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Research Article

Optimizing halloysite nanotubes addition for improving tribological performance of castor seed oil

Sachin GHALME[1,*], Mohammad Athar HAYAT[2], Mahesh HARNE[3]

[1]Department of Mechanical Engineering, Pravara Rural Engineering College, Maharashtra, 413736, India
[2]Department of Mechanical Engineering, St. Vincent Pallotti College of Engineering and Technology, Maharashtra, 441108, India
[3]Department of Mechanical Engineering, Amrutvahini College of Engineering, Maharashtra, 422608, India

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ABSTRACT

Depleting sources of mineral-based oils and environmental issues are pushing the research community for the development of sustainable, environmentally friendly bio-lubricants. However, the primary obstacle to the use of bio-lubricants in industrial applications is their limited lubricating capacity. Nanomaterials are major additives for lubricating oil to improve their lubricating properties. Halloysite nanotubes (HNTs) are naturally available, non-toxic nano-material. In this study, Halloysite nanotubes in five different proportions were blended with castor seed oil for enhancement of tribological properties. The interaction effect of load and Halloysite nanotube addition is considered for improving the load-carrying capacity and frictional property of castor seed oil. The recently proposed Best Holistic Adaptable Ranking of Attributes Technique (BHARAT) is implemented to obtain the optimal level of load and Halloysite nanotubes addition. Experimental results of tribological tests are analysed with the BHARAT approach proposing a 600N load and 1.5% Wt. of Halloysite nanotube addition in castor seed oil is optimum to improve the tribological properties of castor seed oil. The outcome of this study presents an environmentally friendly, sustainable bio-lubricant with improved tribological properties. The article also gives us insight into the innovative BHARAT approach for optimization in tribological studies.

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INTRODUCTION

Lubrication is a process to minimize wear and friction between two contacting surfaces during their static or dynamic condition using different kinds of lubricants. During their application, lubricants have a crucial role in terms of supporting load, minimizing friction and wear by generating a lubricant film between contacting surfaces, preventing surface corrosion by reducing oxidation, and heat dissipation, acting as a sealant agent and restricting foreign particles from entering the system [1-4]. Mineral oils are mainly used for various industrial applications. During use, the release of mineral oil in terms of mist, spills, and

*Corresponding author.
*E-mail address: sachinghalme@hotmail.com

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droplets gets mixed with soil and water, creating a threat to the environment and ecosystem. Toxic mist generated from mineral oils also enters the human body through the skin and respiration system. Depleting sources of mineral oil and their environmental effects is a major concern of study for the research community [5-7]. The dumping of mineral oil wrongly leads to [8]:

- Rise of sewage treatment cost,
- Damage to marine life and the environment,
- Increased air pollution and deteriorating air quality.

With a wide range of applications and several products, the global lubricant market is a growing industry. Lubricants are pivotal for minimizing friction in mating parts, leading to reduced power loss due to friction, improving efficiency and extending life. Lubricants significantly affect the overall efficiency of global industry. The world automotive industry consumes a major portion of the total global lubricant demand. The growth in the global lubricant industry is also driven by the growing popularity of renewable energy sources. This will increase the demand for lubricants in the coming years. As shown in Figure 1 [9], there was a remarkable surge in the total global demand for lubricants, reaching an unprecedented peak of 36.5 million metric tonnes in 2020. This figure is expected to remain high, sailing around 37.4 million metric tonnes by 2023. By 2025, it is expected that the global market will reach a valuation of 182.6 billion USD.

Realization of environmental issues and day-by-day depletion of fossil fuels have encouraged the use of alternative bio-based oils. The biodegradability of bio-lubricants can reduce the risk of environmental damage. In contrast, traditional lubricants with complex synthetic chemical additives remain in the environment for a long duration, creating environmental pollution. Bio-lubricants are biodegradable and decompose easily in the environment and are non-harmful to the environment. They minimize the risk of air, water and soil pollution occurring in case of spills or leakages, making them eco-friendlier. The various natural resources like vegetable oils and animal fats are used for the preparation of bio-lubricants [10, 11].

