Jurnal Tribologi 32 (2022) 40-55

MYTRIBOS Malaysian Tribology Society
Jurnal Tribologi
Website : jurnaltribologi.mytribos.org e-issn: 2289-7232

Rheological and tribological properties of rice bran oil grease (RBOG) with h-BN nanoparticles - An experimental study

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KEYWORDS

Dropping point
Rice bran oil grease
h-BN
Oscillatory experiment
Herschel-Bulkley model
Modular compact rheometer

ABSTRACT

Vegetable oil-based semi-solid lubricants are preferred in applications where environmental pollution is a major concern (soil or water pollution). The motive of the current study is to formulate and assess the rheological and tribological behavior of rice bran oil lithium-based bio-grease. The effect of various concentrations of thickener (lithium stearate) and hexagonal boron nitride nanoparticles on rheological properties of RBOG is studied. The thickener is added in a concentration of 4 wt. %, 10 wt. %, and 16 wt. %. The hexagonal boron nitride nanoparticles are added in a concentration of 1 wt. %, 2 wt. %, and 3 wt. %. The rheological properties studied are viscosity, shear stress, storage modulus, loss modulus, and yield stress. Anton Paar Rheometer MCR-102 is used to evaluate the rheology of the grease. The experimental results reveal that the RBOG without nanoparticles depict the maximum COF whereas, RBOG + 3 wt. % h-BN depict the minimum COF. Further, the addition of h-BN nanoparticles in RBOG has reduced the wear scar diameter from 0.93mm to 0.67mm.

1.0 INTRODUCTION

Owing to increasing environmental problems, there is a high worry for the usage of mineral oils as lubricants. This has led to the research and study of vegetable oil-based lubricants. Vegetable oil-based lubricants possess excellent properties like high biodegradability (Darminesh et al., 2017; Thottackkad et al., 2012), low volatility, low toxicity (Ahmad et al., 2020), and high flash point (Mobarak et al., 2014; Shafi et al., 2021). They are good boundary lubricants and do

Received 5 July 2021; received in revised form 25 November 2021; accepted 14 December 2021.
To cite this article: Bhat and Charoo (2022). Rheological properties of rice bran oil grease (RBOG) with h-BN nanoparticles - An experimental study. Jurnal Tribologi 32, pp.40-55.

not contribute to environmental pollution (Alves et al., 2013; Mushtaq and Hanief, 2021). Lubricating grease is a colloidal suspension of carrier fluid and thickener (Bondi and Eirich, 1960; Kuhn, 2009). The thickener holds the carrier fluid and provides the rheological behavior to the grease (Mas and Magnin, 1994). The execution of grease relies upon its constituent ingredients and microstructure attained by it in the course of fabrication process (Delgado et al., 2005; Delgado et al., 2006). Various bio greases developed have been subjected to tribological and thermal investigations to evaluate their effectiveness as a lubricant. Sukirona et al. (2009) formulated lithium soap based palm grease. The antiwear property of the bio grease is assessed using a four-ball wear tester and compared with mineral oil-based grease (HVI 160S). It is observed that the ball specimen depicts less wear with palm grease. The decrease in wear with palm grease is because of the existence of polar oxygen containing groups in its base oil such as hydroxides –OH, oxirane ring –COC-. The interaction between thickener with polar species in the palm oil is more than with non-polar hydrocarbons in the mineral oil. This makes the palm grease more rigid than mineral oil based grease. El-Adly et al. (2014) investigated the comparison of jojoba oil and castor oil-based bio greases. It is observed that the oxidation strength of jojoba oil based grease is higher than that of castor oil based grease due to the linearity in structure and close range composition of jojoba oil. Hyder A. Abdulbari et al. (2011) developed bio grease based on scrap cooking oil. It is observed that the grease possesses excellent lubrication properties and has been used in plenty of applications where biodegradability is necessary. Rizqon et al. (2010) formulated calcium-based palm grease dedicated to bearing and gear lubrication for food processing industries. The performance of the grease is determined by the four-ball wear and gear wear tester. It is observed that the antiwear performance of the bio grease is as good as conventional food grade greases. Barriga et al. (2005) formulated polymer thickened sunflower oil-based grease for lubrication in earth moving equipments. It is observed that the bio grease shows effective lubrication, wear protection, and friction reduction than mineral oil-based grease.

