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ORIGINAL ARTICLE

Influence of different surfactants on the stability and varying concentrations of TiO_2 nanoparticles on the rheological properties of canola oil-based nanolubricants

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Abstract

The present study addresses the dispersion stability and rheological analysis of canola oil-based nanolubricants. The stability of canola oil-based nanolubricant was examined under different surfactants and different particle to surfactant mass ratios for the period of 30 days. The experimental results demonstrated that nanolubricants with surfactant significantly improve the dispersion stability as compared to non-surfactant lubricant. However, on the basis of visual inspection and absorbance value, the highest stability was recorded for the sample containing TiO_2 to Triton X-100 mass ratio of 1:3. In addition, the influence of different concentrations of nanoparticles on the rheological behaviour of canola oil-based nanolubricants was also assessed. The outcomes showed that the nanolubricants exhibited Newtonian behaviour. The viscosity of nanolubricants increased with increase of nanoparticles concentration and decreased with temperature. Experimental values were compared with available viscosity models and a new correlation model was proposed with the margin of deviation of 1.38%.

Keywords Dispersion stability · Vegetable oil · Rheology · Nanolubricants · Tribology

Introduction

Conservation of energy and deterioration of materials are the major challenges for any mechanical system. Friction and wear are the main roots for such challenges. However, a favourable lubricant can prevent these issues to a certain extent. Lubricants are competent to separate the contacts in relative motion and support the load carried by the tribo-pairs. Mineral oils are the first choice for any process in industries for lubrication due to their impressive physical and chemical characteristics. Thereby, the demand of minerals oils has been increasing progressively. However, these lubricants have not been considered as environmentally friendly lubricants for the reason of poor biodegradability, toxicity and improper disposal. It disturbs the ecological system throughout its life cycle, from the time of production to dumping. According to the forecast, they will only be used for the next 50 years (Katpatal et al. 2018). To save our ecosystem, researchers from different fields are emphasizing extensively on innovation of natural-based biomaterials (Lv et al. 2018) and tribologists are also exploring sustainable biodegradable lubricants and advise that vegetable oils may also be used for non-edible purposes such as fuels and industrial lubricants. It has been confirmed that edible oils have high viscosity index, low volatility, high flash point, high lubricity and are fully biodegradable (Zulkifli et al. 2013; Rani et al. 2015; Gupta and Harsha 2017). However, as far as tribological performance and oxidation stability of vegetable oils are concerned, they are not favourable as much as conventional lubricants. Nevertheless, inclusion of suitable additives in vegetable oil may limit such problems.

Recently, newly advanced materials with addition of nanoparticles have gained popularity with much possibilities (Lv et al. 2020). For instance, blending of micro-sized and nano-sized additives derived from metallic and non-metallic compounds in base oil has received extensive consideration in enhancing the tribo-performance of the base oil. These additives act as anti-friction modifier and improve the load capacity of the base fluid. The tribo-performance of various vegetable oils containing nanoadditives has been widely

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studied by researchers under both boundary and hydrodynamic lubrication regimes.

The tribological behaviour of chemically modified karanja oil with addition of Cu nanoparticles and zinc dialkyl dithiophosphate (ZDDP) as additives was conducted by Garg et al. (2017), which showed that the Cu nanoparticles and ZDDP improve the anti-wear and anti-friction properties of the oil. Gupta and Harsha (2018) studied the tribo-performance of sunflower oil by dispersing varying ranges of CuO and CeO_2 nanoparticles using four ball tester. For enhancing the oxidation stability, esterification of the oil was also carried out. Results stated that the oil containing nanoparticles showed excellent tribological performance, especially at lower concentration instead of higher concentration. Shafi et al. (2018) examined the tribological behaviour of avocado oil blended with copper nanoadditives. They reported that the copper nanoadditives form a protective layer between steel and aluminium contact, which results in reduction in friction and wear. Omrani et al. (2019) characterized the friction and wear performance of canola oil mixed with graphene and graphite additives and observed that 0.7 wt% of additives was the optimum concentration for canola oil. Moreover, a smoother surface of aluminium pins was obtained using these additives in base oil than that of oil without additives. The influence of nanoparticles under hydrodynamic lubrication regime has also been explored by various authors (Kalakada et al. 2012, 2015; Nicoletti 2014; Solghar 2015; Abass and Mohamme 2017). Their study declared that the blending of nanoadditives in base oil notably enhanced the performance of journal bearing in terms of fluid film pressure and load-carrying capacity. The above literature clearly reveals that the blending of nanoadditives into base oil remarkably improves the performance of base oil under both boundary and hydrodynamic lubrication regimes.

To achieve their potential and recognize the industrial applications, a stable suspension of nanolubricants/nanofluids is very essential. It is well known that the compatibility of the nanoparticles with the base fluid is not good and has a tendency to form large nanoclusters due to high surface energy. As time passes, these clusters are separated and precipitated out from the base fluid. Nanolubricants with large aggregates not only lose their lubrication mechanisms such as rolling effect and mending effect, but also create some hindrances such as clogging and abrasion issues between the tribo-pairs.

Till now, dispersion stability has been the most challenging task that became a constraint for practical applications and further evolution of nanolubricants. To mitigate this issue, there are various solutions to enhance the dispersion stability to a certain extent, for instance, sonication time, sonication method, mixing of surfactant and particle size. However, capping of nanoparticles with a suitable surfactant has been considered as an efficient and economical method to improve the stability of nanofluids for a long period (Haddad et al. 2014; Chen et al. 2019).

To date, numerous studies have been devoted to the influence of surfactants on the dispersion stability of water-based and oil-based nanofluids and are presented in Table 1. However, as depicted in Table 1, limited investigations have been performed for mineral oil-based and biodegradable nanolubricants and most of the investigations are related to heat transfer applications. Further, the effect of various surfactants and their different concentrations on the stability of nanofluids is also not widely addressed in the literature. The dispersion stability of any nanofluid not only depends on the surfactant, but also on its concentration. A low amount of surfactant may lead to aggregation of nanoparticles which results in sedimentation, whereas a high amount of surfactants may affect the thermo-physical characteristics of nanofluids. Paramashivaiah and Rajashekhar (2016) investigated the stability behaviour of simarouba oil containing graphene nanoparticles with the addition of different mass fraction ratios of graphene to surfactant of SDS and SDBS surfactants using UV–Vis spectroscopy. The results revealed that the graphene to surfactant mass ratio (1:4) shows the highest absorbance and results in higher stability. Das et al. (2016) demonstrated the role of surfactants on the stability of nanofluids and found that the SDBS surfactant with particle to surfactant mass ratio of 2:1 exhibits the highest stability than the CTAB and SDS surfactants. Al-Waeli et al. (2019) investigated the outcomes of six different surfactants and their different concentrations on the dispersion stability of nanofluids and observed that a solution of ammonia and tannic acid with 0.1 ml concentration showed the highest stability time (88 days) than other surfactants.

The rheological characterization of any lubricant plays a key role in some engineering components where the performance of the machine element is entirely dependent on lubricant viscosity such as journal bearings. It has been reported that the viscosity of the base lubricant is enhanced by dispersing the nanoparticles. In this study, the flow behaviour and viscosity of the nanolubricant were examined under different sets of operating parameters such as shear rate, temperature and shear stress. Till now, a few studies have been carried out on the rheological behaviour of synthetic/mineral oils and vegetable oil-based nanolubricants (Hemmat Esfe et al. 2016a, b; Cortes and Ortega 2019; Rejvani et al. 2019). Nevertheless, the rheology of different vegetable oils-based nanolubricant needs to be explored more. The comparative viscosity analysis of LB2000 vegetable oil-based lubricant and PriEco6000 unsaturated polyol ester by adding varying concentrations of graphite nanoparticles was conducted by Su et al. (2016). The outcomes showed that the viscosity enhances with the rise of nanoparticle concentration

Table 1 A review of available studies on dispersion stability of water-based and oil-based nanofluids using different surfactants