Bio-based lubricants are emerging as strong alternatives to mineral-based oils due to their biodegradability, renewability, non-toxicity, and eco-friendliness. Various studies already have been already published on the potential application of bio-based stock for industrial applications such as palm oil (greases), olive oil (engine oils), coconut oil (engine oils), jojoba oil (greases, lubricants), castor oil (greases, lubricants), and crambe oil (greases) [12]. The various bio-stocks proposed for industrial lubricants are rice bran oil [13], rubber tree seed [14], sesame oil [15], castor oil [16, 17], coconut oil [18], jatropha oil [19], and date seed oil [20].

Castor oil is a non-edible oil produced with the seeds of the Ricinus Communics L Plant and cultivated all around the world in 30 countries, with India, Africa, and South America being major producers [21]. Castor seed oil, with its rich fatty acid content, good lubricating properties, high viscosity, low pour point, and no-drying characteristics, makes it a more favourable bio-based stock for industrial lubrication [22]. Various studies have reported the tribological performance of castor oil individually or in collaboration with other bio-based oils. Hernández-Sierra et al. [23] evaluated castor oil as a bio-lubricant for friction and wear performance of AISI4140 steel and reported satisfactory lubricating performance of castor oil in comparison with reference lubricating oil. Ahmed et al. [24] conducted

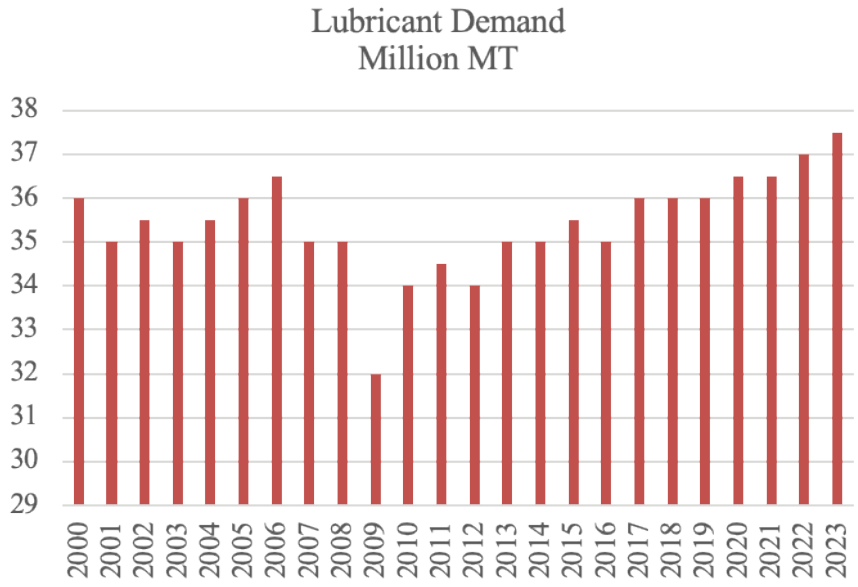


Figure 1. Lubricant demand from 2000 to 2020, with a projection for 2023 [9] [Open Access].

tribological tests for castor oil blended with date seed oil and HNTs as nano-additives; the study proposed an improved bio-lubricant with the addition of HNTs.

Although bio-lubricants are sustainable and economical alternatives to mineral oils, they are poor in hydrolytic, oxidative, and thermal stability and need improvement [25]. Various studies reported the blending of nanoparticles with lubricating oil to improve the tribological properties [26-30]. Despite their applicability in different fields, nanoparticles are harmful in nature [31]. Demand for sustainable development prompted the research community to look for alternative environmentally friendly nanoparticles as additives for bio-lubricants. In this study, an attempt has been made to propose a completely sustainable bio-lubricant of castor seed oil blended with HNTs as nano-additives. Nanomaterials of clay minerals are naturally occurring and are non-hazardous [32]. HNTs are dioctahedral 1:1 clay minerals of the kaolin group with higher water content and were first proposed by Berthier to honour its discoverer, Omalius d'Halloy [33]. Halloysite contains a monolayer of water molecules between every two adjacent alumina-silica double layers; thus its chemical formula is Al_2 Si_2O_2 (OH)4•nH_2O (n=4 for the fully hydrated form) [34].

