbon element due to the coating of oleic acid over the MNPs.

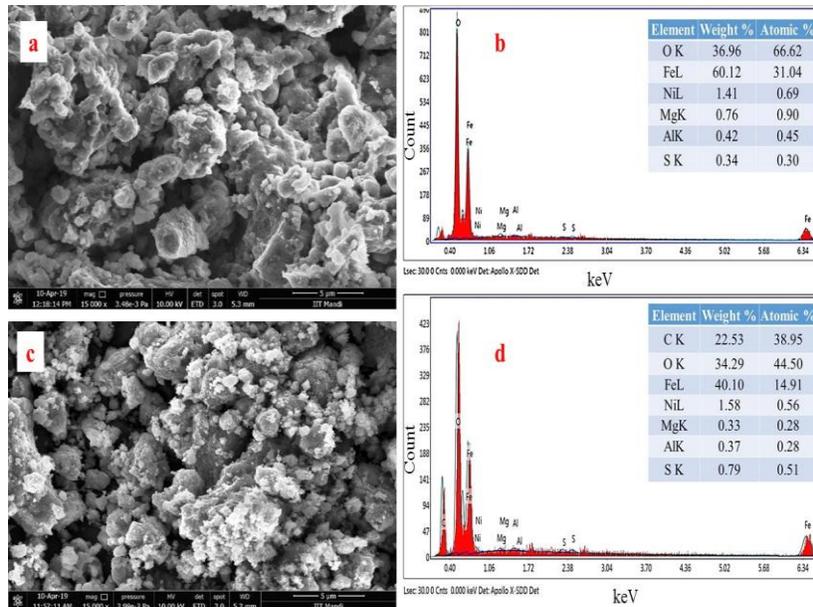


Figure 3: SEM image of (a) uncoated, (b) EDX report of uncoated, (c) oleic acid coated and (d) EDX report of coated  $Fe_3O_4$  magnetic nanoparticles.

Figure 4 represents the X-ray diffraction pattern of the Fe<sub>3</sub>O<sub>4</sub> MNPs. The material consists of the cubic crystal structure Fe<sub>3</sub>O<sub>4</sub> (JCPDS 19-629), i.e magnetite. The broadening of characteristic peaks of the resulting Fe<sub>3</sub>O<sub>4</sub> nanoparticles can be observed because of the smaller particle size. Scherer formula was used to calculate the average grain size and it was found to be 16.16 nm (Pathmamanoharan and Philipse 1998). The calculated particle size is in good agreement with the size specified by the supplier. The results of density and viscosity of the base oil and different FMLs are depicted in Table 4. It can be observed from the table that the addition of surfactant (oleic acid) and MNPs in the base has a significant effect on the density and viscosity of the base oil.

Table 4: Density and viscosity values of the base oil and different FMLs.

Specimens	Density	Viscosity	
	(g/cm <sup>3</sup> )	(mPa sec)	(mPa sec)
	30 °C	40 °C	100 °C
Base oil (G2 N500)	0.8720	100	11.83
1wt.% OA+BO	0.8716	94.98	12.09
3wt.% OA+BO	0.8709	91.46	11.30
5wt.% OA+BO	0.8702	86.22	10.93
7wt.% OA+BO	0.8697	82.39	10.50
BO+5wt.%OA+1wt.% nano-Fe <sub>3</sub> O <sub>4</sub>	0.8729	96.71	11.69
BO+5wt.%OA+3wt.% nano-Fe <sub>3</sub> O <sub>4</sub>	0.8858	96.84	11.70
BO+5wt.%OA+5wt.% nano-Fe <sub>3</sub> O <sub>4</sub>	0.8909	98.89	12.08
BO+5wt.%OA+7wt.% nano-Fe <sub>3</sub> O <sub>4</sub>	0.9000	99.27	12.25