Authors	Base fluid	Nanoadditives	Surfactant	Stability measurement apparatus	Application	Major finding
Kathiravan et al. (2010)	Distilled water	Cu	SDS	TEM	Heat transfer	Suspension of water-based nanofluids was stabler than 10 h
Yousefi et al. (2012)	Double distilled water	MWCNTs	Triton X-100	TEM	Solar system	Nanofluid was stable up to 10 days
Yousefi et al. (2012)	Double distilled water	Al_2O_3	Triton X-100	TEM	Solar system	Nanofluid was stable up to 3 days
Priya et al. (2012)	Water	CuO	Triton	Visual monitoring and Zeta potential	Heat transfer	Highest stability of water-based nanofluids was observed with the addition of particle to surfactant mass ratio of 2.5:1
Colangelo et al. (2016)	Therminol 66 oil	Al_2O_3	Oleic acid	FTIR spectrometer	Solar system	Nanofluids with surfactant showed excellent dispersion stability than fluid without surfactant
Su et al. (2016)	LB 2000 vegetable oil and PriEco 6000 oil	Graphite	–	Zeta potential	Cutting fluid	PriEco 6000 oil containing graphite particles showed better stability than LB 2000 vegetable oil nanofluid
Ghasemi et al. (2018)	SAE 10 engine oil	TiO_2	Oleic acid	DLS	Industrial lubricant	Results stated that the addition of surfactant improved the dispersion stability
Bahararuddin et al. (2019)	Mineral oil	SiO_2	CATB	Visual observation	Transformer oil	Addition of surfactant in nanofluid reduces the surface activity and prevents formation of aggregates

for both oils. However, PriEco6000/graphite-based lubricant shows higher viscosity than the LB2000 vegetable oil-based nanolubricant. Lately, Shafi and Charoo (2019) measured the rheological behaviour of three different vegetable oils containing TiO_2 nanoadditives at different temperatures and observed that all the vegetable oil-based nanolubricants exhibit Newtonian behaviour at a particular temperature. Moreover, the viscosity of all the nano-oils increases as the concentration of nanoparticles increases and avocado oil shows the highest viscosity than apricot and hazelnut oils. Sajeeb and Rajendrakumar (2019) investigated the flow behaviour and viscosity of hybrid CeO_2/CuO coconut-based nanolubricants for a temperature range and different shear rates. They revealed that the hybrid nanolubricants exhibit non-Newtonian behaviour and Newtonian behaviour at low shear rates and high shear rates at a particular temperature, respectively. Further, they also proposed a regression model with the function of nanoparticle concentration for a broad range of temperature.

Lastly, it can be concluded from the above literature that in spite of growing attention towards biodegradable lubricants, the stability and rheological analysis of vegetable oil-based nanolubricants have not been investigated significantly. Moreover, the assessment of dispersion stability of vegetable oil-based nanolubricants using different surfactants has also not been explored, which indicates a major gap in research. According to the authors' knowledge, there has been no comprehensive work dedicated in the literature that deals with the study of dispersion stability and rheological behaviour of canola oil-based nanolubricants. In this view, the present study investigates the dispersion stability of canola oil-based nanolubricant using different surfactants and different particle to surfactant mass ratios. The stability of prepared nanolubricants has been examined experimentally using UV–Vis spectroscopy and visual monitoring for a period of 30 days. For this case, the amount of TiO_2 nanoparticles was kept constant (i.e. 0.04%). In addition, the rheological behaviour of canola oil-based nanolubricants containing different volume fractions of nanoparticles at different temperatures and different shear rates has also been investigated. The findings of this novel research could offer a roadmap for future advances in the field of biodegradable nanolubricants and also provide inspirations to the researchers that the physical properties of vegetable oil-based nanolubricants such as dispersion stability and rheological behaviour play a significant role. The work process of the current experimental work is illustrated in Fig. 1.

Table 2 Properties of TiO_2 nanoparticles

Properties	Values
Particle size (nm)	10–25
Density (g/cm^3)	3.9
Purity (%)	99.9
Specific surface area (m^2/g)	> 30
Appearance	White powder
Crystal form	Anatase

Experimental strategy

Materials

For this work, dry titanium dioxide (TiO_2) nanoparticles was used. The physical characteristics of nanoparticles are presented in Table 2. TiO_2 is widely used for various applications such as preparation of nanofluids, due to its impressive properties in terms of covering power, chemical stability, heat resistance and non-toxicity. Four different types of surfactants, viz. Triton X-100, SDS, oleic acid and Tween-20, were used to ensure better dispersion stability of nanoparticles in base oil. The specifications of surfactants are given in Table 3. Non-ionic surfactants were chosen due to very less toxicity and suitability with the oil-based nanolubricants (Azman and Samion 2019). However, one anionic surfactant

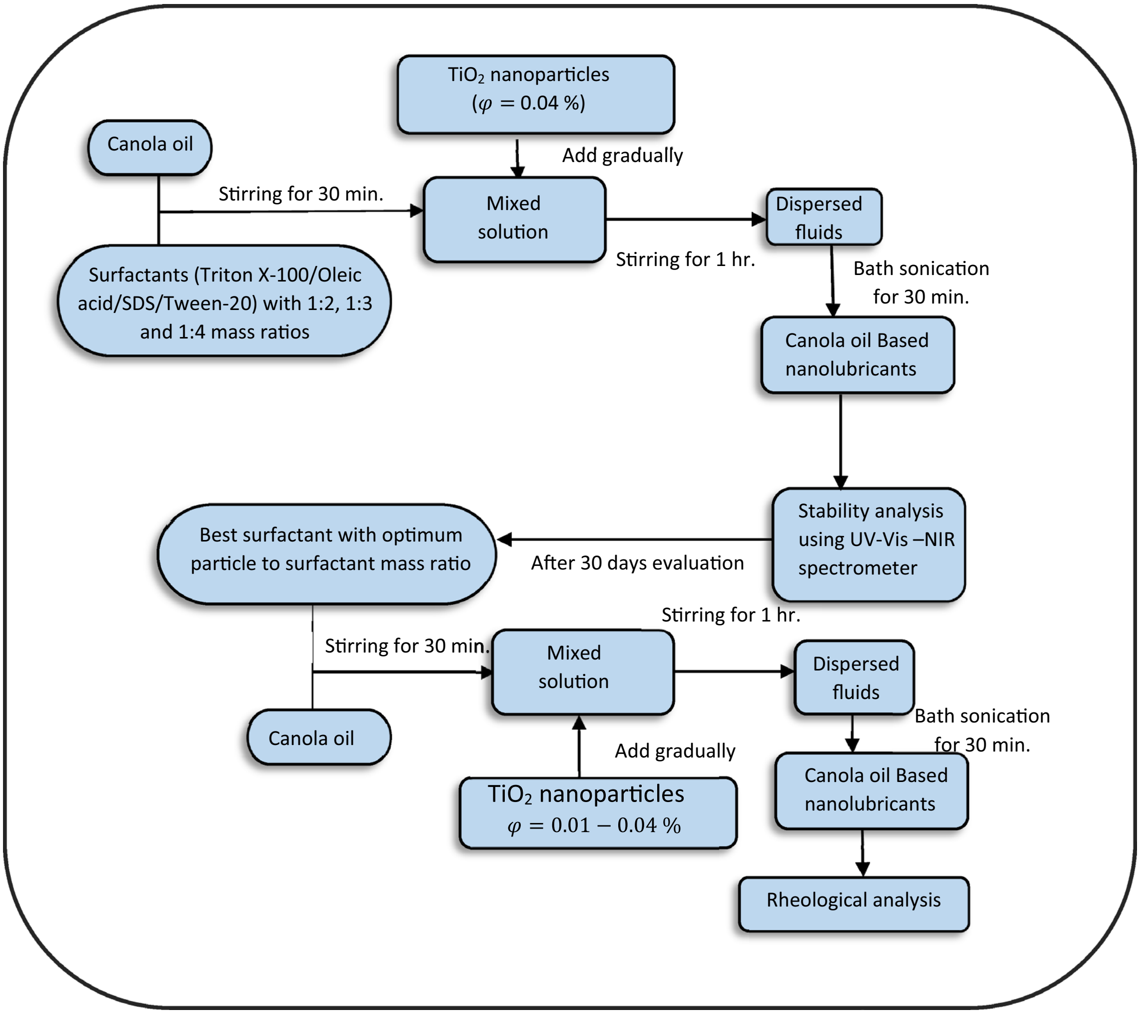


Fig. 1 Work process of the current experimental work

Table 3 Specifications of surfactants

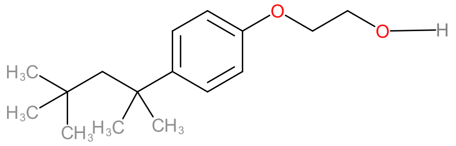
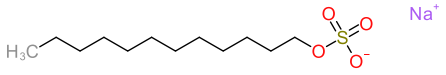
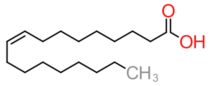
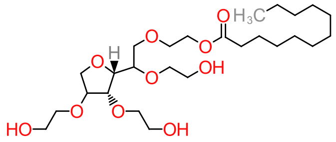
Surfactants	Chemical name	Molecular structure	Density (g/cm^3)	Appearance	Type
Triton X-100	Octyl phenol ethoxylate		1.07	Clear liquid	Nonionic
SDS	Sodium dodecyl sulfate		1.01	White powder	Anionic
Oleic acid	Oleic acid		0.89	Clear liquid	Nonionic
Tween-20	Polysorbate		1.1	Clear liquid	Nonionic

Table 4 Properties and fatty acid compositions of canola oil

Properties	Values
Density (g/cm^3)	0.918
Kinematic viscosity at 40 °C (mm^2/s)	37.71
Kinematic viscosity at 100 °C (mm^2/s)	8.59
Viscosity index	216.47
Appearance	Clear and bright
Flash point (°C)	275–290
Specific heat (J/g at 20 °C)	1.910–1.916
Smoke point (°C)	220–230
Pour point (°C)	− 18
Fatty acids composition (%)	
Monounsaturated	63
Polyunsaturated	31
Total unsaturated	94
Saturated	7

was also used to identify the compatibility with canola oil. All the above chemicals were supplied from HPLC Pvt. Ltd. Mumbai (India), and were used without any further purification. As the base lubricant, canola oil was imported by Borges India Pvt. Ltd. New Delhi (India). Canola oil is an edible oil and has the ability to be used directly as a lubricant without any further chemical modification (Biresaw et al. 2011). Canola oil is biodegradable and non-toxic in nature and does not create any hazardous issues to humans, animals and environment. The properties and fatty acid compositions of canola oil are tabulated in Table 4. Vegetable oils composed of a high proportion of monounsaturated fatty acids provide better lubricity and ensures superior lubricant properties in terms of viscosity, viscosity index and oxidation stability (Shafi and Charoo 2019).