Sneha et al. [35] conducted tribological tests using a four-ball tester for rice bran oil with turmeric oil and HNTs blend. The addition of HNTs and turmeric oil was found to improve the oxidation stability and load-carrying capacity of rice bran oil. Laura Peña-Parás et al. [36] evaluated the tribological role of polymeric lubricants for metal forming applications with varying proportions of HNTs; the study reported a reduction in wear volume loss and CoF with the addition of HNTs. Ahmed et al. [24] evaluated the effectiveness of HNTs addition for the tribological performance of date seed and castor seed oil; this study also reported improvement in the tribological properties of bio-lubricant with the addition of HNTs.

Going through these studies, it is clear that HNTs are naturally available nanomaterials and will contribute significantly to the development of completely sustainable, environmentally friendly bio-lubricants. Different researchers have investigated the effect of HNT addition on the tribological performance of bio-lubricants. The present study aims to understand the optimum proportion of HNTs addition with castor seed oil in relation to the applied load. The recently proposed BHARAT approach has been implemented to obtain the optimum proportion of HNTs and load for improved tribological performance of castor seed oil.

MATERIALS AND METHODS

Materials Used

Base Oil: The Castor seed oil was used as base oil for the study and was procured from the NIE-Centre for Renewable Energy and Sustainable Technology, Mysore, India. Table 1 presents below the physical properties of castor seed oil.

Table 1. Physical properties of castor seed oil

Property	Value
Colour	Golden yellow
Density at 26^0C	959 kg/m^3
Viscosity at 40 °C	281.8 mm^2/s
Viscosity at 100 °C	72.53 mm^2/s
Flash point	190 °C
Pour point	2.7 °C
Refractive index at 40 °C	1.48

Table 2. Physical properties of HNTs

Property	Value
Typical specific surface area	65 m^2/g
Pore volume	1.25 mL/g
Refractive index	1.54
Specific gravity	2.53 g/cm^3
Refractive index at 40 °C	1.48
average size	30–70 nm × 1–3 μm

Nano-additives: The HNTs were procured from Sigma-Aldrich. The physical properties are presented in Table 2.

Figure 2 presents the SEM micrograph of HNTs as used for this study.

Method

To understand the interactive effect of parameters involved in the study and to conduct a systematic study, the experiments were planned with the Design of Experiments

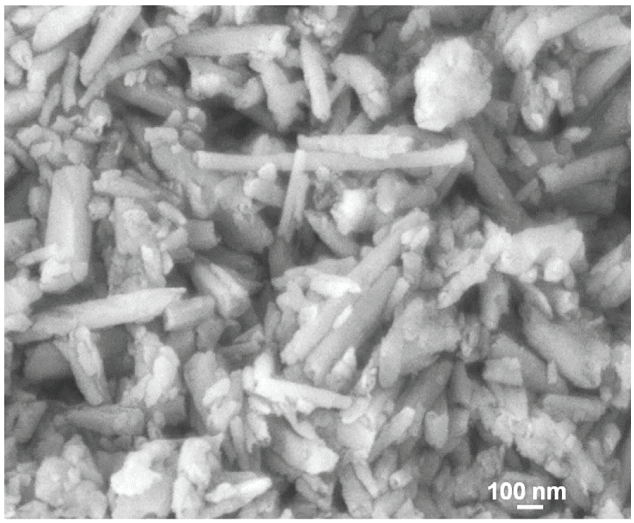


Figure 2. SEM image of HNTs used as additives.

Table 3. Experimental factors and levels

Factors	L1	L2	L3	L4	L5
Load (N)	200	300	400	500	600
%Wt. of HNTs	0	0.5	1.0	1.5	2.0

(DoE)-Taguchi method [37-39]. Load and HNTs were considered as two factors selected at five different levels for study as presented in Table 3.

Sample preparation: Five different proportions of HNTs, as presented in Table 3, were blended with castor seed oil. 1% of oleic acid as a dispersant agent was added to oil samples to avoid the agglomeration of HNTs. The oil samples were magnetically stirred at 300 rpm for 30 minutes and ultrasonicated for 30 minutes to achieve a homogeneous blend of castor seed oil and HNTs. The stability of samples was observed for 24 hrs and then used for testing. The addition of oleic acid results in micelle formation and restricts the HNTs to form a cluster and minimize the agglomeration.