BO=Base oil (G2 N500), OA=Oleic Acid

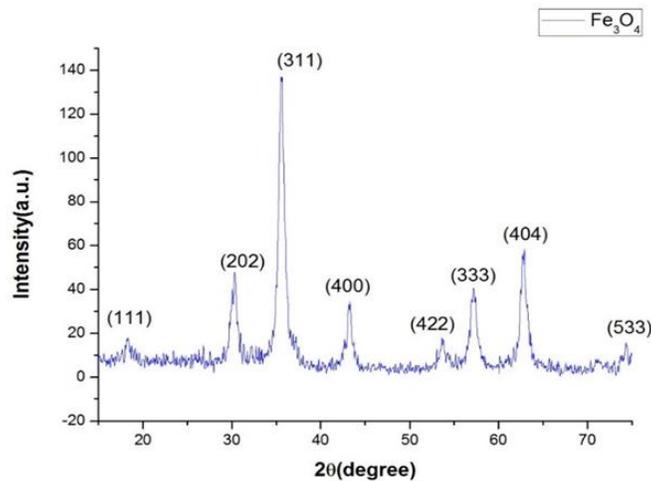


Figure 4: XRD pattern of the Fe<sub>3</sub>O<sub>4</sub> MNPs.

The tribological performance of the base oil and modified oils having different proportions of the oleic acid is presented in Figure 5(a-d) and Figure 6. The oleic acid in the base oil was added in different weight percentages (0%, 1%, 3%, 5% and 7%) in order to determine its optimum proportion to be used as an effective additive and surfactant for the uniform dispersion of MNPs

in the base oil. It can be observed from the Figures that the sample having 5 wt. % of oleic acid in base oil has shown minimum average WSD (marked on the optical image) as compared to the other samples. The sample containing the oleic acid with 5 wt. % concentration in the base oil showed a reduction in average WSD by 23% (approx.) as compared with the base oil. Therefore, the optimum concentration of oleic acid was found to be 5 wt. % in the base oil.

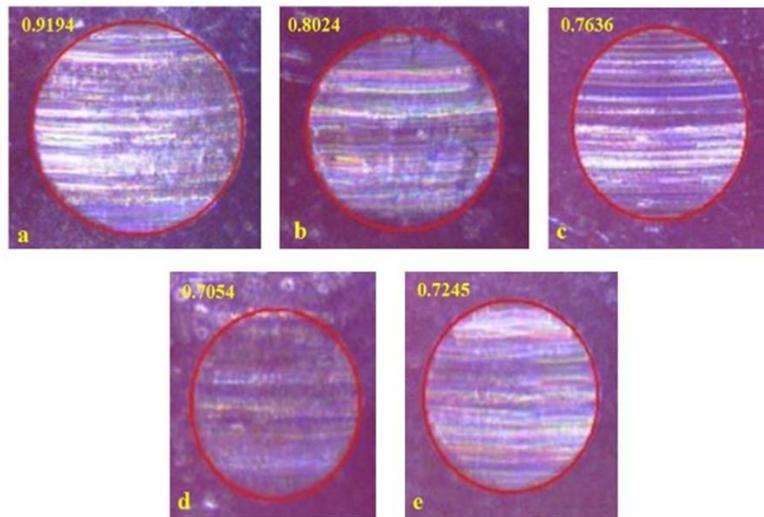


Figure 5: Optical image showing wear scars of (a) base oil (BO), (b) 1 wt. % OA+BO, (c) 3 wt. % OA+BO, (d) 5 wt. % OA+BO and (e) 7 wt. % OA+BO samples.

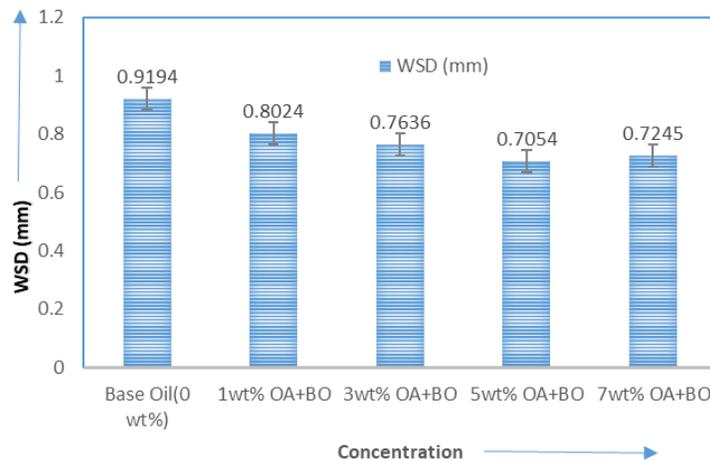


Figure 6: Wear scar diameter as a function of wt.% of surfactant in the base oil.