Characterization of nanoparticles

The morphology of the TiO_2 nanoparticles was examined under FESEM (Quanta FEG 200) at an operating voltage of 15 kV and working distance of 9.8 mm, and the elemental characterization was done using Zeiss EVO18. Phase analysis and the size of nanoparticles were determined using X-ray diffractometer (Rigaku SmartLab). The XRD pattern was recorded under 1.540 Å Cu–Kα radiation source in 2θ range from 20° to 70° with step size 0.02 and scan rate of 1^0 min^{-1}.

Preparation of nanolubricants

All the samples of nanolubricants for stability analysis and rheological analysis were formulated by means of two-step method. For stability evaluation, 13 samples of different surfactants (Triton X-100, oleic acid, SDS, and Tween-20) with three different particle to surfactant mass ratios (i.e. 1:2, 1:3 and 1:4) including sample without surfactant were formulated. The volume fraction of TiO_2 was kept constant (i.e. 0.04%) and it was calculated using Eq. 1 (Das et al. 2016). The chemicals were weighed with an electronic balance (Shimadzu-AY 220, range 10 mg to 220 g, accuracy 0.1 mg).

$$\varphi = \left[\frac{\left(\frac{w}{\rho}\right)_{TiO_2}}{\left(\frac{w}{\rho}\right)_{TiO_2} + \left(\frac{w}{\rho}\right)_{\text{canola oil}}} \right], \quad (1)$$

where φ refers to the volume fraction of nanoparticles, w_{Tio2} is the weight of nanoparticles, $w_{\text{canola oil}}$ is the weight of the base fluid and ρ_{TiO2} and $\rho_{\text{canola oil}}$ are the density of nanoparticles and canola oil, respectively.

For these samples,the concerned surfactant and its particular concentration was added to 100 ml canola oil and stirred for 30 mins in a magnetic stirrer (Remi 20 ML plus) at 300 rpm to confirm homogeneous mixing. φ of 0.04% was then suspended gradually in the mixed solution and stirred for one more hour in a magnetic stirrer at 800 rpm so that the particles could be well distributed in the mixed solution. At the end, the obtained canola oil-based nanolubricant was sonicated in a bath sonicator (BR Biochem, Model: SB-800DTD, operating frequency: 40 kHz, ultrasonic power: 800 W) for 30 min to break down the loose clusters present in the sample even after magnetic stirring. Another set of samples for the rheology analysis was prepared in which a varying range of φ (0.01%, 0.02%, 0.03% and 0.04%) was added to canola oil. To achieve a better dispersion stability of the nanolubricant, the best surfactant and its particle to surfactant mass ratio (revealed from stability evaluation results) were added. The same preparation methodology of samples was adopted as described above and all the preparations were conducted at room temperature. The prepared samples for rheological analysis is presented in Fig. 2.

Stability evaluation of nanolubricants

Assessment of stability of nanofluids can be done by means of various methods such as visual observation, zeta potential, dynamic light scattering and UV–Vis spectrum. In the current work, the stability of the samples was not only done by visual monitoring, but also using ultraviolet-to-visible-to-near-infrared (UV–Vis–NIR) spectroscopy (Varian Cary 5000, Model: EL05013676 (Fig. 3), wavelength range: 200–3300 nm) for the period of 30 days, because stability cannot be finalized based on visual monitoring solely. Stability measurement using UV–Vis spectroscopy is considered the most reliable method (Paramashivaiah and Rajashekhar 2016). Stability results were presented by the recorded absorbance values as a function of time. For performing the test, initially, approximately 3 ml of base oil (canola oil) was poured into both quartz cuvettes (outer dimensions: 12 mm × 12 mm × 44 mm) to set the baseline. After adjusting the baseline, canola oil was considered as the reference and nanolubricant was considered as the specimen for the measurement. Each test was performed at least three times under wavelength ranges 260–800 nm with step 1 nm and 10 nm/s scan rate at room temperature, and the mean value was considered for the analysis. Before performing each test, the cuvette was cleaned thoroughly with water, followed by tissue paper to prevent contamination of the samples.

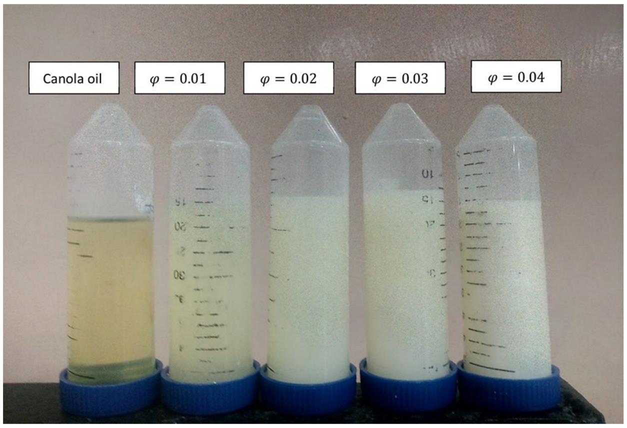


Fig. 2 Photograph of the prepared samples of canola oil-based nanolubricant of varying range of φ of TiO_2 nanoparticles for rheology analysis

Rheological analysis of nanolubricants

Rheological analysis of the prepared canola oil-based nanolubricant with varying ranges of φ of TiO_2 nanoparticles and base oil was examined using Anton Paar rotational rheometer (MCR 702 MultiDrive) as depicted in Fig. 4a. MCR 702 MultiDrive is a versatile type of rheometer as it comprises additional drive unit and torque transducer at the lower end. This facility is sufficient to provide precise results. However, in this study all the data were measured in combined motor transducer mode (single EC drive). The measuring head of the device comprises major parts such as air bearings to support the electronically communicated motor (EC), normal force sensor to measure the force during transient and steady state condition and high-resolution optical encoder to control the smallest speeds and angular deflections up to 50 nanoradians. Both radial and axial air bearings are dedicated to keep the shaft stabilized and aligned with the centre and to hold the weight of the rotating parts, respectively. In this study, cone-plate geometry (presented in Fig. 4b) with a plate diameter of 50 mm and 2° cone angle was used and the gap was maintained at 0.1 mm. Approximately, 0.57 ml of

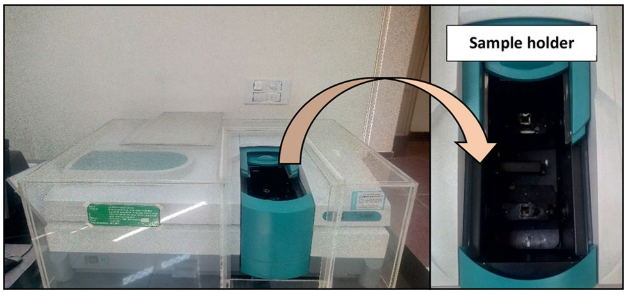


Fig. 3 UV–Vis–NIR spectroscopy (Varian Cary 5000, Model: EL05013676)