Tribological Testing: The experiments were planned according to the DoE orthogonal array, considering two factors at five levels. L25 OA was selected for experiment conduction. The tribological tests were conducted with a Ducom TR 30L four-ball tester. A four-ball tester is preferable to understand the characteristics of the lubricant under test. During testing, balls made from AISI E52100 steel with a 12.7 mm diameter and a hardness of 64-66 HRC were used. The TR 30L machine is equipped with WinDucom software that manages data acquisition and the display of results. The Ducom four- ball tester and schematic arrangement for loading are shown in Figure 3. To evaluate the wear-preventive characteristics of bio-lubricant, ASTM D 4172-94 was followed [40]. Each test was conducted at a rolling speed of 1200 rpm, bath temperature of 75^{0}C, test duration of 60 min and as per load defined in Table 3. The four-ball tester comes with a data acquisition system to record the coefficient of friction, and later wear scar diameter was measured with an image acquisition system. Each test was conducted two times, and average results were recorded.

RESULTS AND DISCUSSION

During the tribological test, coefficient of friction (CoF) was recorded online and wear scar diameter (WSD) was measured after the test using image acquisition system with the help of an optical microscope available with setup. Figure 4 shows the sample optical micrographs for WSD generated after the test 1 (Load: 200 N & % Wt. HNTs: 0) and test 20 (Load: 500 N & % Wt. HNTs: 20). Optical

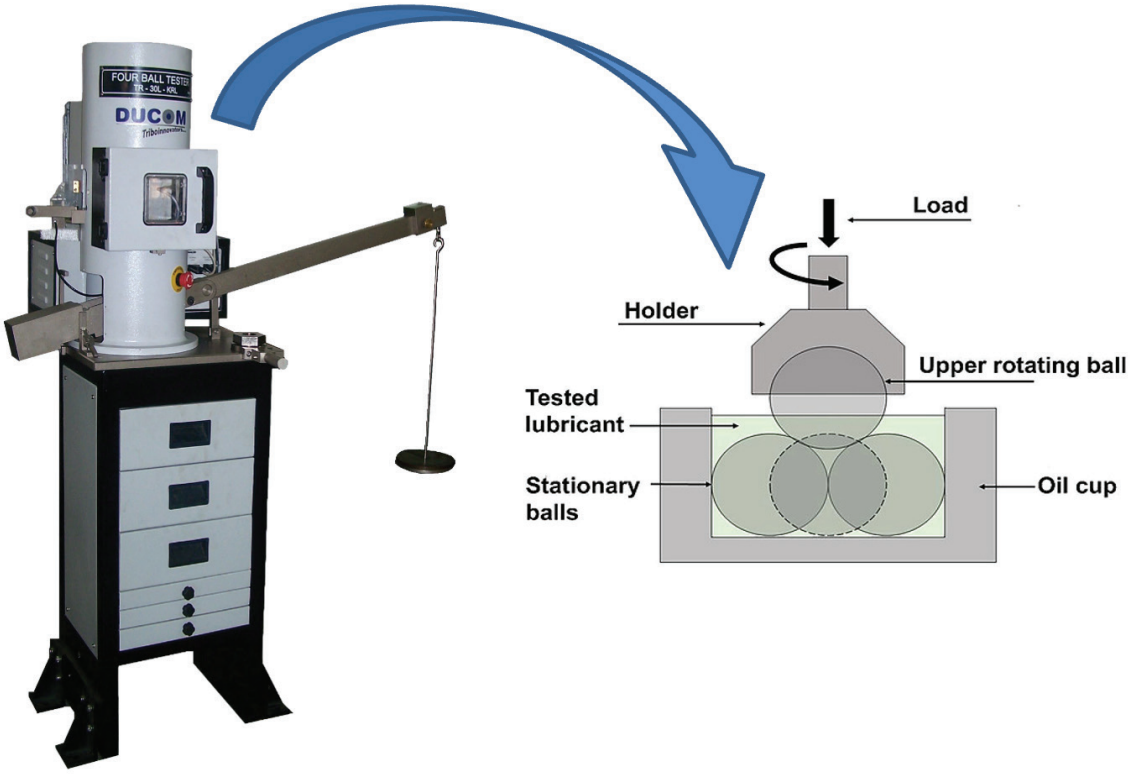


Figure 3. Four ball tester and schematic loading arrangement.