The use of vegetable oil-based greases provides a solution for energy conservation which is a major existing problem. The replacement of petroleum-based greases by vegetable oil-based greases can be immensely productive in the nearing times. The incorporation of nanoparticles has also resulted in resource conservation by reducing the friction and wear in various tribological applications. The rheological, antiwear and friction mitigation qualities of liquid and semi-solid lubricants have been enhanced by adding nano-materials to them. Liu Hongtao et al. (2014) scrutinized the sequel of carbon nanotubes on tribological properties of poly-alpha-olefin oil-based grease. It is noticed that the nano grease shows better lubricity and wear protection in contrast to base grease. The amelioration in the performance of nano grease is attributed to high thermal conductivity and unique hexagonal structure of carbon nanotubes. Alaa Mohamed et al. (2015) synthesized carbon nanotubes of diameter 10 nm and length 5 μm using the electric arc discharge method. The sequel of carbon nanotubes is studied on tribological behavior of lithium grease. Four ball tester is used to evaluate the properties of the nano grease. It is found that wear scar diameter and coefficient of friction have reduced by 63% and 81% respectively, when nano grease is used instead of base grease in the tribological experiments. Escalation in properties of the grease is because of the inception of boundary film of nanotubes. Jibin T-Philip et al. (2020) examined the sequel of nano ceria fragments on tribological features of lithium-based coconut grease. The frictional and wear characteristics of nano coconut grease are observed. It is found that wear scar diameter (WSD) of 0.63 mm is obtained when nano grease is used which is less compared to 0.82 mm obtained when the base grease is used. Charoo et al. (2017) observed that anti-wear properties of lubricant SAE20W50 greatly enhances with the incorporation of h-BN nanoparticles when used as a lubricant in piston and cylinder liner tribo-pair. It is perceived that WSD of the balls reduces by 20% due to the development of a thin protective layer by nanoparticles that result in enhancement in tribological qualities of grease. Abdullah et al. (2016) investigated the effect of h-BN nanoparticles on the 15W40 engine oil. The extreme pressure properties of nano-lubricant are investigated. It is found that the load sustainability of nano lubricant is high in contrast to base oil. The amelioration in the properties of nano-lubricant is due to the development of tribo-films on the facet of tribo-pair by h-BN nanoparticles. The rheological features of conventional greases have been scrutinized by various researchers. Alla et al. (2013) reported that shear stress and the viscosity of lithium grease additivated with carbon nanotubes increase by 81.8% and 67.3% respectively at 2wt. % nanoparticle concentration. Yao, et al. (2011) analyzed the sequel of temperature on thixotropy of grease. It is perceived that rise in temperature minimizes force of interaction among grease fibers and hence decrease in thixotropy is perceived. Tiejun et al. (2016) reported that the rheological characteristics of lithium calcium based grease diminish with escalation of zinc dialkyldithio phosphate (ZDDP). Bahaa M Kamel et al. (2017) reported that viscosity and shear stress of calcium based grease depends on the aggregation of graphene nano sheets and both properties ameliorate by 52%, and 65% respectively at 3 wt. % graphene nanosheet concentration. Yan et al. (2017) reported that yield stress of grease intensifies with a drop in temperature. The internal shear stress of the grease increases with a drop in temperature, consequently increasing the properties of grease. Yeong et al. (2004) reported that the yield stress, storage modulus and loss modulus of lithium stearate grease enrich with the rise in the concentration of lithium stearate. This is ascribed to the fact that a dense matrix formation results with an increase in concentration of lithium stearate which enhances the grease properties.

The current paper investigates the rheological properties of rice bran oil based grease. The authors have found no literature on the impact of thickener and h-BN concentration on rheological features of rice bran oil based grease. The study is novel determining the impact of thickener and h-BN concentration on the rheological properties of rice bran oil based grease. The presence of gamma-oryzanol, a natural anti-oxidant in the rice bran oil (RBO) makes it oxidatively more stable than other vegetable oils, and hence is selected as base oil for grease synthesis. It contains 42% mono-unsaturated fatty acids, 24% saturated fatty acids, and 31% poly-unsaturated fatty acids. Rice bran oil has less saponification tendency than other oils (coconut, sunflower, and palm oil) at high temperatures. Rice bran oil has an Iodine value that lies in middle of coconut oil and sunflower oil and is thermally more stable due to the presence of double bonds. Lithium stearate is used as a thickener. The lithium greases have high thermal resistance, high dropping point compared to other soap-based greases and hence, lithium stearate is used as a thickener. The majority of nano-materials contain sulphur, phosphorous atoms which are a serious threat to the environment. Therefore green additive, hexagonal boron nitride having a lamellar structure is used in the study. The bond between the molecules within a layer of h-BN is covalent, and the layers are supported jointly by weak van der walls forces. Effective lubrication is provided by h-BN as it is sheared easily due to the weak interlayer forces. Furthermore, the h-BN has proved very useful in enhancing the rheological qualities of oil lubricants (Afzal et al., 2020). The motive of current study is to assess the rheological behavior of rice bran oil-based grease additivated with various concentrations of thickener and h-BN at various shear rates and temperatures. The effect of thickener and h-BN concentration on the viscosity, shear stress, storage modulus, and loss modulus of the RBOG is studied on MCR-102 rheometer with parallel plate geometry (PP-25).