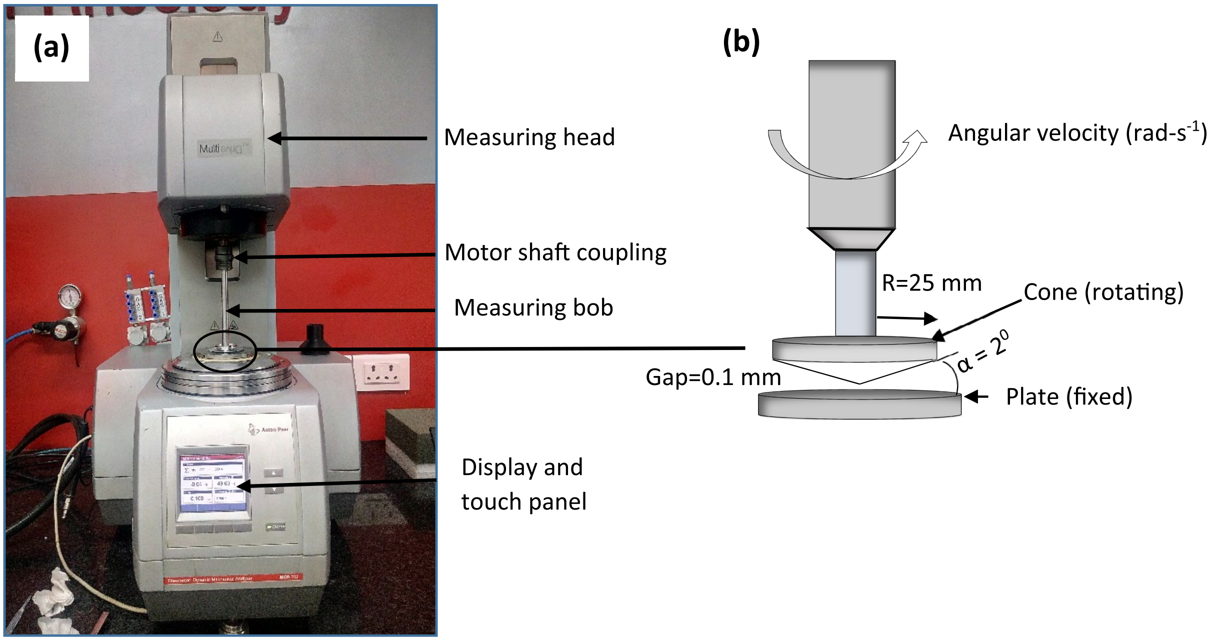


Fig. 4 **a** Anton Paar MCR 702 (MultiDrive) rheometer, **b** Cone-plate geometry

the prepared sample quantity was required to fill this gap. A Peltier system was attached through the flange ring to control the plate temperature. Further, before the experiments, the MCR 702 rheometer was calibrated with silicon oil at room temperature (25 °C). After doing three tests of silicon oil, the average uncertainty of the measured viscosities was found to be 2%. The dynamic viscosity and flow behaviour of the samples were measured for temperatures 20 °C–100 °C with a shear of 1–1000 s^{-1}.

Results and discussion

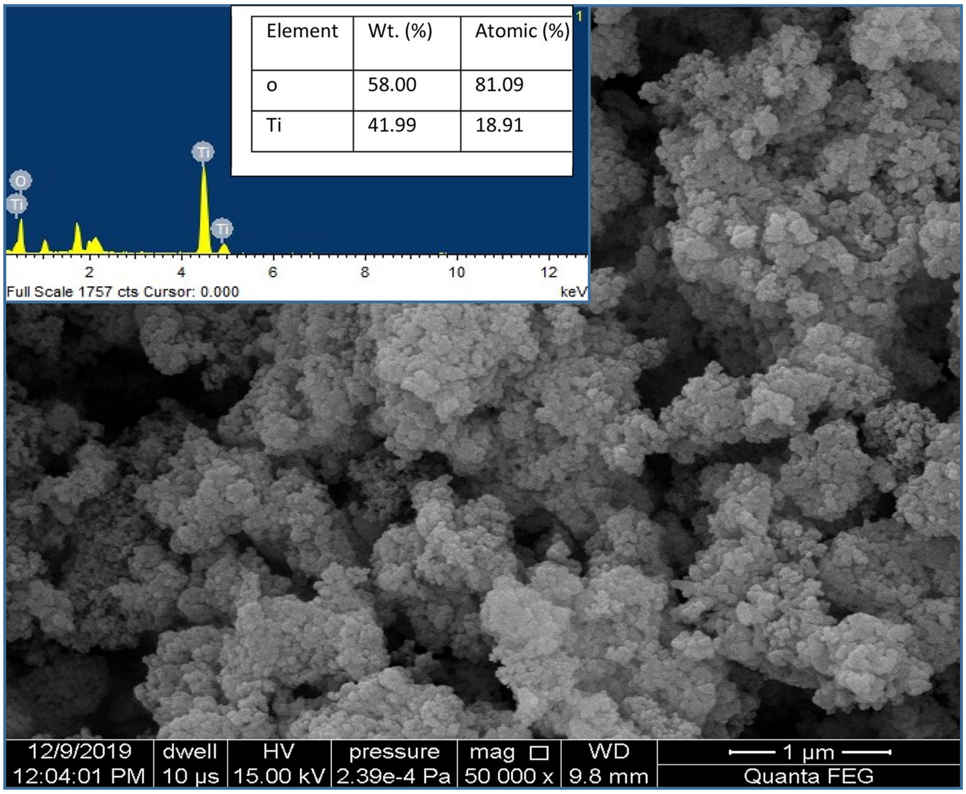
Morphology and structural study of TiO_2 nanoparticles

The surface morphology and structural study of TiO_2 nanoparticles were examined under FESEM and XRD techniques as shown in Figs. 5 and 6, respectively. It is observed from the FESEM image that the morphology of the nanoparticles is near-spherical. It can also be seen that nanoparticles form aggregates due to the presence of moisture. However, these loose aggregates were disaggregated by using a magnetic stirrer and applying ultrasound energy during sample preparation. Energy-dispersive X-ray spectroscopy (EDS) of TiO_2 nanoparticles confirmed the purity of nanoparticles as shown in the figure attached to Fig. 5. From the XRD pattern (Fig. 6), various peaks at 2θ value of 25.08°, 37.62°, 47.84°, 53.73°, 54.88° and 62.56° are indexed with the corresponding planes (110), (200), (211), (220), (221) and (311). The obtained peak positions agreed with the JCPDS card no. 21-1272 (TiO_2-anatase) and reported by the author (Ba-abbad et al. 2012). The particle size of TiO_2 nanoparticles can be obtained using Scherrer formula and it was observed with an average size of 13 nm, which was in good agreement with the size provided by the supplier.

Dispersion stability on the basis of visual monitoring

To examine the impact of different surfactants on the stability of the prepared samples, the samples were formulated with and without adding the surfactants to the colloids. Figure 7 illustrates the prepared nanolubricants with 0.04% volume fraction of TiO_2 nanoparticles used with and without different surfactants and different particle to surfactant mass ratios for a period of 30 days. It can be clearly stated that the sample without any surfactant (Fig. 7a) exhibits worst stability as compared to the other samples with respect to time. However, this sample was stable only on the first day of preparation and huge sedimentation was seen within just 5 days of preparation. The outcome of adding different surfactants into the suspension with different particle to surfactant mass ratios over the duration of 30 days is depicted in Fig. 7b–e. For a particular surfactant, it is seen that the surfactant remarkably improved the stability of the nanolubricant. It can be observed from Fig. 7b, that the addtion of Triton X-100 as a surfactant shows physical stability with less sedimentation even after 30 days of preparation at a particular

Fig. 5 FESEM image of TiO_2 nanoparticles with EDS spectra



Element	Wt. (%)	Atomic (%)
o	58.00	81.09
Ti	41.99	18.91

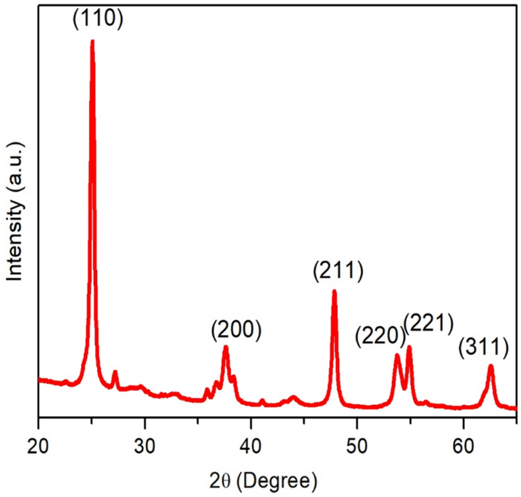


Fig. 6 XRD spectrum of TiO_2 nanoparticles

TiO_2:Triton X-100 mass ratio. Samples of oleic acid show stability only till 20 days of preparation as shown in Fig. 7c, while samples of SDS and Tween-20 seem to be physically stable just for a period of 15 days and sedimentation occurs within 25 days of preparation as presented in Fig. 7d, e, respectively.

