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EXPERIMENTAL STUDY ONRHEOLOGICAL PROPERTIES OF CHILLI SEED OIL WITH GRAPHENE NANOPARTICLES

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Abstract—**This research paper focuses on investigating the rheological properties of chilli seed oil blended with graphene nanoparticles (GNPs). The rheological behaviour of chilli seed oil was analyzed using the Physica MCR 101 rheometer, manufactured by Anton Paar. A cone and plate geometry setup were utilized to measure the shear stress and shear rate of the oil. Additionally, the density of chilli seed oil was determined at a temperature of 25°C. The study reveals that chilli seed oil exhibits Newtonian behaviour under different applied loads, and this behaviour was quantified using the power-law model. It was observed that the introduction of graphene nanoparticles into the chilli seed oil significantly influenced its viscosity. The results indicate that even at a concentration of 0.25 wt%, the viscosity of the oil increased notably. The tested concentrations of 0.25 wt% GNP addition exhibited the maximum enhancement in viscosity. The research further analyzed the variation in viscosity concerning shear rate, torque, and shear stress to establish a comprehensive understanding of the oil's modified flow behaviour. The obtained viscosity values were then compared and correlated with existing theoretical models to evaluate their accuracy and reliability. This study provides valuable insights into the potential applications of graphene-enhanced chilli seed oil in various engineering and industrial fields where improved lubrication properties are desired. The findings contribute to the growing research on nanoparticle-infused lubricants and their impact on fluid dynamics.**

Keywords—**Chilli seed oil, flow behaviour, lubrication, graphene-nanoparticles ,rheological properties, viscosity**

I. INTRODUCTION

Lubrication is essential in mechanical systems to manage friction and wear by introducing lubricants between moving surfaces. These lubricants, available in liquid, solid, or plastic forms, minimize friction and ensure smooth operation, vital for optimal machine performance. Effective lubrication reduces wear, prevents strain, and avoids issues like bearing seizures. Insufficient lubrication can lead to heat buildup, structural damage, and system failure. Bio-based lubricants, derived from renewable sources like vegetable oils and animal fats, offer a biodegradable and less toxic alternative to mineral oils. They provide excellent lubricity and performance while reducing environmental impact, making them a promising solution to the challenges posed by traditional petroleum-based lubricants. Bio lubricants are becoming increasingly significant in the field of sustainability due to their biodegradable and eco-friendly properties. Several tribological studies have been conducted on vegetable oils to evaluate their effectiveness as lubricants. Also the enhancement of oil's tribological properties through nanoparticles relies on factors such as size, morphology, chemical composition, concentration, nanostructure, and surface functionalization of the nanoparticles [1]. Nevertheless, nanoparticles with an onion-shaped morphology are correlated with reduced frictional coefficients and the greatest reduction in wear [2]. As a two-dimensional material, graphene presents distinctive friction and wear characteristics that diverge from those of conventional materials. In addition to its established thermal, electrical, optical, and mechanical properties, graphene can function as a solid or colloidal liquid lubricant. Also, it has high thermal stability and ability to absorb gas when added to an inert substance [3]. Recently, numerous studies have investigated the wear characteristics of graphene when used as a lubricant additive [4-9]. Graphene also demonstrates enhanced flexibility, high specific strength, improved fatigue resistance.