The improved wear performance of the modified base oil may be attributed to the molecular structure of oleic acid having a carboxylic group as the polar head and a long oil-soluble hydrocarbon tail. The polar end is attached to a surface by chemically or physically reacting with the oxide present on metal surfaces. The tail of hydrocarbons is normal to the metal surface and

is aligned with other tails and may form single layer or multi layers which can slide easily over each other to reduce the friction and thus better protects the surface from wear (Guo et al. 2017). The samples containing the oleic acid in concentrations of 1, 3, 5 and 7 wt. % also exhibited some dark spots on the worn-out surface of tested balls. These dark spots are probably caused by the polar end (carboxylic group) of the oleic acid, which chemically reacts with oxide present on the surface of the balls. The area covered by these dark grooves increases with the increasing concentration of oleic acid (Han et al. 2019).

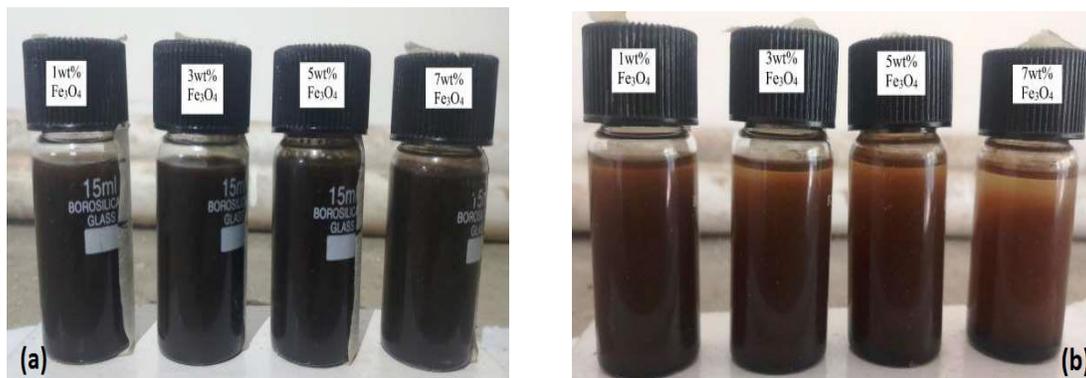


Figure 7: Stability of FMLs having different wt. % of MNPs at (a) 0<sup>th</sup> (b) 196<sup>th</sup> day.

For checking the stability of nano-Fe<sub>3</sub>O<sub>4</sub> based FMLs, four different samples having MNPs in the concentrations of 1 wt. %, 3 wt. %, 5 wt. % and 7 wt. % in the base oil (having 5 wt. % proportion of oleic acid as surfactant) were investigated. Four vials containing different concentration of nano-Fe<sub>3</sub>O<sub>4</sub> based FMLs were kept in the lab environment without any disturbance and exposure to the direct light, as shown in Figure 7(a & b). Their stabilities (%) in terms of the sedimentation were calculated over a period of 196 days. (Huang et al. 2009) had evaluated the stability of their lubricants for maximum of 10 days. While, (Shi, Huang, and Wang 2018; Trivedi, Parekh, and Upadhyay 2017) had conducted their stability tests for maximum of 30 and 60 days, respectively. The sedimentation was obtained by visualizing the interface between the supernatant and the concentrated suspension. It can be observed from Figure 8 that FML based on 1 wt. % nano- Fe<sub>3</sub>O<sub>4</sub> showed the lowest sedimentation as compared to the FMLs containing Fe<sub>3</sub>O<sub>4</sub> MNPs in 3 wt. %, 5 wt. % and 7 wt. % respectively. The stability of 1 wt. % nano-Fe<sub>3</sub>O<sub>4</sub> based FML was found to be approximately 73 %, which has shown a good potential of commercial viability.

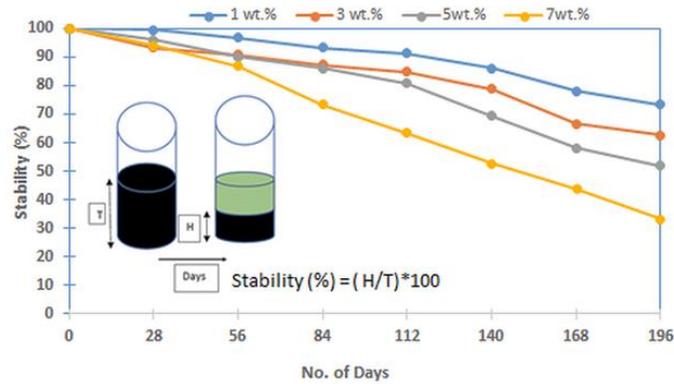


Figure 8: Stability behaviour of nano- Fe<sub>3</sub>O<sub>4</sub> based FMLs.