Dispersion stability on the basis of UV–Vis–NIR spectroscopy

To validate and compare the results obtained from visual monitoring, the stability of the prepared samples with and without surfactants and different particle to surfactant mass ratios was also examined using UV–Vis–NIR spectroscopy for the duration of 30 days. After 5 days, the UV–Vis spectra of the samples were recorded without any prior ultrasonication. The absorbance was measured at the end of 5 days. Figure 8a–c shows the UV–Vis spectra of the samples with and without Triton X-100 surfactant and different TiO_2:Triton X-100 mass ratios of 1:2, 1:3 and 1:4, respectively, for the period of 30 days of standing time. It can be seen that the intensity of the absorbance of the sample without surfactant dropped from 3.67 to 0.513 just within a period of 5 days. It indicates that the huge sedimentation of the nanoparticles is observed without addition of surfactant. Therefore, for this case, absorbance was not carried out for other days. Surfactant (Triton X-100) added samples show good absorbance than sample without surfactant and the spectrum decreases negligibly with the elapse of time. It proves that the addition of surfactant in base oil remarkably enhances the absorbance, which results in reduction in sedimentation of the nanoparticles. Peak absorbance of the non-surfactant sample and different TiO_2:Triton X-100 mass ratios as a function

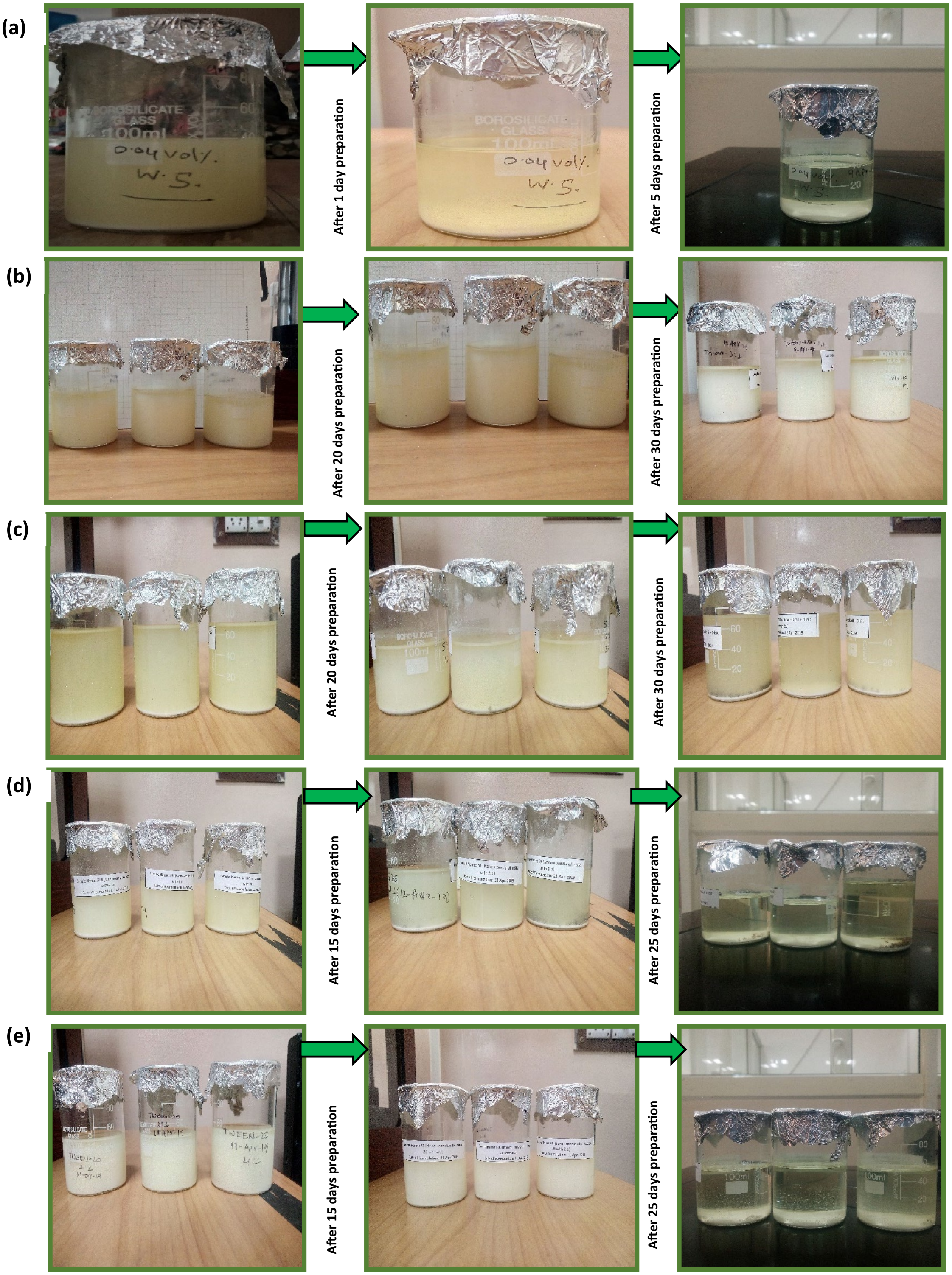


Fig. 7 Canola oil-based nanolubricant at the volume fraction of 0.04% with and without different surfactants and different particle to surfactant mass ratios for the period of 30 days. **a** Without surfactant, **b** Triton X-100, **c** oleic acid, **d** SDS, **e** Tween-20

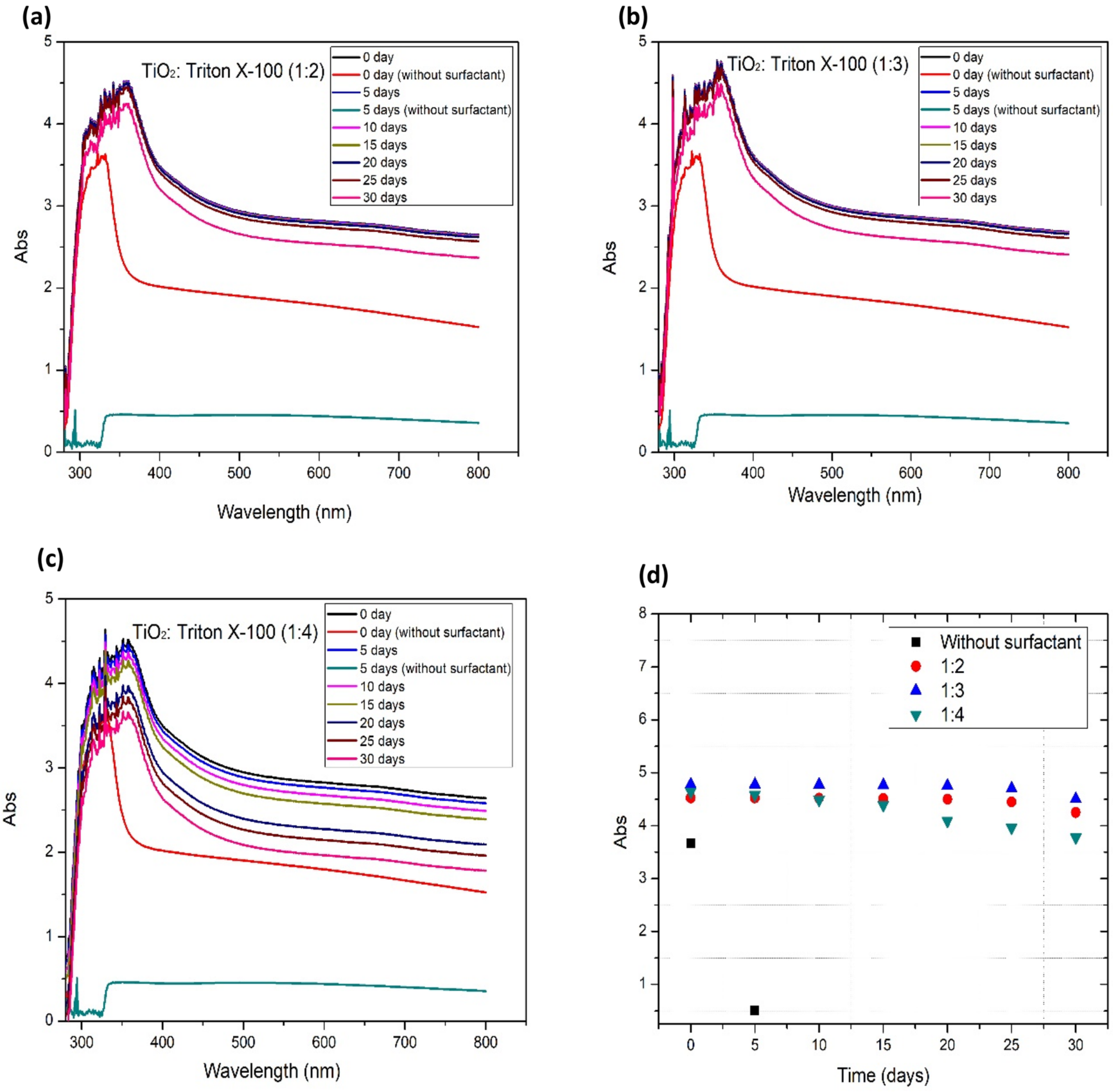


Fig. 8 **a–c** UV–Vis spectra and **d** absorbance vs. time of canola oil-based nanolubricants used with and without Triton X-100 surfactant and different TiO_2:Triton X-100 mass ratios for different period of time

of time are depicted in Fig. 8d. For the mass ratio of 1:3, minor decrement in absorbance with the elapse of time was observed as compared to the other samples. Moreover, at a particular time, the highest absorbance was also obtained for this case, followed by 1:2 and 1:4. A low concentration of surfactant may lead to low adsorption of Triton X-100 molecules with TiO_2 nanoparticles, and consequently weak steric repulsion. At higher concentration, molecules of surfactant form a thin dense layer on the surface of the particle, which cannot provide a strong steric repulsion. This may be because the dense molecular layers induce a higher linking between surfactant molecules adsorbed on to different particles, promoting further agglomeration of surfactant-coated particles. The peak absorbance is decreased by 6.13%, 5.74% and 18.53% for mass ratio of 1:2, 1:3 and 1:4, respectively. According to the suggestion of Yu and Xie (2012), the oil-soluble surfactants are preferred for dispersion of TiO_2 in oil. Miscibility of a non-ionic surfactant is done by its