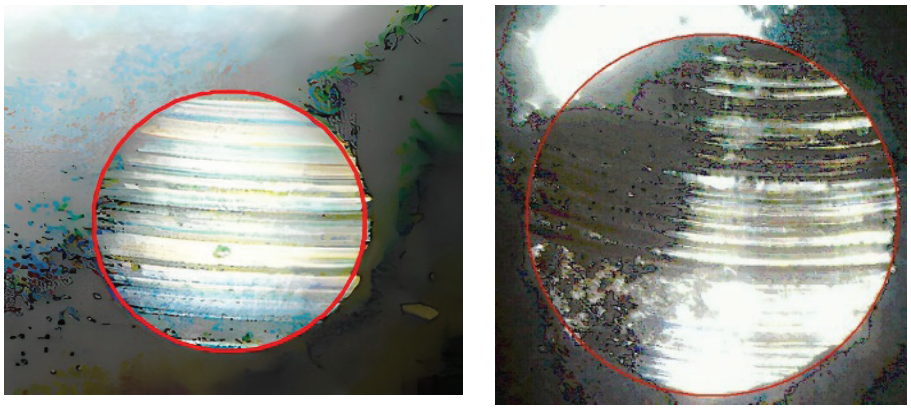


Figure 4. Optical micrographs for WSD with ball surface.

microscope measures the WSD generated at the contact point of balls.

Result Analysis: BHARAT Approach

The tribological tests for bio-lubricants with HNT additives were conducted to understand the effect of HNTs addition and load on the tribological performance of bio-lubricant in terms of CoF and wear preventive characteristics. In this study, we are trying to minimize both CoF and WSD by optimizing HNTs addition and load. So, this is a kind of Multi-Criteria Decision Making (MCDM) problem. In the area of MCDM, various methods are proposed and implemented. Recently Rao [41, 42] proposed the Best Holistic Adaptable Ranking of Attributes Technique (BHARAT) for MCDM problems. BHARAT is simple, transparent and easy to implement method in the area of MCDM. In the BHARAT method, to assess their alternatives in relation to each other for decision-making, the attributes are ranked using a streamlined normalization process. BHARAT is a simple and convenient method to rank the order of importance of the attributes. Conversion of qualitative data to quantitative data is easier without any complex fuzzy logic. BHARAT can handle both qualitative and quantitative problems easily.

The step-wise implementation of the BHARAT approach is as follows:

Step 1: Identification of response variables

Identification of the desired response variables, O_i = *[i = 1,2,3……m]* and the available alternatives, Aj = *[j= 1,2,3,…..n]*. The response variable may be desirable or undesirable. In this study both response variables, delamination factor and thrust force and undesirable.

Step 2: Weight assignment to response variables

Following the objective approach of weight assignment, the response variables are ranked as per the choice of the decision maker in terms of 1, 2, and 3 ……. So on. When two or more response variables are of equal significance then they are ranked with average value. After ranking the R-method [43] is used to evaluate the objective weights.

Table 4. Objective weights

	Output Responses	
	WSD	**CoF**
% Weight	60	40

$$w_i = \frac{1/\sum_{k=1}^{i} 1/r_k}{\sum_{i=1}^{m}[1/\sum_{k=1}^{i} 1/r_k]} \quad (1)$$

Where,

w_i= *weights for response variables, i (i = 1, 2, 3,……m)*

r_k = *rank of response variables, k (k = 1, 2 ,3 ….i)*

m = number of response variables.

The weights for each response variable are shown in Table 4.

Step 3: Data normalization

The available response variables may have different dimensions and scales, it is necessary to convert them into non-dimensional characteristics to compare mutually across the criteria. Normalize the response variables for different alternatives with reference to the 'Best' response variable. The word 'Best' refers to the highest value of the response variable for the desirable response variable and the lowest value of the response variable for the undesirable response variable.

Let there be *n* response variable with *m* values, the decision matrix will be as:

$$A = (x_{ij})_{mn} = \begin{bmatrix} a_{11} & \cdots & a_{1n} \\ \vdots & \ddots & \vdots \\ a_{m1} & \cdots & a_{mn} \end{bmatrix} \quad (2)$$

Equation 3 is used to normalize the desirable response variable and Equation 4 is used to normalize the undesirable response variable.

$$NMa_{ij} = \frac{a_{ij}}{Maxa_{ij}} \quad (3)$$

$$NMa_{ij} = \frac{Mina_{ij}}{a_{ij}} \quad (4)$$

In this work all response variables, wear scar diameter and coefficient of friction are undesirable, so Equation 4 is used for normalization.