As discussed in the previous section, the results of parametric study using the Taguchi's L16 orthogonal array is presented in Table 5. A total of 16 parametric combinations were performed to develop the ferromagnetic lubricants and their performances in terms of the corresponding average WSDs was evaluated. The output responses of WSD were converted in the SN ratios. Thereafter, the experimental results were analysed with the help analysis of variance (ANOVA), which presented the influence of process parameters (concentration, speed and time) and their interactions on the WSD of tested ferromagnetic lubricant samples. The ANOVA was conducted for a confidence level of 95 %. Table 6 shows the ranking of process parameters based on the SN ratio of measured WSD and the different levels of these process parameters. The rankings of process parameters were calculated in the basis of delta value.

Table 5: Experimental design matrix using L16 orthogonal array with response test results.

Run order	Concentration (wt. %)	Speed (rev/min)	Time (min)	Wear Scar Diameter (mm)	SN ratio (db)
1	1	1200	15	0.7034	3.05595
2	1	1800	30	0.6615	3.58940
3	1	2400	45	0.8212	1.71102
4	1	3000	60	0.7558	2.43186
5	3	1200	30	0.7754	2.20948
6	3	1800	15	0.7046	3.04115
7	3	2400	60	0.9496	0.44919
8	3	3000	45	0.6857	3.27732
9	5	1200	45	0.8173	1.75237
10	5	1800	60	0.8164	1.76194
11	5	2400	15	0.8032	1.90353
12	5	3000	30	0.6931	3.18408
13	7	1200	60	0.8633	1.27677
14	7	1800	45	0.7555	2.43531
15	7	2400	30	0.8217	1.70573
16	7	3000	15	0.7187	2.86905

Table 6 shows the rank of process parameters without their interactions on the average WSD of samples tested under ferromagnetic lubrication. The results of ANOVA are presented in Table 7. It is observed that mechanical mixing speed (rev/min) has the highest influence on WSD which is followed by time (min), while the concentration (wt. %) has the least influence on WSD. The percentage contribution of each process parameter is shown in the Table 7.

Table 6: Ranking of significant process parameters based on delta values.

No.	Parameters	Level 1	Level 2	Level 3	Level 4	Delta	Rank
1	Concentration by weight %	2.697	2.244	2.150	2.042	0.625	3
2	Speed (rev/min)	2.074	2.707	1.442	2.941	1.498	1
3	Time (minutes)	2.717	2.672	2.294	1.480	1.237	2

Table 7: Response Analysis of Variance for Means of WSD.

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Percentage contribution
Conc. (wt. %)	3	0.007229	0.007229	0.002410	3.87	0.075	8.18
Speed (rev/min)	3	0.044338	0.044338	0.014779	23.72	0.001	50.22
Time(min)	3	0.032976	0.032976	0.010992	17.64	0.002	37.35
Residual Error	6	0.003739	0.003739	0.000623			4.23
Total	15	0.088282					

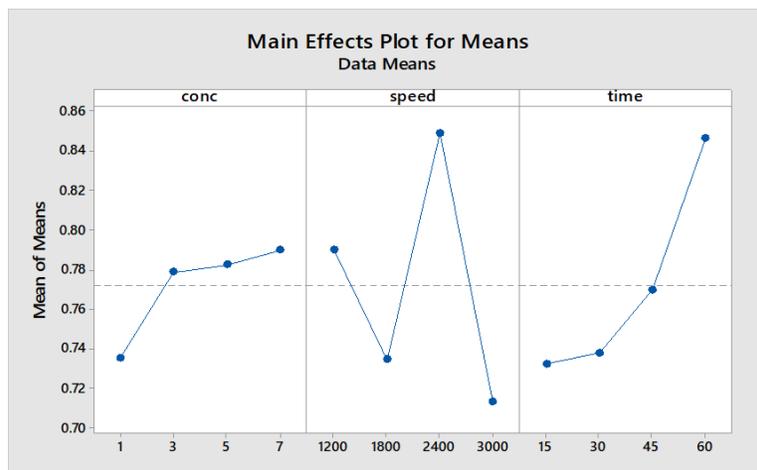


Figure 9: Mean S/N ratio response graph for WSD.