2.0 EXPERIMENTAL METHODOLOGY

2.1 Materials and Sample Preparation

The oil employed in this study for grease synthesis is rice bran oil (RBO). The selection of vegetable oil as a lubricant largely depends on its fatty acid composition. RBO has proved to be a potential bio cutting fluid (Jayadas, 2008). The presence of 42% mono-unsaturated fatty acids in the RBO makes it a suitable candidate for various industrial operations. At higher temperatures, the oil has less saponification tendency than coconut, sunflower, and palm oil (Rani et al., 2015). The thickener used is Lithium stearate purchased from Tokyo Chemical Industry (TCI) Japan. The molecular weight of lithium stearate (C18H35LiO2) used is 290.42 g/mol. Hexagonal boron nitride, a green additive with good antiwear property is used as an additive. For the synthesis of nano grease, the nanoparticles in the concentrations (1, 2, and 3 wt. %) are added to the RBO. The oil- nanoparticle solution is then sonicated with the help of a bath sonicator for about 3 hours to get a solution of uniform composition. The solution is then heated up to 90oC with the help of hot plate magnetic stirrer. After wards, lithium stearate in required concentration is added to the oil-nanoparticle solution. The mixture (oil+ nanoparticle +thickener) thus formed is heated to 215oC at which the oil starts developing grease characteristics. The mixture is kept at 215oC for about 20 minutes and cooled slowly resulting in the development of nano grease.

2.2 Apparatus

Rheometer MCR 102 used to study the rheology of nano grease is shown in the Figure 1. The essential components of MCR 102 are the motor, bearings, force sensor, and optical encoder. A torque of 200 mNm is generated by the motor in the rheometer. Two types of bearings are present in the rheometer namely radial and axial bearing. For geometrical alignment, the radial bearing is used, and to sustain the weight of rotating elements axial bearing is used. An electric capacity method is used by a force sensor to get normal force from bearing deflections. MCR 102 measures normal force from 10-3 N to 50N. The optical encoder measures deflection from 5×10-1 to infinity. The measurement of rheological behavior of the bio grease is evaluated at different thickener concentrations (4, 10, and 16wt. %), h-BN concentrations (1, 2, 3wt. %), and temperatures (25oC, 75oC). A parallel plate of dia. 25 mm is used in the study. A gap of size 1 mm is maintained between upper rotating and lower stationary plate. Varying shear rate, temperature to the lubricant is provided by the rotating and stationary plates respectively.

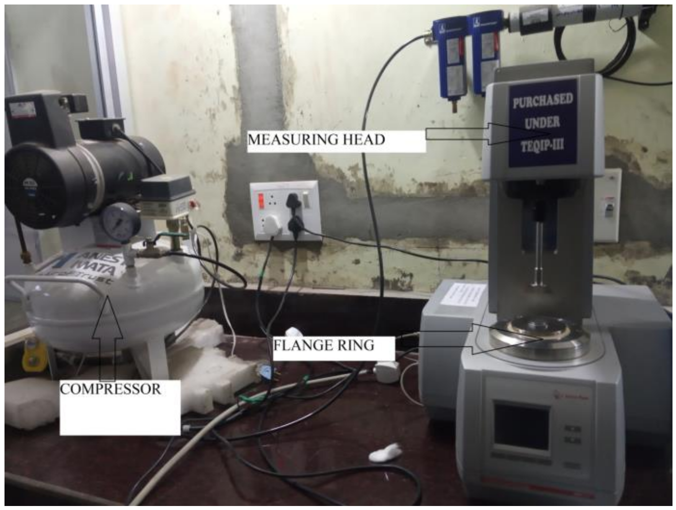

Figure 1: MCR 102 Anton Paar rheometer.

3.0 RESULTS AND DISCUSSION

3.1 Characterization of h-BN Nanoparticles

The field emission scanning electron microscope (FESEM) images show the size and morphology of h-BN nanoparticles. The images are taken at different magnifications and at a working distance of 2.1 mm as shown in Figure 2. The shape of h-BN nanoparticles is nearly spherical with an average size of less than 100 nm.

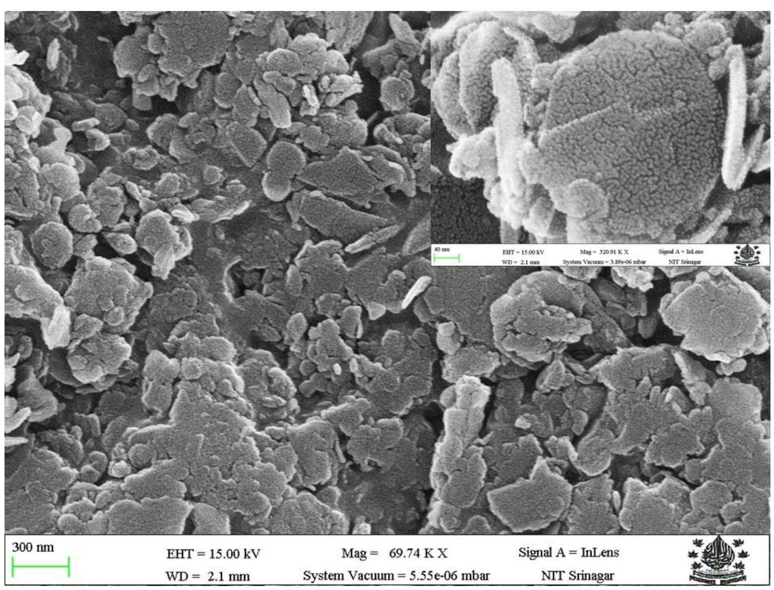

Figure 2: FESEM images of h-BN nanoparticles.