Step 4: Calculation of the total score

The total score for the response variable is calculated using Equation 5.

$$Total\ Score(C_i) = \sum w_i * (x_{ji})_{normalized} \quad (5)$$

Step 5: Ranking

The ranking order is to be managed with respect to the values of the total score (C_i) in descending order from 1 to 0. The value which is closest to 1 has attained the first rank and that alternative should be selected as the best among multiple decision-making response variables. The evaluated values for each alternative/experiment and corresponding ranking are presented in Table 5. From the calculation and total score presented in Table 5, it can be deduced that the higher value of total score corresponding to experiment 24 with load: 600N and %Wt. of HNTs: 1.5 is the optimal combination of input parameters to achieve minimal value for WSD and CoF.

Effect of HNTs Addition

Effect of HNTs addition on WSD: Figure 5 below presents the effect of HNTs addition on load bearing capacity of castor seed oil.

Table 5. Experimental results, total score and ranking

Test No.	Input Parameters		Measured Outputs		Total Score	Ranking
	Load (N)	%Wt. HNTs	Wear Scar Dia (Micron)	Coefficient of Friction		
1	200	0	533	0.068	0.793	2
2	200	0.5	740	0.066	0.632	20
3	200	1.0	851	0.057	0.606	22
4	200	1.5	896	0.062	0.571	24
5	200	2.0	924	0.051	0.605	23
6	300	0	553	0.071	0.763	6
7	300	0.5	676	0.061	0.732	11
8	300	1.0	723	0.058	0.669	17
9	300	1.5	740	0.058	0.627	21
10	300	2.0	816	0.033	0.788	4
11	400	0	622	0.057	0.747	9
12	400	0.5	679	0.052	0.724	12
13	400	1.0	699	0.054	0.702	14
14	400	1.5	749	0.037	0.784	5
15	400	2.0	738	0.053	0.683	15
16	500	0	596	0.061	0.753	8
17	500	0.5	668	0.055	0.719	13
18	500	1.0	735	0.043	0.744	10
19	500	1.5	743	0.053	0.678	16
20	500	2.0	862	0.045	0.661	18
21	600	0	724	0.064	0.647	19
22	600	0.5	690	0.046	0.752	7
23	600	1.0	762	0.036	0.791	3
24	600	1.5	807	0.033	0.797	1
25	600	2.0	875	0.070	0.553	24

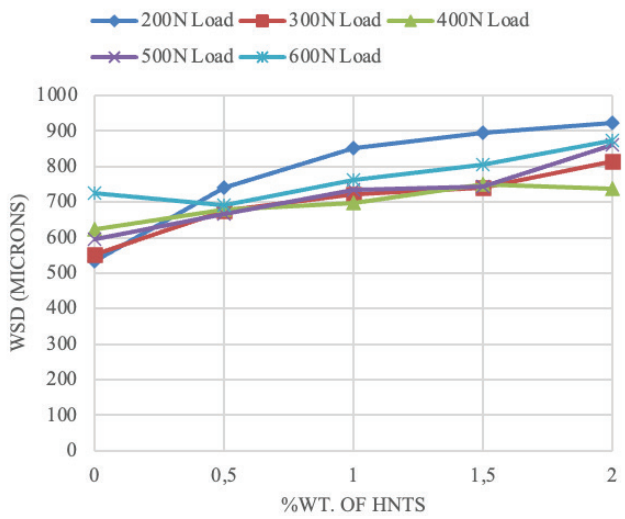


Figure 5. Effect of HNTs addition on WSD.