Chillies from Byadgi, Guntur Sanam, Mathania, Kashmiri, Resham Patti, and Teja were analyzed to guide the design of equipment for processing dry chillies, including sorting, grading, drying, and destalking [10]. Key properties assessed included geometrical, gravimetric, optical, aerodynamic, mechanical, and frictional characteristics [11,12]. Whole chillies measured 83.83 to 146.03 mm in length and 9.66 to 41.41 mm in width. Bulk densities ranged from 100 to 130 kg/m^3, and true densities from 260 to 560 kg/m^3. Colour parameters and hardness values varied, providing insights for equipment design. Terminal velocities were between 3.66 to 9.03 m/s (experimental) and 5.33 to 10.69 m/s (theoretical), with coefficients of friction for various materials ranging from 0.34 to 0.65. The engineering properties of the chilli pods and seeds were studied to finalize the designing parameters of the chilli seed extractor [13]. Y Hwang et al. studied the shape and size of carbon nano additives for their effect on the tribological properties of mineral oil. All particles are added at 0.1 vol.% to the oil and tribological tests are performed using a disc-on-disc tester. The spherical morphology of nano-graphite particles results in the greatest improvement in oil C.O.F.Graphite nanoparticles, carbon black, graphite nanofibers, and carbon nanotubes reduce the COF of raw oil by 68.5%, 65.7%, 42.8%, and 20%, respectively.[14].The friction and wear of the worn surfaces was studied in automobile engines[15].The objective of the work was to improve the tribological behaviour using graphene (Gr) nanolubricant.Anti-friction and anti-wear properties of Gr nanolubricant have been evaluated using tribometer based on ASTMG181.The tribological results showed that the lubrication via Gr-nanolubricant improves the anti-friction and anti-wear properties by 29–35% and 22–29%, respectively. Furthermore, the exhaust emissions(CO, CO_2, HC and NOx) were decreased by 2.79–5.42%.Graphene was studied for its hybrid by Srivastava, Isha, Ankit Kotia, et al.[16]graphene–MWCNT,andgraphene–SiO_2 hybrid nanoparticles in engine oil(n-decane).The nanoindentation deduced that for graphene–MWCNT/engine oil nanoparticles, at a volume fraction of 1.8%, the nanoparticles outperformed all other nanolubricants. At a volume fraction of 0.3%, the adhesion between the solid and liquid interface was increased by 2.5% graphene nanolubricant, 4% graphene–MWCNT nanolubricant, and 8% graphene–SiO_2 nanolubricant. At 1.8% volume fraction, 240% increase in graphene–MWCNT nanolubricant, 72% increase in graphene nanolubricant, and 20% increase in graphene–SiO_2 nanolubricant. Li, Jia, et al. investigate the effect of concentration, size, temperature, and shear rate on the viscosity, mean square displacement (MSD), and density of graphene on lubricating oil[17]. The results show that the viscosity of GLO increases with the increase of graphene concentration and decreases with the increase of temperature (293 K–330 K).Compared to the base oil, the simulation and experiment viscosity values of the GOL with 3G increased by 22.9 % and 47.6 %, respectively.

The influence of graphite nano-flakes (GNF) and graphene nano-platelets (GNP) additives were examined in engine oil grade SAE 15W40. Singh, Gurtej, M. F. Wani, et al. analysed the brake power (BP), engine torque (ET) and mechanical efficiency (ME) of the engine using 0.1 wt.% GNP + SAE 15W40 and 0.1 wt.% GNF + SAE 15W40 and realised an enhance by 3.7–4.8%, 3.1–6.3% and 2%; and by 1.8–3.8%, 2.3–5.2%, and 1.4% compared to virgin SAE 15W40, respectively. However, the brake specific fuel consumption (BSFC) and emissions were reduced by 1.7–3.8% and 2.0–4.0% for 0.1 wt. % GNP + SAE 15W40 and by 1.2–3.4% and 1–3% for 0.1 wt. % GNF + SAE 15W40, respectively [18].

II. PROPOSED METHODOLOGY

The study utilized formulated chili seed oil.

Chili seeds procured from local market (Valley Health Care, Pulwama) and were first air blown to remove any extraneous materials, then sun-dried and stored in zip bags in the refrigerator at 4 ºC until used in experiment

Petroleum ether is a flammable, nonpolar solvent made up of 50–80% paraffins, 20–40% monocycloparaffins and 2–10% aromatics. With a boiling point of 38–150°C and a flash point of −18–13°C, it is used in industries such as rubber, pharmaceuticals, fuels, paints, inks, and pesticides.