3.2 Viscosity Measurement

The rheological features of grease depend on the concentration of thickener. The thickener holds the carrier fluid and provides the rheological behavior to the grease. The viscosity and the shear stress of the bio-grease containing 16 wt. % thickener is higher than the greases containing 4wt. % and 10wt.% thickener as shown in Figures 3 and 4. The increase in the concentration of thickener increases the number of fiber contacts within the grease structure, which increases the resistance offered by the grease. This is ascribed to an increase in cohesive forces among grease fibers with an increase in the concentration of thickener.

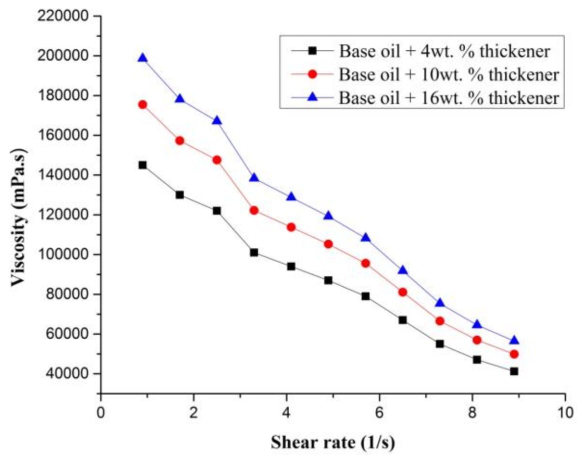

Figure 3: Effect of lithium stearate (thickener) concentrations on viscosity of RBOG at various shear rates at 25°C.

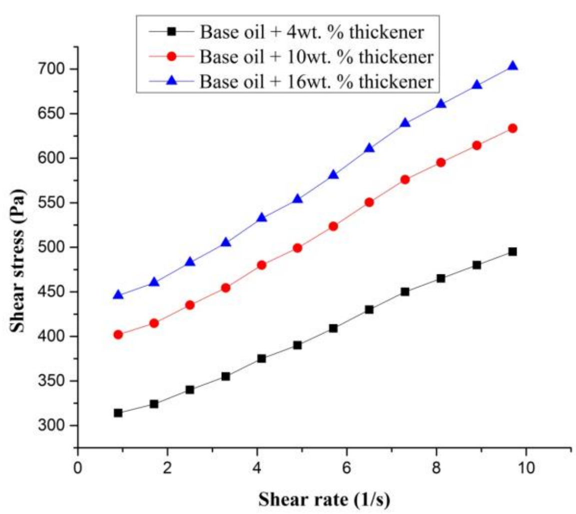

Figure 4: Effect of lithium stearate (thickener) concentration on shear stress of RBOG at various shear rates.

The incorporation of h-BN nanoparticles in bio grease results in an increase in grease viscosity. The viscosity of bio grease declines with shear rate as depicted in the Figure 5. Further, the viscosity of RBOG ameliorates with rise in the concentration of nanoparticles at both temperatures (25oC and 75oC). The nano grease displays greater viscosity at low temperature and vice versa as shown in Figures 5 and 6. The force of interaction among grease fibers diminishes with rise in temperature and hence decrease in viscosity is observed. The percentage increase in viscosity of bio grease at 1, 2, 3 wt. % h-BN concentration, at a temperature of 25oC, and at a shear rate of 20s-1 equals to 14, 36, 48% respectively. The rise in h-BN concentration increases the flow resistance of the grease. The escalation in flow resistance is ascribed to the agglomeration of nanoparticles, which intensifies the clump size of the particles.

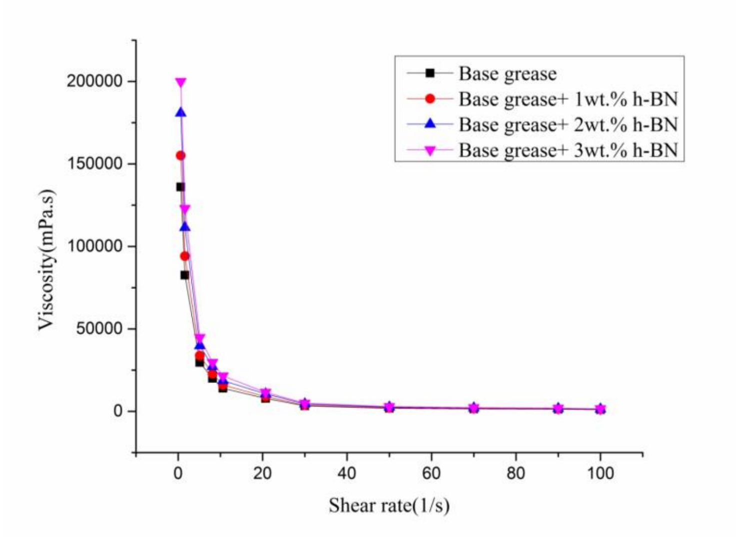

Figure 5: Effect of h-BN concentration on viscosity of RBOG at various shear rates at 25°C.