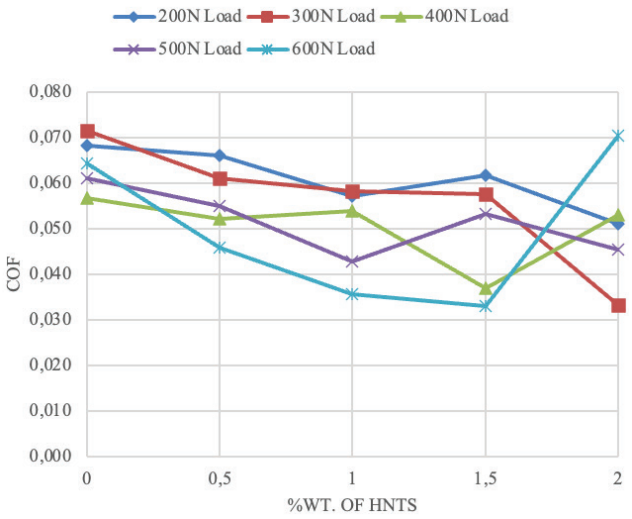


Figure 6. Effect of HNTs on CoF.

It is clear from the figure that with the addition of HNTs, the WSD goes on increasing suggesting that the load-bearing capacity of castor seed oil goes on decreasing and HNTs fail to improve the load-bearing capacity of castor seed oil. Effect of HNTs addition is more significant for higher loading condition in comparison to low loading conditions.

Effect of HNTs addition on CoF: Figure 6 presents the effect of HNTs addition on CoF for castor seed oil. It is clear from the figure that with the addition of HNTs the CoF between mating pairs decreases with an increase in the HNTs addition. So, HTNs addition is contributing to the improvement in the frictional properties of the castor seed oil except for the 600N load.

With the addition of HNTs in castor seed oil the load-bearing capacity decreases with increasing WSD and at the same time, frictional property improves with decreasing CoF. So, it is essential to evaluate the optimal level of %Wt. of HNTs addition with castor seed oil to minimize both WSD and CoF in relation to load. This will present the optimal level of % Wt. of HNTs for improving load-carrying capacity and frictional property of castor seed oil.

It is necessary to bring these two responses to a common platform in terms of non-dimensional numbers. WSD

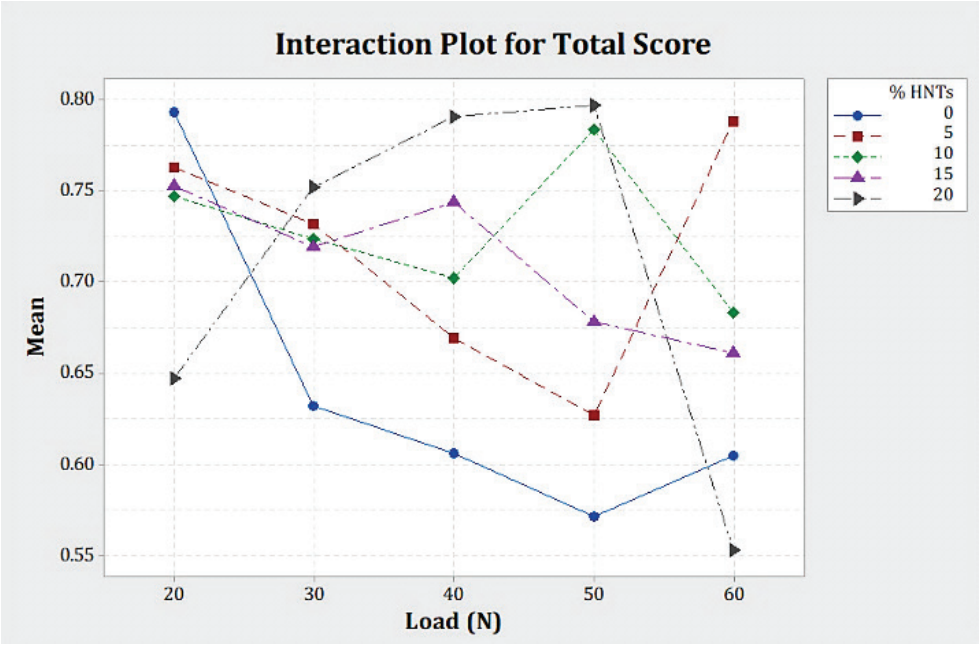
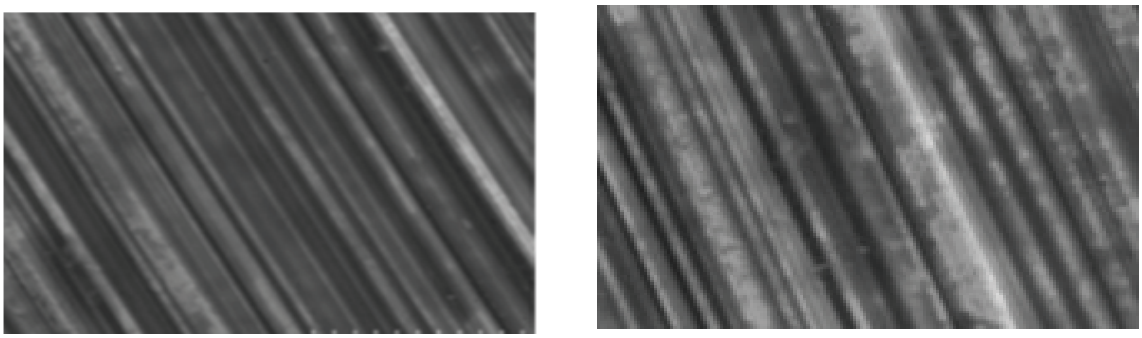