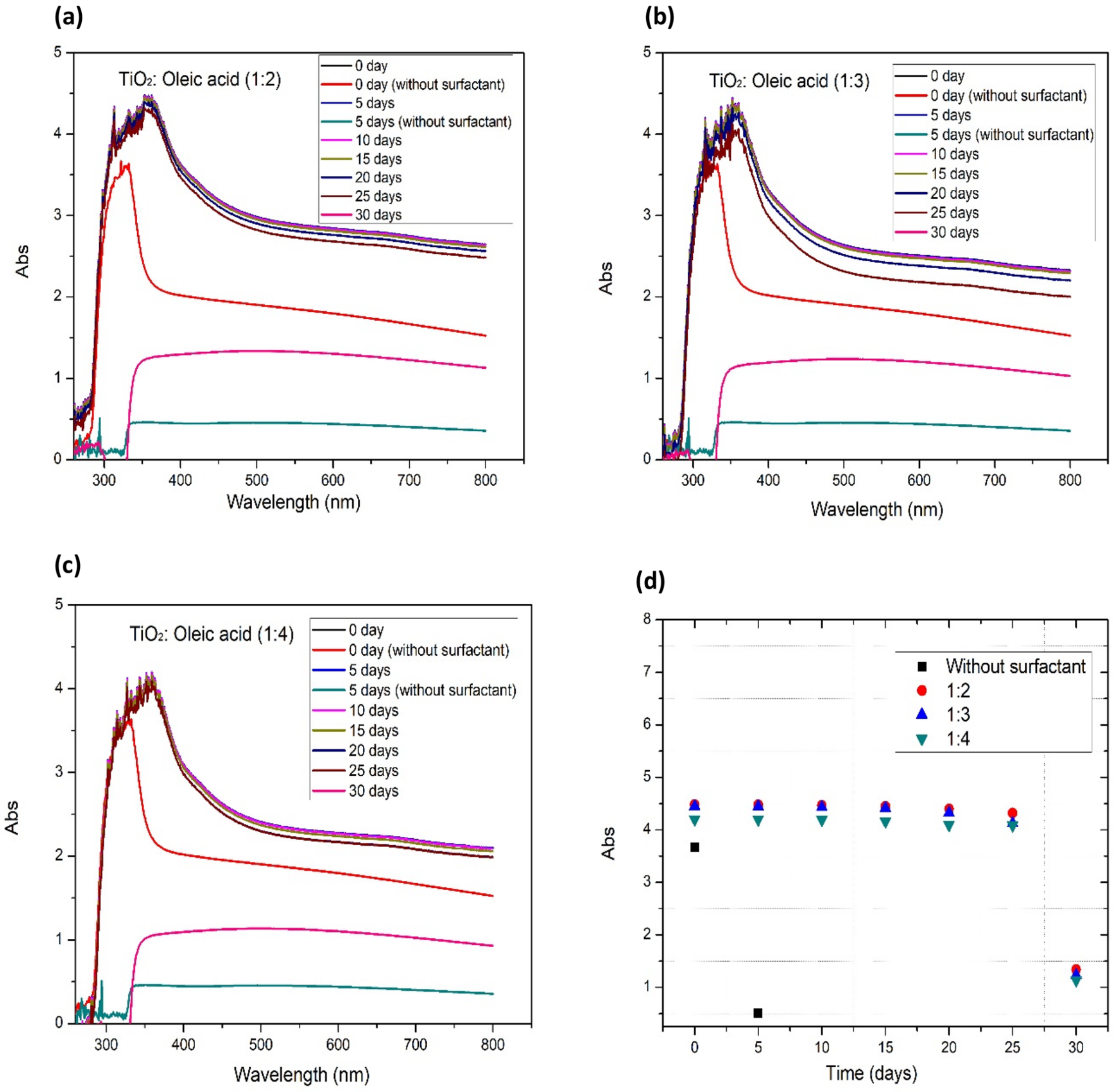


Fig. 9 **a–c** UV–Vis spectra and **d** absorbance vs. Time of canola oil based nanolubricants using with and without oleic acid surfactant and different TiO_2:oleic acid mass ratios for different periods of time

hydrophilic/lipophilic balance (HLB) number. Generally, surfactants having lower number of HLB are preferred for oil miscibility. Triton X-100 is reported to have the least HLB number (expect oleic acid) among other surfactants and hence is a good oil-miscible surfactant, so Triton X-100 shows excellent stability.

The UV–Vis spectra of different TiO_2:oleic acid mass ratios and non-surfactant sample after several days of standing time are presented in Fig. 9a–c. For samples without surfactant, mass ratio of 1:2, 1:3 and 1:4, the peak absorbance of 3.67 occurred at 353 nm, 4.48 at 322 nm, 4.45 at 352 nm and at 4.2 at 359 nm, respectively, just after preparation of the samples. At a particular mass ratio, UV–Vis spectra decreased with the passage of time. However, at a particular time, all the mass ratios have higher absorbance than that of the sample without surfactant. It