By analyzing the percentage contribution, it can be observed that the mechanical mixing speed exhibited the highest contribution of 50.22%, followed by the time and concentration by weight

of MNPs in the contributions of 37.35 and 8.18%, respectively. The other interactions were not considered as they were statistically insignificant owing to their P-values above 0.05. The results shown in Table 6 are graphically presented in Figure 9, which show the main effect plots of SN ratios. If the curve of a particular process parameter is closer to the horizontal, then its influence is small. Whereas, the higher inclination of curve from the horizontal shows its significant influence on the output response. With the consideration of these rules, it can be inferred that a combination of process parameters with levels A1: B4: C1 will give minimum WSD. It means that the minimum WSD could be predicted for the MNPs concentration of 1 wt. %, mixing speed of 3000 rev/min and stirring for a time of 15 minutes. With these predicted values of control factors the confirmatory experiment was carried out to evaluate the anti-wear performance of developed ferromagnetic lubricant. The results of process parameter optimization are presented in Table 8. Thereafter, the anti-wear performance of the developed optimized FML (with 1 wt.% of MNPs) was evaluated under the application of magnetic field with the help of modified four-ball tester. Figure 10 represents the wear behaviour of the optimized FML under the varying magnetic field intensity.

Table 8: Validation of optimized result.

	Optimum control factors	
	Optimized Predicted value	Confirmatory Experimental value
Level	Concentration-1 wt. %, speed-3000 rpm and time-15 minutes	Concentration-1 wt. %, speed-3000 rpm and time-15 minutes
Signal to Noise Ratios (WSD)	0.6380	0.6250
Net % error between optimized and experimental value		2.06%

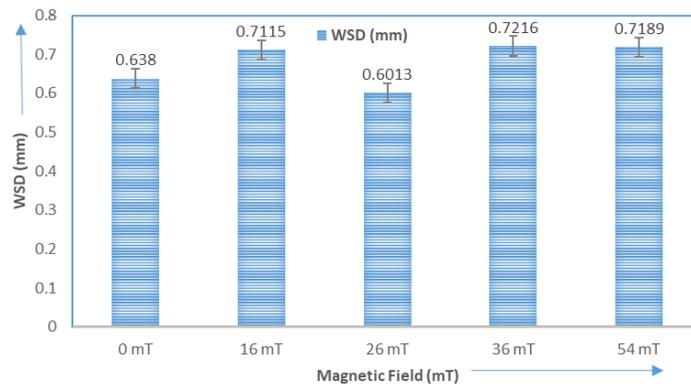


Figure 10: Wear behaviour of the optimized FML under varying magnetic field intensity.

Figure 11 shows WSD images of balls lubricated with the optimized nano-Fe<sub>3</sub>O<sub>4</sub> based FML when the magnetic field intensity was varied from 0 to 54 mT. The minimum WSD was found to be 0.6013 mm at 26 mT of magnetic field intensity. A reduction of 5.73% in WSD of tested balls

was observed as compared to the sample tested without the application of magnetic field. The overall reduction in WSD with optimized FML (in the presence of magnetic field) was found to be 35% (approx.) as compared to that of the base oil. On further increasing the magnetic field, the WSD again increases and the surface becomes rough and parallel groove appears on the surface as shown in Figure 11 (d). The mechanism for this observed behaviour is explained as follows: In the absence of a magnetic field, the particles are evenly dispersed in the carrier base oil (Figure 12 (a)), and the rheological behaviour of FML with just 1 wt. % of  $\text{Fe}_3\text{O}_4$  nanoparticles is expected to be similar to the base oil. At this moment, the  $\text{Fe}_3\text{O}_4$  nanoparticles will act like spacers to eliminates the metal-to-metal contact between the steel balls and will also contribute in the rolling friction. Therefore, these effects will reduce the shear stress between the contacting surfaces and eventually the friction and wear will be reduced (Li-jun, Chu-wen, and Yamane 2008).