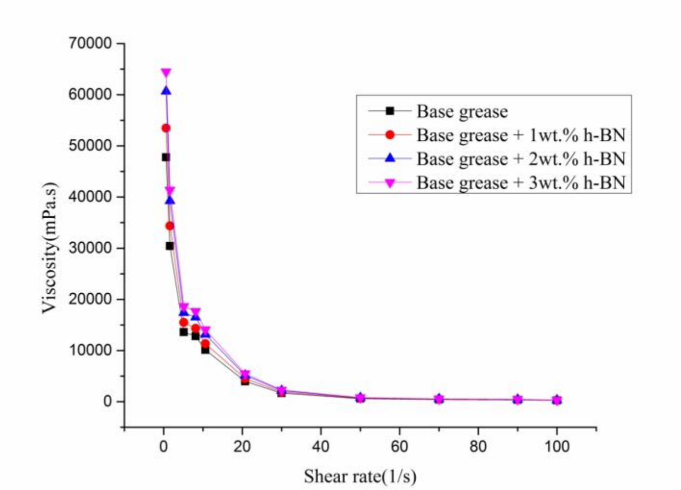

Figure 6: Effect of h-BN concentration on viscosity of RBOG at various shear rates at 75°C.

3.3 Oscillatory Experiment

The oscillatory experiment is used to quantify G', G'', G^* and yield stress of the grease. The experiment is carried out at a frequency of 10 radians per second. In the experiment, the amplitude changes from 0.01-100%. Further G', G'', and G* are acquired from stress (τ_o) and strain (γ_o) amplitudes to phase angle shift δ. Storage modulus (G') is the portion of energy stored in grease microstructure and is the measure of grease stiffness. Loss modulus (G'') is the part of energy lost by grease sample during shearing (Ferry, 1961). The ratio of G'' to $G'$is a loss factor. Complex modulus G^* indicates grease resistance and is equal to the vector sum of G' and G'':

$$G^* = \frac{\tau_0}{\gamma_0} \tag{1}$$

$$G' = G^* \times \cos\delta \tag{2}$$

$$G'' = G^* \times \sin\delta \tag{3}$$

$$(G)^2 + (G'')^2 = (G^*)^2[\sin^2\delta + \cos^2\delta] \tag{4}$$

$$\tan\delta = \frac{G'}{G''} \tag{5}$$

The sequel of shear stress on the G' and G" of RBOG is depicted in Figure 7. It is perceived that the value of storage modulus is larger than loss modulus and both are almost constant within a certain region called the viscoelastic region and the value of loss factor within this region is less than unity. A decrease in G' and G" is observed after the viscoelastic region up to a point called a crossover point at which the value of G' and G" is equal (Yeong et al., 2004). The equivalent stress at crossover point is yield stress and value of the loss factor is unity. The stress required to make lubricating grease flow is called yield stress (Barnes, 1999). The loss modulus and storage modulus decreases even after crossover point. The value of storage modulus is smaller than the loss modulus after cross over point and the grease starts to flow. The value of the loss factor within this region is greater than unity.

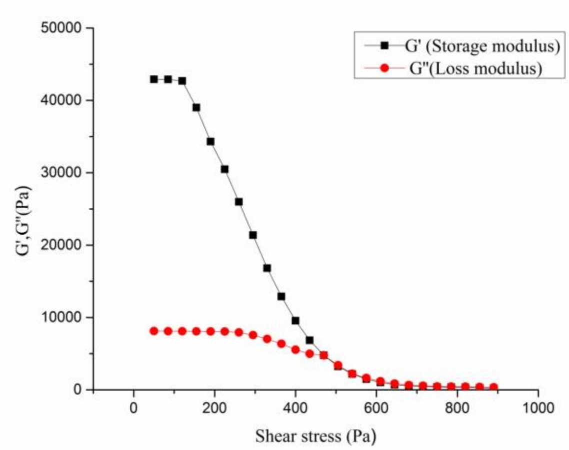

Figure 7: Effect of shear stress on storage modulus and loss modulus of bio base grease.

The effect of thickener concentration on G', G" and yield stress of RBOG is shown in the Figure 8. It is noticed that G', G" and yield stress of the grease improve with the rise in concentration of thickener. Further, it is observed that the addition of nanoparticles in the bio grease has resulted in enhancement of G', G" and yield stress as shown in Figure 9. The enhancement in mentioned characteristics of grease is ascribed to the strong microstructure achieved by the bio grease due to the agglomeration of nanoparticles. The effect of nanoparticle concentration on the shear stress of RBOG at 75oC is shown in Figure 10. It is observed that shear stress of RBOG decreases with rise in temperature. This is ascribed to decrease in cohesive forces among grease fibers, consequently reducing the internal shear stress of the grease. Tables 1 and 2 display the values of G', G", loss factor, and yield stress as a function of thickener and h-BN concentration.