Table -1Chilli oil specification

S.No	**Type**	**Specification**
1	Sample	Chilli oil
2	Temperature	25
2	Viscosity	0.0236
3	Flow behaviour index (n)	0.0065
4	Consistency coefficient (k)	K=0.0236x +0.0065
5	r^2 Value	0.999

A. Chilli oil properties-

Rheological behaviour: The flow behaviour of the CSO was observed by shear stress test with the help of rheometer (Physica MCR 101, Anton Paar). Cone plate geometry was used to obtain the shear stress (Pa) while varying shear rate from 0.1 to 100 rad/s. The flow behaviour was obtained by modelling the change in shear stress with the shear rate (Hosseini-Parvar et al., 2010)

Density: Density was measured using micro-tip. The bottom of micro-tip was sealed with tape then weighed along with the tape using weighing balance (W_1). With the help of micropipette using another micro-tip, 1ml of CSO was pipette out and poured into previously weighed tip. Then micro-tip was again weighed along with oil (W_2). The mass of CSO was calculated by using the formula.

$$\mathrm{massofCSO}(W_0) = W_2 - W_1 \qquad (1)$$

W_1 is the weight of empty micro-tip, W_2 is the weight of micro-tip with CSO.
Density of CSO was calculated using this equation

$$\mathrm{density} = \frac{\mathrm{mass}}{\mathrm{voume}} (2)$$

Contact angle: Sample CSO extracted by conventional method was taken and a glass slab as a solid material. With the help of micropipette, 20 µl of CSO was placed on a glass slab. After placing the CSO on glass slab, image of sample was taken directly with camera and their contact angle was measured by software.

Rheometer: This examines formulated chili seed oil viscosity and thermal stability.

Graphene nanoparticles are added to oil and sonicated for one hour, with interactions analyzed using Fourier Transform Infrared Spectroscopy (FTIR). Rheological tests are conducted immediately to minimize agglomeration. Rheological properties are measured using an Anton Paar Modular Compact Rheometer (MCR-102), featuring an electronically communicated motor and high-resolution optical encoder for precise control. The cone and plate geometry has a 40 mm diameter and temperature is regulated via a Peltier effect flange ring. Viscosity index is calculated according to ASTM D 2270 standards, assessing the impact of graphene concentrations and comparing experimental results with theoretical models.

B. **Extraction of chilli seed oil**-

The procedure carried out for conventional extraction is as follows:

Prior to extraction process, Chilli seeds were manually cleaned to eliminate the contamination. Since the contaminants present in the sample can affect the properties as well as the outcomes of our results. Thus, the sample is cleaned by manual elimination ensuring great precision. The seeds were then dried well in sunlight to eliminate the presence of moisture and figure 2.1 shows the dried chilli seeds. Moisture presence can be of nuisance when the seeds are to be grinded. The moisture may lead to forming of lumps and even contaminating the oil with molecules of water.

Fig.1 Chilli seeds

The dried seeds were grinded manually first in order to get smaller size. Fig 2 shows the powdered chilli seed after getting dried in sunlight for 48 hours.

Fig 2. Dried Chilli seed powder

The chilli seeds were then grounded into powder form in FOSS grinder. The job of this grinder is to grind the seeds to our desired size thus meeting our requirements. The grinder used for further grinding is shown in fig.3.

Fig. 3 Foss grinder

The seeds were then sieved to get the particle size desired ranging from 300- 425µm using sieves of 300-425µm. This implies that the maximum size of particle we have on hand is 425 µm.In order to study results in-depth and to find out which ratio gives better yield,two different samples bearing proportions of 1:5and 1:7 were taken into analysis and investigation.

Fig.4 Petroleum ether mixture

For making 1:5 Sample, 30g of powdered Chilli seeds were placed in a 250ml beaker, followed by addition of 150ml of solvent petroleum ether (solid/solvent ratio 1:5). Fig.4 and fig.5 shows petroleum ether solution and chilli weight marked chilli seed powder respectively.