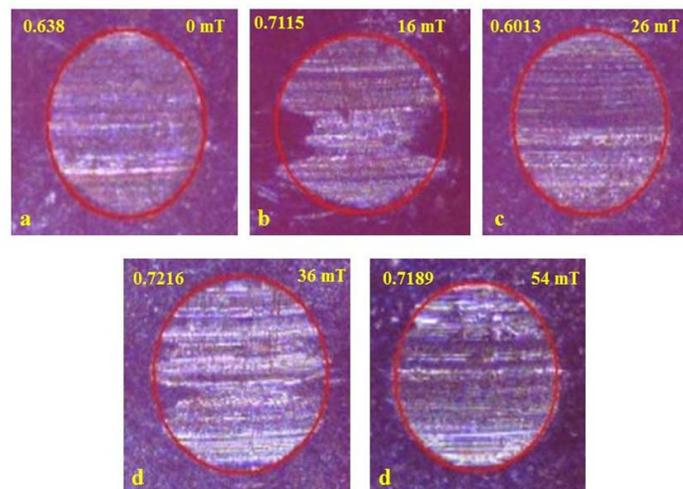


Figure 11: Optical images of WSD of optimized FML under varying magnetic field intensity.

In the presence of a magnetic field (Figure 12 (b)), the nanoparticles will be attracted to this external field and then align themselves to form chain like structures in the base oil, which increases the viscosity of the lubricant. Consequently, an increase in the viscosity will help to reduce the direct contact between the metal balls and reduce the friction and wear significantly (Pereira et al. 2012; Pervaiz and Gul 2013; Pileni 2003). Figure 12 (c) shows that on increasing the magnetic field above a threshold value will cause the agglomeration of magnetic nanoparticles, which behave like abrasives between the contacting surfaces. As a result, wear of the steel balls will be increased (Li-jun et al. 2009).

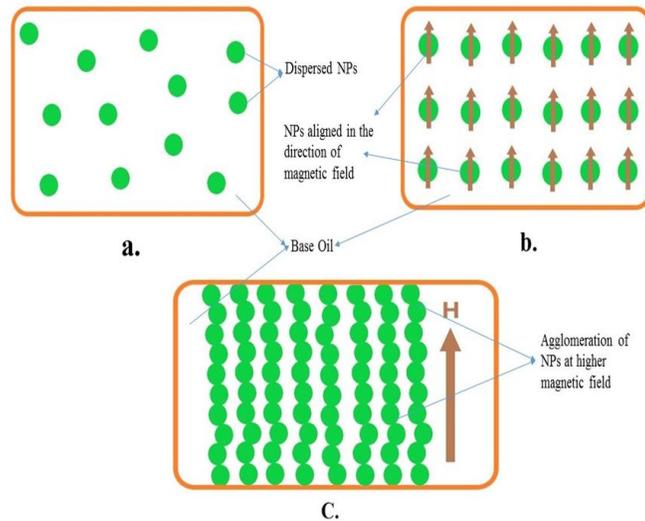


Figure 12: Dispersion of magnetic particles in base oil under (a) no magnetic field (b) optimum magnetic field intensity and (c) and higher magnetic field intensity.

#### 4.0 CONCLUSIONS

The main objective of the present research was to develop an optimized FML and to study its wear performance under varying magnetic field. For this purpose, Taguchi's L16 orthogonal array was selected for the experimental design and optimization of lubricant composition and mechanical mixing process parameters. The following conclusions were drawn from the present research work.

- (a) The optimum 5 wt.% concentration of oleic acid as surfactant improved the wear performance of selected group-2 N500 base oil and also helped in the uniform dispersion of MNPs. The improved performance of base oil may be attributed to the formation of thin layers of over the metal surface which can glide over each other, thus, reduces the friction between the surfaces.
- (b) The ANOVA study revealed that mechanical mixing speed is the most dominant parameter followed by mixing time and MNPs concentration by weight.
- (c) The optimum parametric setting was found to be 1 wt.% concentration, mixing speed of 3000 rev/min, and mixing time of 15 minutes.
- (d) The stability of FML having 1 wt.% concentration of MNPs was found to be highest amongst all FMLs.
- (e) The optimized FML having just 1 wt.% MNPs exhibited a significant reduction of 35% in WSD under the applied magnetic field intensity of 26 mT as compared to WSD obtained with base oil.
- (f) Under the applied magnetic field, the MNPs dispersed in the base oil align themselves in the direction of externally applied magnetic field and form spacer like structure between

the rubbing surfaces. These MNPs also act as nano-size rolling elements which helps in reducing the friction and wear of the surfaces in contact. At higher magnetic field intensities these MNPs get agglomerated and behave like abrasives between the interacting surfaces which results in the increased wear.

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