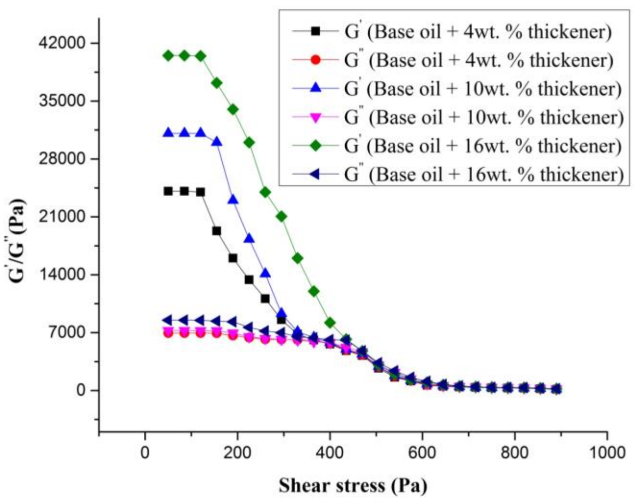

Figure 8: G', G" versus shear stress at different thickener concentrations (25°C).

Table 1: Values of storage modulus, loss modulus, loss factor and yield stress of bio- grease at different thickener concentrations.

Grease type	Storage modulus (Pa)	Loss modulus (Pa)	Loss factor	Yield stress (Pa)
Base oil + 4 wt. % thickener	19,300	6,940	0.360	345.00
Base oil + 10 wt. % thickener	30,020	7,230	0.240	400.00
Base oil + 16 wt. % thickener	37,250	8,010	0.215	443.98

Table 2: Values of storage modulus, loss modulus, loss factor and yield stress of bio- grease at different h-BN concentrations.

Grease type	Storage modulus (Pa)	Loss modulus (Pa)	Loss factor	Yield stress (Pa)
S_1	42,913	8,322.1	0.194	470
S_2	54,987	9,447.0	0.171	505
S3	78,764	13,400.0	0.170	575
S4	96,739	14,394.0	0.149	610

S_1 =Base grease (20 wt. % thickener)
S_2= S_1 +1wt.% h-BN
S_3= S_1+ 2wt. % h-BN
S_4= S_1+3wt. % h-BN

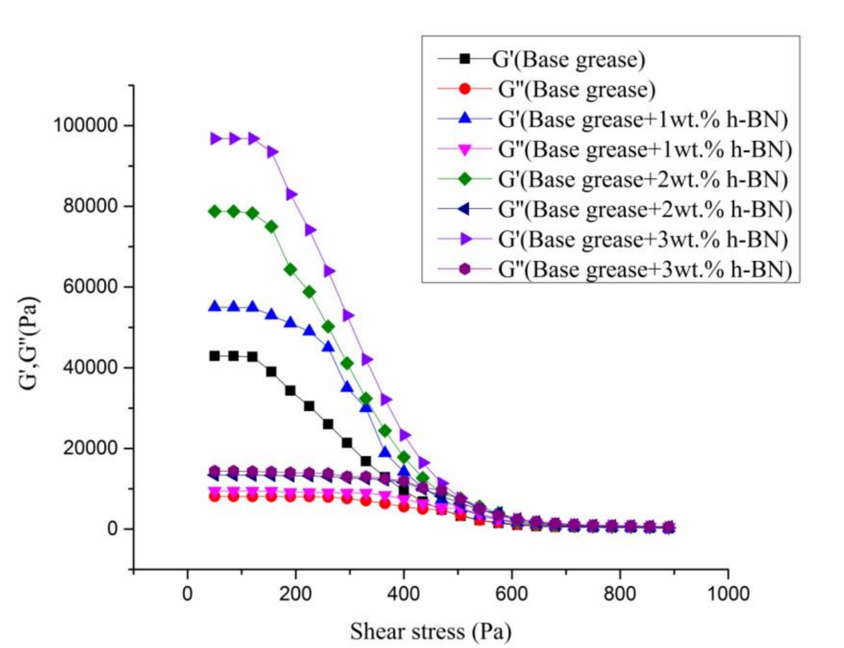

Figure 9: G',G" versus shear stress at different h-BN concentrations at 20wt. % thickener concentration (25 °C).

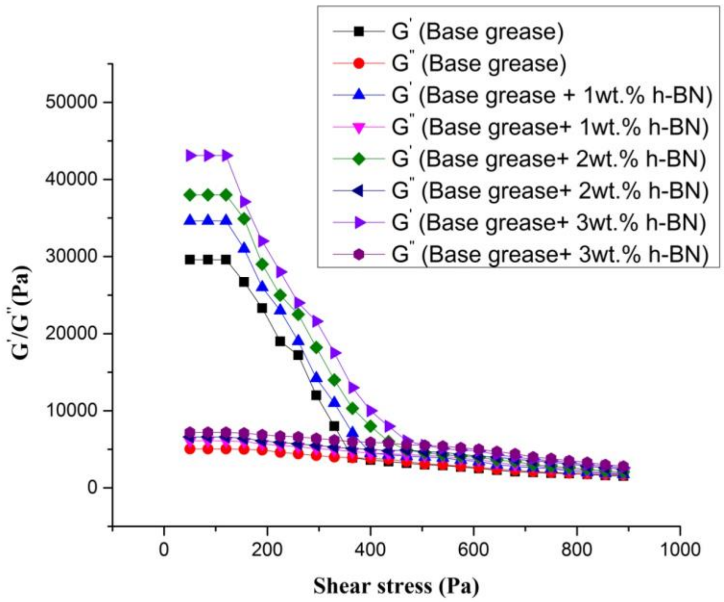

Figure 10: G',G" versus shear stress at different h-BN concentrations at 20wt. % thickener concentration (75 °C).