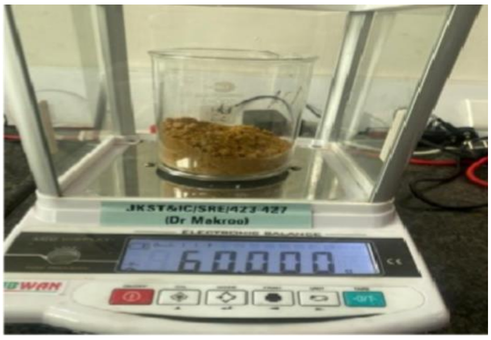

Fig. 5 Weighing machine

Similarly for making 1:7 Sample, 25g of powdered chilli seeds were placed in a 250 ml beaker, followed by addition of 175 ml of solvent petroleum ether (solid solvent ratio 1:7). Since 1:5 ratio was favourable in terms of oil yield so the solution was considered final for extraction of oil and tastings. Fig.6 shows the final chilli seed powder in petroleum ether as solution.

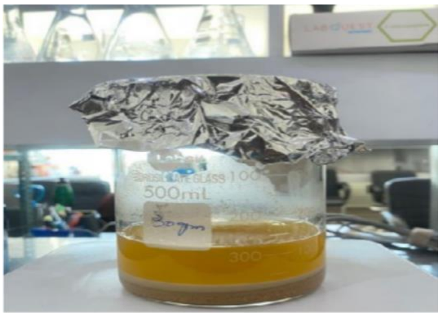

Fig. 6 Ether chilli oil solution

The sample was kept on a magnetic stirrer as shown in fig.7 at 45°C for 1 hour at 550 rpm for extraction of oil from seed powder for 1:5 and at 40°C for 1 hour at 600 rpm for 1:7. Magnetic beads were added to the sample in order to crush the lumps and achieve a uniform texture.

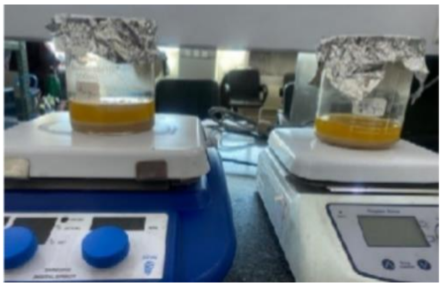

Fig. 7 Magnetic stirrer

Once the sample is mixed it is left an hour so that the contents settle down. The sample that is above in layer is Oil and ether mixed which were then to be separated by means of filtration.

Fig. 8 Chilli oil solution

After stirring of the filtered sample with Whatman filter paper number 42 marked with pore size of 2.5μm and then let to rest for overnight. Then placed in the oven at 50°C overnight to evaporate the solvent and hence the chilli seed oil was obtained. Fig.9 shows the filtration process of chilli oil.

Fig. 9 Oil filtration process

The oil yield was estimated using the formula below:

$$\text{Yield}\left(\frac{\text{ml}}{\text{g}}\right) = \frac{\text{oilrecovered (ml)}}{\text{weightofagribyproducts}} \text{x10}$$

And hence in this way the chilli seed oil was obtained.

Petroleum ether and chemicals were sourced from Qualikems Fine Chem Pvt. Ltd. in Vadodara, India, and provided by the Food Technology department at the Islamic University of Science and Technology, Pulwama, India.

Graphene stands out among all nanoparticles due to its exceptional mechanical, physical, and electrical properties. It has found applications in various fields and is often referred to as a "super material" or "all-in-one material" in materials science.

Table -2 Powder specification:

S.No	Property	Specifications
1	State	GNP Powder
2	Purity	99.5%
3	Thickness	2-4 nm
4	Lateral size	5 µm
5	Density	2.3g/cm³
6	Melting Point	3800 K
7	Tensile strength	1TPa

The rheological test of chilli seed oil is conducted on physica MCR 101, Anton paar. Cone and plate geometry was used to obtain shear stress and shear rate.

The resultant parameters of shear stress,shearrate, torque and viscosity of chilli seed oil were compared at different loads.

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III. EXPERIMENT AND RESULT

The formulated chilli seed oil was extracted at different mixture ratio of dried chilli seed powder and petroleum ether in order to attain most efficient chilli seed oil yield. Two proportions of ratio 1:5 and 1:7 were checked upon for the results.