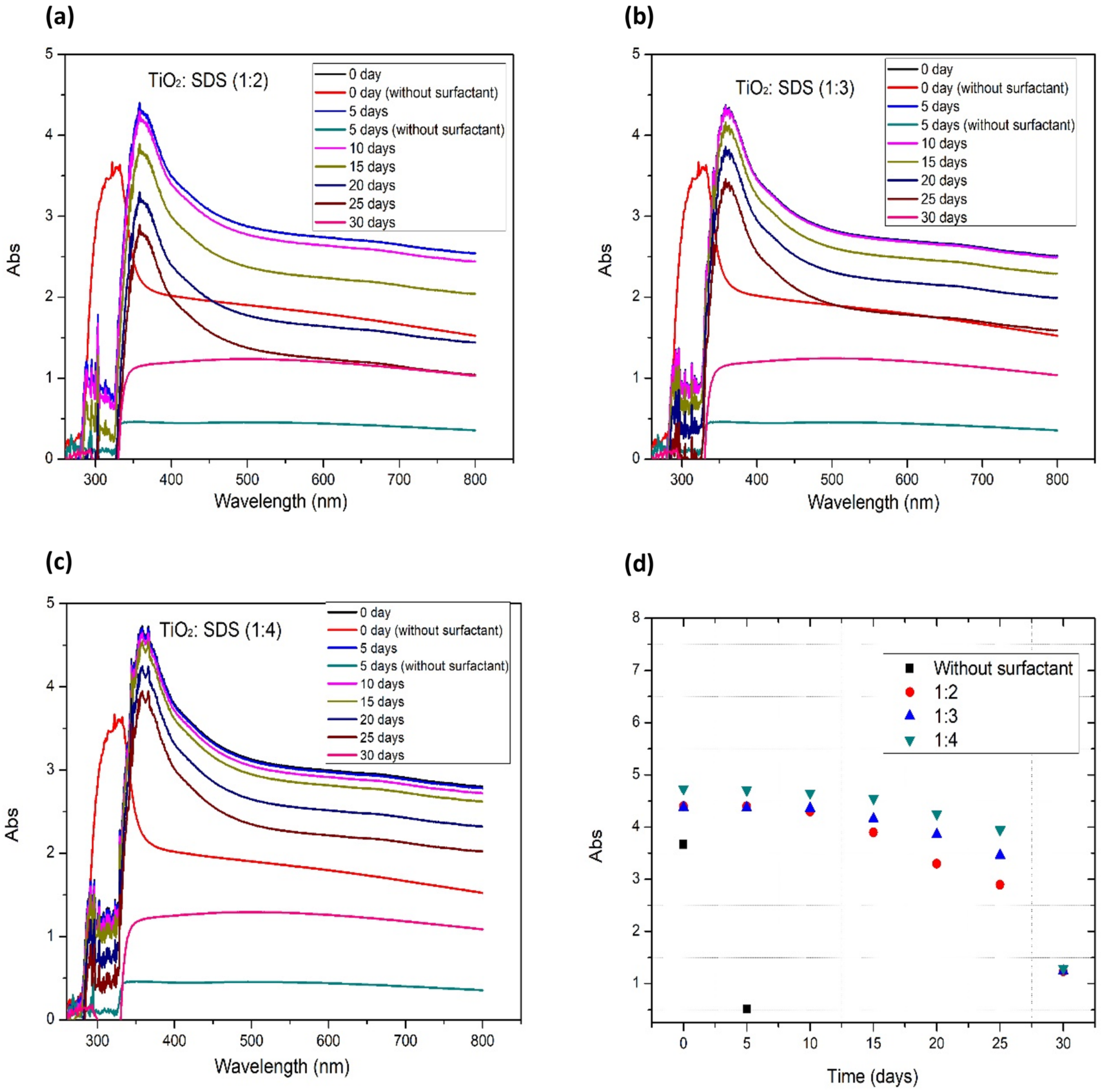


Fig. 10 **a**–**c** UV–Vis spectra and **d** absorbance vs. time of canola oil-based nanolubricants used with and without SDS surfactant and different TiO_2:SDS mass ratios for different periods of time

means nanoparticles coated with surfactant are able to keep the suspension stable for a longer time. Peak absorbance of samples with and without surfactant and different TiO_2:oleic acid mass ratios as a function of time are depicted in Fig. 9d. It can be seen for the samples with added surfactant that the absorbance is nominally decreased with time till 25 days of storage, but it decreased unexpectedly after 30 days of preparation. It means high sedimentation of nanoparticles occurred between 25 days and 30 days of storage. However, TiO_2:oleic acid mass ratio of 1:2 exhibits slightly higher absorbance value as compared to the others. It is important to note that inspite of having a smaller HLB number of oleic acid than Triton X-100, the samples are not able provide good stabilzation. This may be attributed to the fact that the vegetable oils already contain some amount of oleic acid which results in an excess amount of oleic acid, so the stability of nanolubricants may be affected. Further, a similar reason for variation in stability with oleic acid concentration

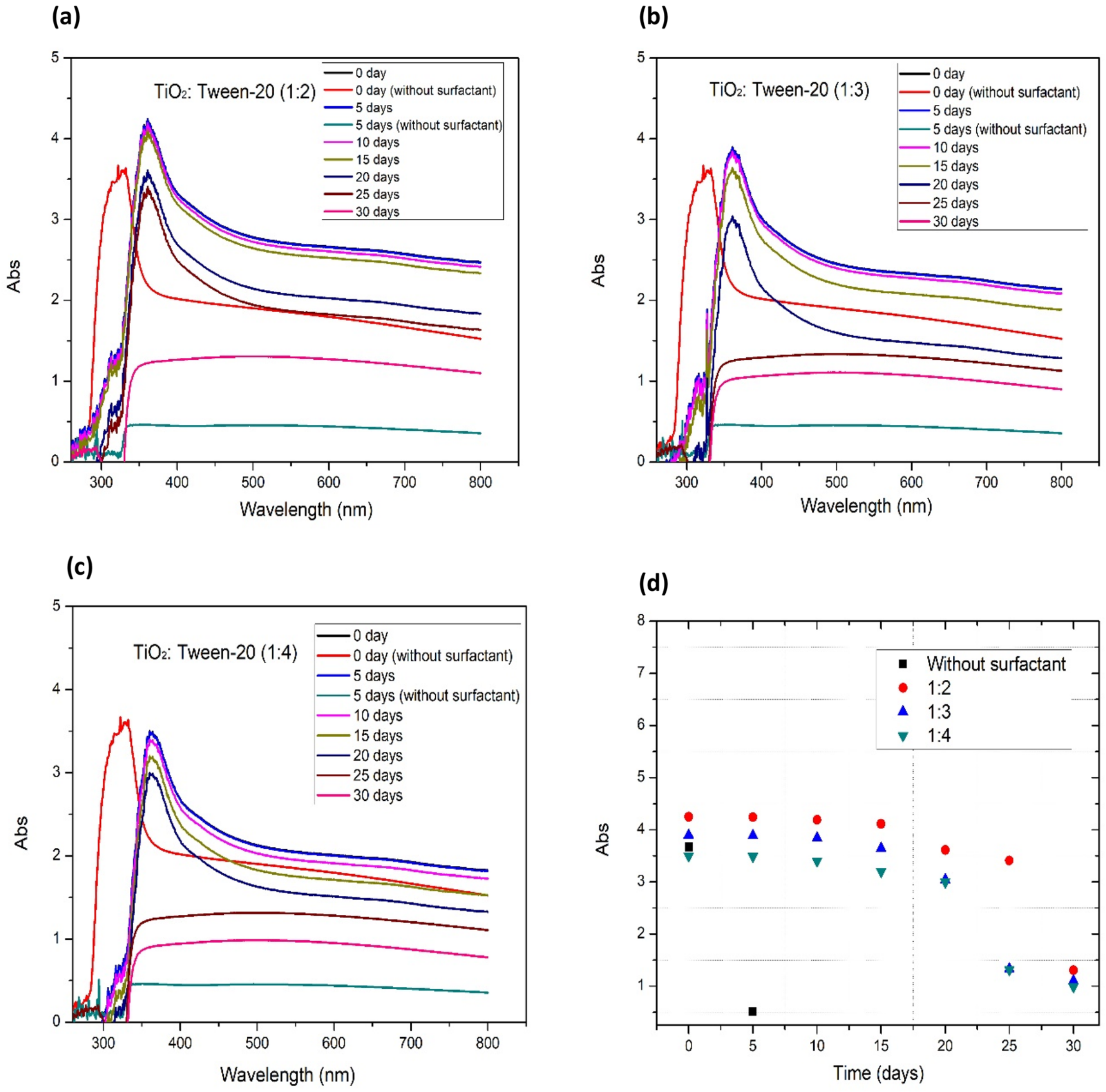


Fig. 11 **a**–**c** UV–Vis spectra and **d** absorbance vs. time of canola oil-based nanolubricants used with and without Tween-20 surfactant and different TiO_2:Tween-20 mass ratios for different periods of time

could be expected as discussed for Triton X-100. For the mass ratio of 1:2, 1:3 and 1:4, the peak absorbance decreased by 70.15%, 72.20% and 72.92%, respectively, at the final day of stability evaluation.

Absorbance of TiO_2:SDS mass ratios and non-surfactant sample as a function of wavelength for several days of standing time is illustrated in Fig. 10a–c. For freshly prepared samples of SDS, a peak absorbance of 4.4 was recorded at 358 nm, 4.38 at 357 nm and 4.73 at 358, respectively, for TiO_2:SDS mass ratios of 1:2, 1:3 and 1:4. UV–Vis spectra decreased significantly with the elapse of time. However, the observed absorbance values of these samples are more than those of the sample without surfactant over several periods of time, which leads to the stability of the nanolubricants. The variation in peak absorbance with and without surfactant samples and different TiO_2:SDS mass ratios with the elapse of time is shown in Fig. 10d. It can be seen for each mass ratio that absorbance decreases significantly with

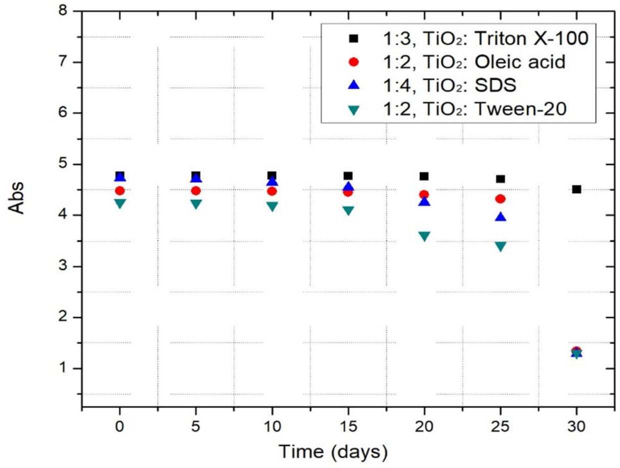


Fig. 12 Comparison of absorbance of different optimum particle to surfactant mass ratios with several days of storage time