3.4 Measurement of Flow Behavior

Grease is a non- Newtonian fluid with yield stress. The non-Newtonian behavior of the bio grease is expressed using a power-law proposed by Herschel and Bulkley, known as Herschel-Bulkley model given below

$$\tau = \tau_0 + K\gamma^n \quad \text{(H-B model)} \tag{6}$$

Where τ = shear stress (Pa), τ_0 = yield stress (Pa), K = consistency factor, γ = shear rate (s-1) and n = shear thinning index.

For shear-thinning fluid n <1, for shear thickening fluid n >1 and for Newtonian fluid n =1 and τ_0=0

For n =1, equation (6) reduces to

$$\tau = \tau_0 + K\gamma \quad \text{(Bingham model)} \tag{7}$$

Figure 11 shows the sequel of shear rate on shear stress at different h-BN concentrations. It is noticed that the shear stress increases with shear rate, not in a linear manner, depicting non-Newtonian behavior. The parameters of the Herschel- Bulkley equation at different h-BN concentrations are shown in the Table 3. The yield stress for the nano grease is determined by the oscillatory experiment. The value of K and n are measured using the least square method.

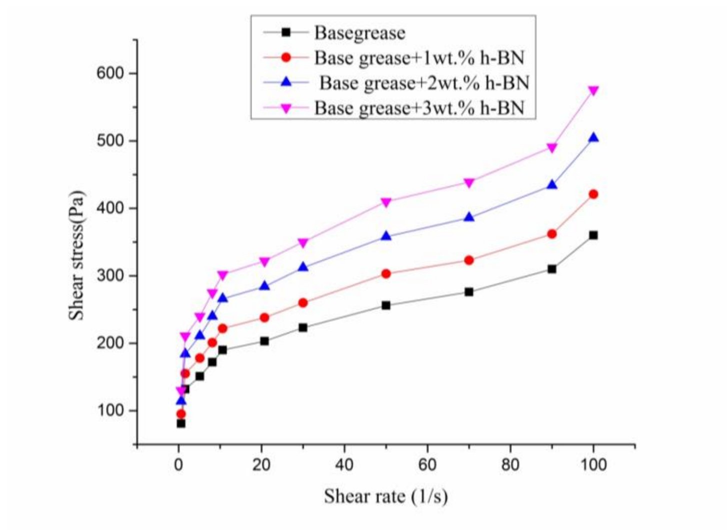

Figure 11: Effect of h-BN concentration on the shear stress of RBOG at different shear rates.

Table 3: Values of different parameters of Herschel- Bulkley model.

Grease type	τ_0 (Pa)	K (Pa.s^n)	Loss factor
S_1	470	119.03	0.270
S_2	505	139.00	0.243
S3	575	157.32	0.263
S4	610	193.00	0.280

S_1 =Base grease, S_2= S_1 + 1 wt. % h-BN, S_3= S_1+ 2wt. % h-BN, S_4= S_1+ 3wt. % h-BN

3.5 Dropping Point Measurement

The dropping point apparatus is used for the measurement of the dropping point (DP) of RBOG. The grease changes to a liquid state at a certain temperature, known as the dropping point. A nipple fitted with a thermometer is filled with a grease sample of about 0.5 g. The sample is heated till a drop is produced on basal opening of nipple. When the drop falls into the test tube, the temperature is recorded and is the DP of grease. The effect of nanoparticle concentration on the dropping point of RBOG is shown in Figure 12. It is found that the dropping point of RBOG increases with a rise in nanoparticle concentration. This is ascribed to the accumulation of nanoparticles that increases the strength of grease microstructure. The percentage increase in the DP of bio- grease at 3 wt. % h-BN concentration is 17.93%.