Yield (1:5) = $\frac{\text{Oil recovered (mL)}}{\text{weight of agri by products}} \times 10 = \frac{5.6}{60} \times 10 = 0.93$

Oil yield at 1:7 ratio is :

Yield (1:7) = $\frac{\text{Oil recovered (mL)}}{\text{weight of agri by products}} \times 10 = \frac{5.5}{62} \times 10 = 0.88$

Since 1:5 ratio showed better yield in the conventional extraction using petroleum ether as solvent, that is for 1:5 ratio the yield came out to be 0.93 i.e. 93% .So 1:5 ratio extraction yield of chilli seed oil is used for the experiments.

Results obtained for TPC extraction from chilli seed oil is :

Table -3 TPC Extraction Result

Sample	Time (min)	Oil Yield (%)	Total phenolic content (mg GAE/ g oil)
Conventional extraction	60	16.57±0.028	18.34 ± 0.01

The TPC is measured in gallic acid equivalents and they determine the redox properties of chilli seed oil. The TPC can widely vary and don't have a single ideal value but the round about ideal value is considered to be around 25.[19]

Table-4 Colour results for chilli seed oil

Sample	L*	Chroma	Hue angle(°)
Chilli seed oil	**0.023±0.005[a]**	**0.178±0.032[a]**	**14.3±0.54[a]**

Here it was concluded that hue angle comes out to be 14.3 ± 0.54 confirming the colour to be in between the pure red code and pure yellow code.

The density of the lubricating oil varies in range from 0.91 to 0.93 g/ml or g/cm^3.There is no direct relation between viscosity and density and both these are the characteristics of a lubricant .The denser fluids have been showing high viscosity characteristic because of more internal resistance so the lubricating oil having a upper hand on density is considered to be ideal.[20]Density of chilli seed oil obtained by conventional extraction method comes out to be 0.932±0.002 g/ml.

The hydrophobic surfaces have contact angle > 90 while hydrophilic have contact angle < 90[20]. The formulated chilli seed oil extracted by conventional method shows an angle of 27.032 ± 0.698.

The addition of the graphene nanoparticleschanges the contact angle of the chilli seed oil from 27 to 153 and this change was seen at 0.025g weight percentage addition of graphene nano particle. The flow behaviour of chilli seed oil extracted by conventional method is given by the parametric measure of its viscosity, shear stress and shear rate.

As the load on oil increases, the shear rate also increases resulting in gradual increase in the shear stress. The shear stress and shear rate relation for the chilli seed oil gives a linear graph which validates its change in viscosity. From the results it can be termed that the viscosity of oils increases with gradual load, enhancing the lubricant efficiency.

Fig. 10 Shear stress and shear rate graphs of CSO without graphene

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The graph specifies the viscosity of oils and the study of viscosity change would give us the lubricant specifications.
The oils demonstrate non-newtonian behaviour and this behaviour occurs due to the change in local interactions in the fluid produced by shear relaxes in timescale similar to the period 1/ω. [21] and because of the causality principle and to comply with kramerskronig relations these fluids possess finite shear modulus G' and behave as non-Newtonian.