Figure 7. Interaction plot for total score.



(a) **(b)**

Figure 8. SEM micrograph for a worn surface of steel balls: **(a)** castor seed oil without HNTs; **(b)** castor seed oil with 0.5 %Wt. of HNTs.

and CoF values were normalized and converted into total score for comparing and obtaining the optimal level of load and %Wt. of HNTs addition for castor seed oil with the BHARAT approach.

BHARAT approach presented the optimal values for load and %Wt. of HNTs for minimizing WSD and CoF simultaneously, suggesting that there is a strong interaction between load and %Wt. of HNTs addition. Considering 5 levels for both input parameters load at level 5:600N and %Wt. of HNTs at level 4: 1.5% Wt. are optimum to minimise WSD and CoF during tribological testing for castor seed oil. Both responses were converted in terms of a total score by the BHARAT approach and Figure 7, presents the interaction between load and %Wt. of HNTs for the total score.

Worn Surface Analysis

The worn surface of the lower stationary ball was examined with SEM as shown in Figure 8. SEM images clearly show the long trenches generated with ploughing suggesting the presence of adhesive wear during testing for all oil samples.

The level of abrasive wear decreased as observed from SEM images. More abrasive wear was observed during testing for pure castor seed oil without HNTs in comparison to castor seed oil with 0.5 %Wt. of HNTs addition. Surface shows small pitting and indentation indicating metal to metal contact for base castor seed oil. Some traces of HNts were also observed to be trapped in asperities. With increasing HNTs concentration the mending and rolling effect of nanotubes is significant to minimize the abrasive wear. In a similar kind of study for sesame oil with rod-shaped ZnO nanoparticles, resulted into reduction of CoF and WSD by 24% and 13.7%, respectively, with fewer abrasive marks compared to base sesame oil. The presence of ZnO nanoparticles were also observed in asperities with FESEM analysis [44].

CONCLUSION

The tribological tests were conducted for castor seed oil blended with naturally occurring Halloysite nanotubes to propose the completely environmental friendly bio-lubricant. The following major conclusions are drawn based on this study:

1. Castor seed oil is itself bio-lubricant with the addition of naturally occurring Halloysite nanotubes, it presents environmentally friendly biodegradable bio-lubricant with improved tribological properties for replacement to mineral-based oils.
2. Load has a significant effect on the tribological properties of lubricating oil. So, the interactive effect of load and Halloysite nanotubes addition is necessary to consider for evaluating tribological properties of bio-lubricant.
3. An innovative Best Holistic Adaptable Ranking of Attributes Technique approach is implemented to obtain the optimal level of load and Halloysite nanotubes blending with castor seed oil for improving load-carrying capacity and frictional properties of castor seed oil. Analysis of experimental results presented 600N load and 1.5 %Wt. of Halloysite nanotubes with castor seed oil is optimum for improving tribological properties of castor seed oil bio-lubricants.
4. The microscopic image analysis of the worn surface suggest there is the presence of adhesive wear but with increasing Halloysite nanotubes from 0 % wt. to 2 % wt., the effect of abrasive wear diminishes and improves frictional property of castor seed oil.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

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