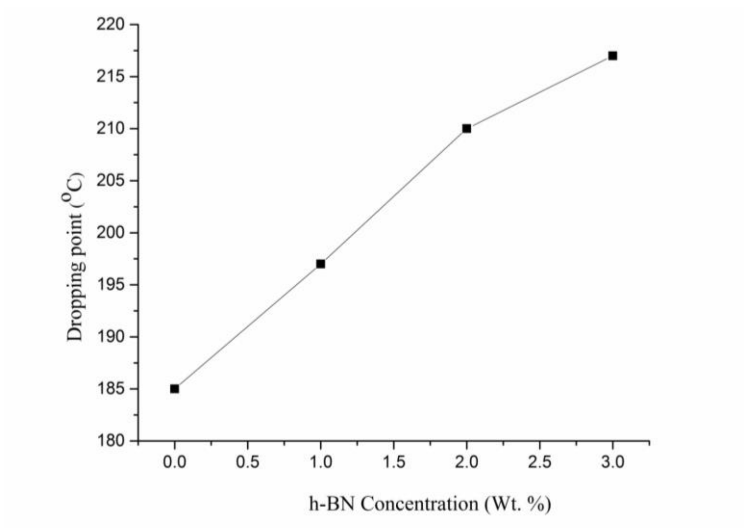

Figure 12: Variation of dropping point of grease with h-BN concentration

4.0 TRIBOLOGICAL TEST

The tribological performance for the antiwear property of RBOG is examined on four-ball tester (Figure 13) as per ASTM D2266. The test conditions used are (load=40 kgf=392 N, Duration=60 m, Temperature =75 oC, and Speed = 1200 rpm). One of the balls is fixed in the collection and placed in the spindle. The other three balls are fixed in the ball pot and filled with RBOG. A load of 40 Kgf is exerted slowly using a lever mechanism. Wear scar diameter is computed using an optical microscope and the coefficient of friction (COF) is measured from the average tangential load as showed by the machine. The variation in the coefficient of friction with different concentrations of h-BN is shown in Figure 14. The RBOG devoid of nanoparticles has shown the maximum COF and RBOG +3 wt. % h-BN has shown the minimum COF. The variation of wear scar diameter (WSD) with different concentrations of h-BN is shown in Figure 15. The addition of h-BN nanoparticles in RBOG has reduced the wear scar diameter from 0.93 mm to 0.67 mm. The decrease in COF and WSD with the addition of h-BN nanoparticles is due to reduction in contact between the asperities as rolling friction is facilitated by nanoparticles.

Figure 13: Four ball tester (TR-30H-PNU-IAS-KRL-RF).

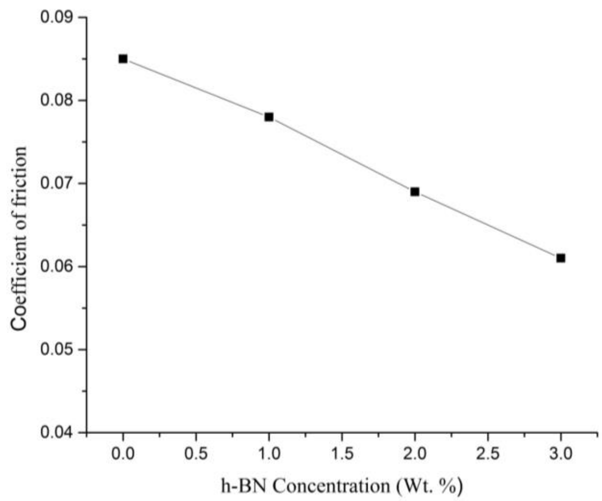

Figure 14: Coefficient of friction as a function of h-BN concentration.

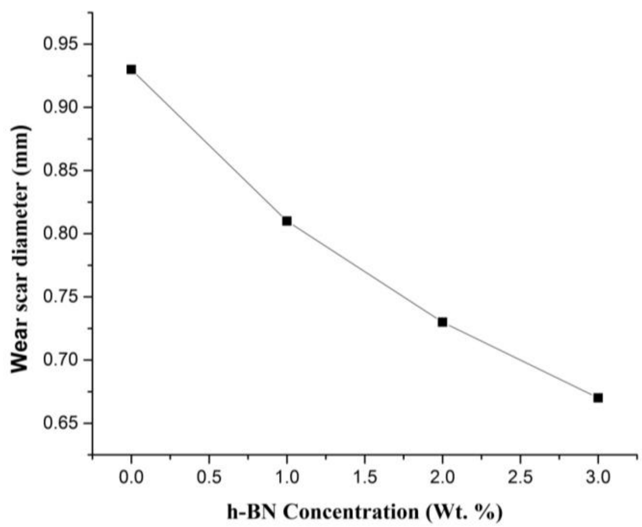

Figure 15: Wear scar diameter as a function of h-BN concentration.

CONCLUSION

The paper discusses the formulation, effect of thickener (lithium stearate) and h-BN concentration on the rheological characteristics of rice bran oil grease (RBOG). The optimum concentration of thickener and h-BN is 16 wt. %, and 3wt. % respectively. It is observed that the grease containing 16wt. % thickener has a 28.7% increase in yield stress, 37% increase in viscosity and 42% increase in shear stress over the grease containing 4wt. % thickener. The increase in lithium stearate concentration increases the number of fiber contacts within the grease structure, which in turn increases the resistance offered by the grease. The grease containing 3wt. % h-BN has 48% increase in viscosity, and 38.6% increase in yield stress over the base grease. Further, it is also observed that the dropping point (DP) of the RBOG ameliorates with the rise in nanoparticle concentration. The percentage increase in the DP of the grease at 3 wt. % h-BN concentration is 17.93%. This is ascribed to the resting of h-BN nanoparticles between the grease fibers, consequently increasing the grease resistance. Finally, it is concluded that RBOG with 3wt. % h-BN can be a propitious lubricant alternative for industries with regard to environmental concerns. Furthermore, the friction and wear properties of RBOG has improved with the addition of h-BN nanoparticles.

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