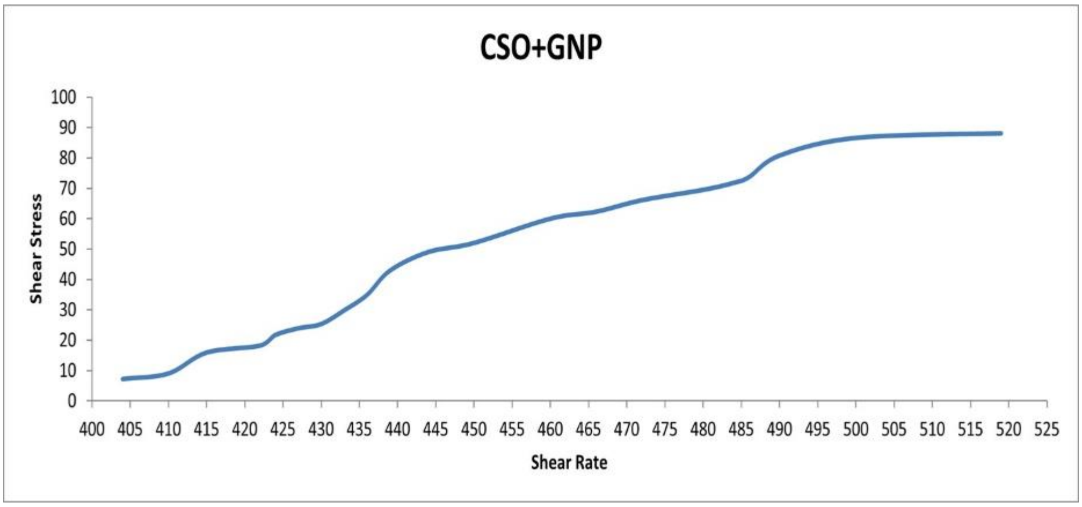

Fig. 11 Shear Stress Shear Rate Graphs Of CSO With Graphene**.**

After addition GNP the oils exhibits Newtonian behaviour. The ideal value of R^2 for an efficient lubricant should range between 0.998 to 0.999 [22]. The chilli seed oil indicates the Newtonian behaviour as $R^2 = 0.999$ which signifies the chilli seed oil as a Newtonian fluid. The chilli seed oil exhibits Newtonian nature due to each π bond contained in a cis configuration fatty acid, a fold may occur in a straight chain, avoiding the chains to be close to each other, i. e. the fatty acids with a higher number of double bonds do not have a rigid, fixed structure, causing them to be less densely packed.

Viscosity measurement and evalvation of rheological behaviour

For the rheological parameter of viscosity, it is observed that viscosity shows an increase with the change in torque. As the torque increases in the rheometer, the viscosity gradually increases for the oils. This result has been validated by the fact that fatty acid composition is a major factor in determining the viscosity of oil. The monosaturated fatty acids exhibit high viscosity and it is attributed to the fact that increase in double bond impacts packing of fatty acid molecules of oil making them less rigid. The higher viscosity of chilli seed oil is attributed to presence of higher content of saturated fatty acid [23].

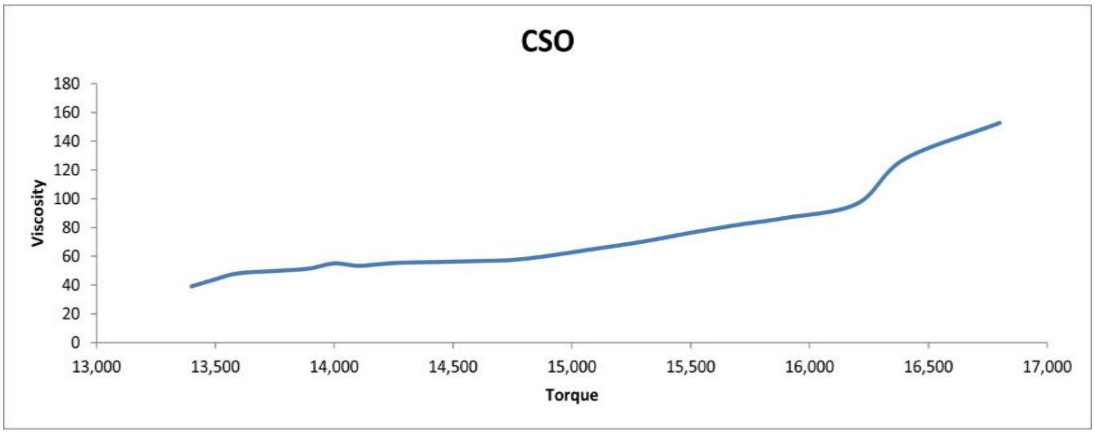

Fig. 12 Viscosity torque graph of CSO without graphene

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It is evident that the viscosity of all oil increased gradually with increasing load. However, chilli seed oil showed the poor performance graph at its natural state.

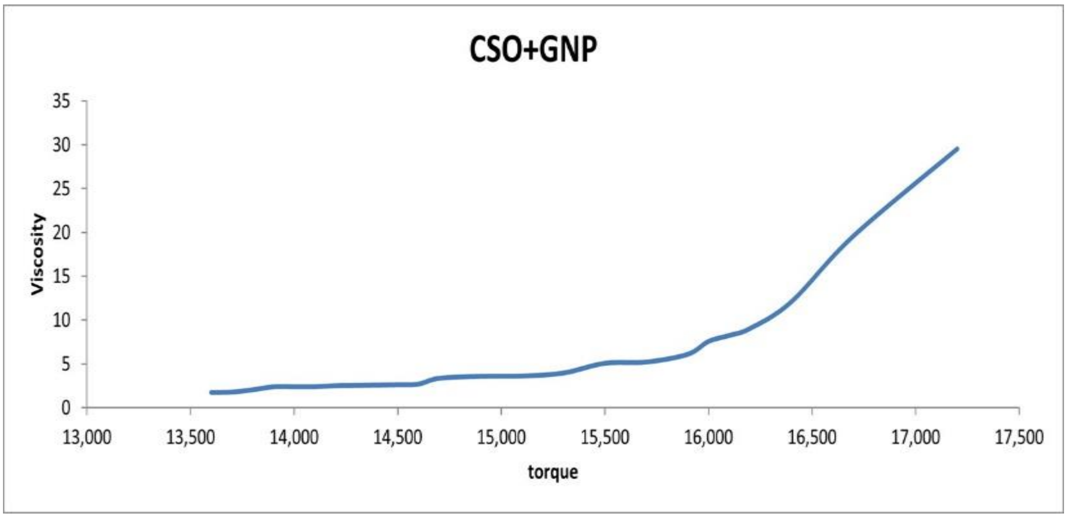

Fig.13 Viscosity torque graph of CSO with grapheme

The graph shows a noticeable change in the viscosity of oils after the addition of graphene nano particle. Efficient changes discerned in chilli seed oil as noticeable change could be observed.. It is because of the addition of graphene np greater molecular moments are introduced in the oil composition and in addition to that introduction of graphene reduces the intermolecular space that considerably decreases the intermolecular bond forces contributing to the hike in the viscosity.

IV.CONCLUSION

In this research work,.Chili seed oil was mixed with graphene nanoparticles (GNP) to evaluate changes in rheological behaviour, as GNP offers unique frictional and wear properties not found in conventional materials.Chili seed oil (CSO) analysis revealed a total phenolic content (TPC) of 18.34 ± 0.01, supporting its medicinal uses. The hue angle was 14.3 ± 0.54, indicating a colour between pure red and yellow. The density was 0.932 ± 0.002, within the ideal range for lubricating oils (0.91 to 0.93 g/ml), and the contact angle was 27.032 ± 0. 698.The chilli seed oil shows high viscosity influenced by its higher saturated fatty acid content. The viscosity of CSO+ GNP increases with change in torque because by the addition of graphene np greater molecular moments are introduced in then oil composition and in addition to that introduction of graphene reduces the intermolecular space that considerably decreases the intermolecular bond forces contributing to the hike in the viscosity.

Chili seed oil (CSO) exhibited Newtonian behaviour, as indicated by an R^2 value of 0.999, confirming it as a Newtonian fluid. This characteristic is attributed to the presence of π bonds in the cis configuration of its fatty acids. The CSO when mixed with GNP shows changes in the characteristic features as the contact angle increased from 27.032 to 153 and this was because of the weaker intermolecular forces between oil molecules changing by introduction of GNP and a solid surface compared to the cohesive forces within the oil itself changing by introduction of GNP bonds leading to a greater tendency for the oil to form a droplet with a larger contact angle on the surface. Essentially, the oil molecules prefer to stick to each other more than to the solid surface, resulting in a higher contact angle.

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