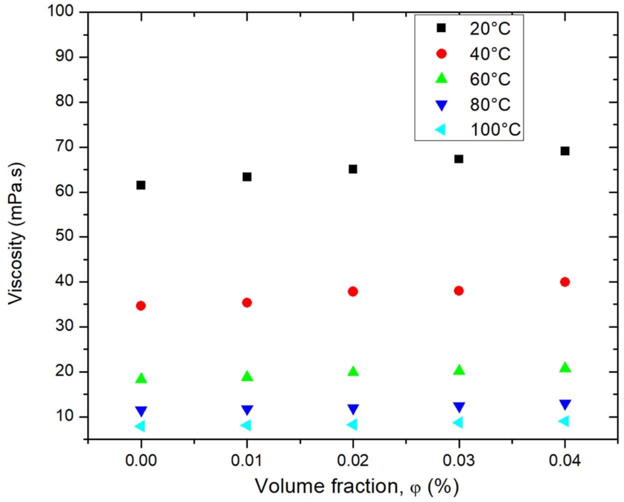


Fig. 13 Variation of viscosity with different volume fractions of nanoparticles for different temperatures

the elapse of standing time. The reason for this issue is the poor compatibility of SDS with canola oil and particles. It is well known that oil is a non-polar fluid due to the symmetrical molecular formation and uniform allotment of electrons. Further, TiO_2 normally possesses a structure which is neither totally ionic nor covalent (Xue et al. 1997). Thus, the prepared suspension could be considered non-polar. SDS is an ionic surfactant and usually such type of surfactants are not recommended for oil-based nanofluids due to poor electrostatic repulsion (Azman and Samion 2019). Moreover, at a particular time, the absorbance increases with the increase of mass ratio. For the mass ratio of 1:2, 1:3 and 1:4, the peak absorbance is decreased by 71.88%, 71.57% and 72.62%, respectively, at the last day (30 days) of stability evaluation.

Figure 11a–c illustrates the absorbance of TiO_2:Tween-20 mass ratios of 1:2, 1:3, 1:4 and non-surfactant sample as a function of wavelength for 30 days of standing time. The UV-Vis spectra of all the mass ratios is observed lower than the samples of Triton X-100, oleic acid and SDS with the storage of time. Moreover, the observed absorbance values for the Tween-20 samples are much lesser than the values of Triton X-100, oleic acid and SDS samples. The potential reason for poor stability is the highest HLB value of Tween-20 (approx. 17) than that of Triton X-100 and oleic acid, and hence it is the least oil-miscible surfactant. UV–Vis spectra decrease with the rise of TiO_2:Tween-20 mass ratio for a particular time. For samples of 1:2, 1:3 and 1:4, the peak absorbance of 4.25 is observed at 361 nm, 3.39 at 362 nm and 3.5 at 360 nm, respectively, immediately after preparation of the samples. Changes in the peak absorbance of the samples of TiO_2:Tween-20 mass ratios and non-surfactant sample as a function of the elapse of time are presented in Fig. 11d. As presented in the figure, the absorbance decreases significantly with elapse of time at each mass ratio, and at a particular time it decreases with the increase of mass ratio. It indicates that the aggregation rate of the nanoparticles increases resulting in the sedimentation of nanoparticles. In addition, the the same reason for changes in stability with Tween-20 concentration may be expected as discussed for Triton X-100. However, higher absorbance (i.e. 1.3) is to be observed for TiO_2:Tween-20 mass ratio of 1:2 at the end day of stability evaluation. In addition, for the mass ratio of 1:2, 1:3 and 1:4, the peak absorbance is decreased by 69.24%, 71.61% and 71.8%, respectively.

From the above stability results, it can be concluded that the TiO_2:Triton X-100 mass ratio of 1:3, TiO_2:oleic acid mass ratio of 1:2, TiO_2:SDS mass ratio of 1:4 and TiO_2:Tween-20 mass ratio of 1:2 show optimum particle to surfactant mass ratio with their respective mass ratios. But to identify the best surfactant with optimal particle to surfactant mass ratio in terms of absorbance between them,

Table 5 Percentage enhancement in viscosity

Enhancement in viscosity (%) $\frac{(\mu_{nf}-\mu_{bf})}{\mu_{bf}} \times 100$				
Temperature (°C)	0.01 vol.%	0.02 vol.%	0.03 vol.%	0.04 vol.%
20	3.07	5.92	9.53	12.41
40	2.1	9.2	11.2	15.4
60	2.5	8.8	10.1	13.5
80	2.1	3.9	7.9	12.6
100	2.43	4.41	10.78	13.89

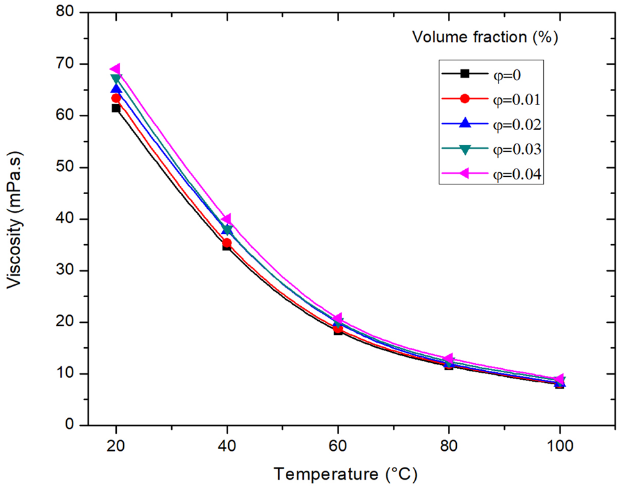


Fig. 14 Variation of viscosity with temperature for different volume fractions of nanoparticles

a comparison is made with the standing time of 30 days as presented in Fig. 12. It can be clearly observed that the TiO_2:Triton X-100 mass ratio of 1:3 has highest absorbance for a particular storage time in comparison to other optimal particle to surfactant mass ratios. The absorbance for this case decreased insignificantly with the elapse of time; hence, it can be stated that this sample shows excellent dispersion stability and the sample is stable even after 30 days of storage time. These experimental data are also well in agreement with the results of visual monitoring of this study.

Rheological studies of canola oil-based nanolubricants

Impact of TiO_2 nanoparticle concentration on viscosity

The curve between viscosity and volume fractions of nanoparticles for temperatures from 20 to 100 °C is illustrated in Fig. 13. The results show that as the concentration of the particle increases, the viscosity of the base fluid (canola oil) is also increased and the viscosity of the nanolubricants is higher than that of the base oil. Mostly, when the nanoparticles are blended in base fluid at a specific amount, they tend to attract each other in the base fluid due to van der Waals forces. Consequently, large clusters of the particles are formed and the movability of the lubricant layers with each other is interrupted which results in increase in viscosity. This trend is reported to be similar at all the temperatures. However, the enhancement in viscosity decreases with increase of temperature as shown in the figure. An increase in viscosity of the nanolubricants is in well agreement with the published results of the studies (Katpatal et al. 2018; Hemmat Esfe et al. 2016a). Table 5 presents the percentage enhancement in viscosity of the base oil due to the addition of nanoparticle concentrations for different temperatures. For varying range of nanoparticles from 0.01 to 0.04%, the percentage viscosity enhancement ranges are reported to be 3–12% 2–15%, 2–13%, 2–12% and 2–13% for temperatures 20 °C, 40 °C, 60 °C, 80 °C and 100 °C, respectively. Moreover, for a particular temperature, the percentage enhancement increases with the increase of concentration.

Impact of temperature on viscosity of canola oil based nanolubricants

Figure 14 shows the variation in viscosity with temperature for different nanoparticles concentrations in base oil. The outcomes show that the viscosity of nanolubricant decreases exponentially with the rise of temperature for all concentrations of nanoparticles. This is due to the decrease of the intermolecular adhesion force between nanoparticles and lubricant molecules with the rise of temperature as stated by Kole and Dey (2011). Further, the behaviour of viscosity of nanolubricants in regard to variation in temperature is similar to the behaviour of canola oil. The viscosity of all nanolubricants approaches canola oil's viscosity, especially at higher temperature. The observed trend is well in agreement with the studies of Hemmat Esfe et al. (2016a, b). With the temperature ranging from 20 to 100 °C, the viscosity of the canola oil and prepared nanolubricants are decreased by 87.16%, 87.24%, 87.34%, 87.01% and 86.99% for canola oil, $\varphi = 0.01$, $\varphi = 0.02$, $\varphi = 0.03$ and $\varphi = 0.04$, respectively.

Impact of shear rate on viscosity of canola oil-based nanolubricants

Viscosity as a function of shear rate ranging from 1 to 1000 s^{-1} for different volume fractions of nanoparticles at temperature of 20 °C, 40 °C, 60 °C, 80 °C and 100 °C is illustrated in Fig. 15. It can be noted from the figure that the viscosity of the canola oil and nanolubricants is unchanged with the increase of shear rate at a particular temperature; hence, it can be stated that the lubricant behaves as Newtonian. Moreover, the blending of TiO_2 nanoparticles with different concentrations in canola oil could not change the behaviour of the lubricant. The results agree with the review article of Sharma et al. (2016) that the fluid containing low nanoparticles concentration exhibits Newtonian behaviour.

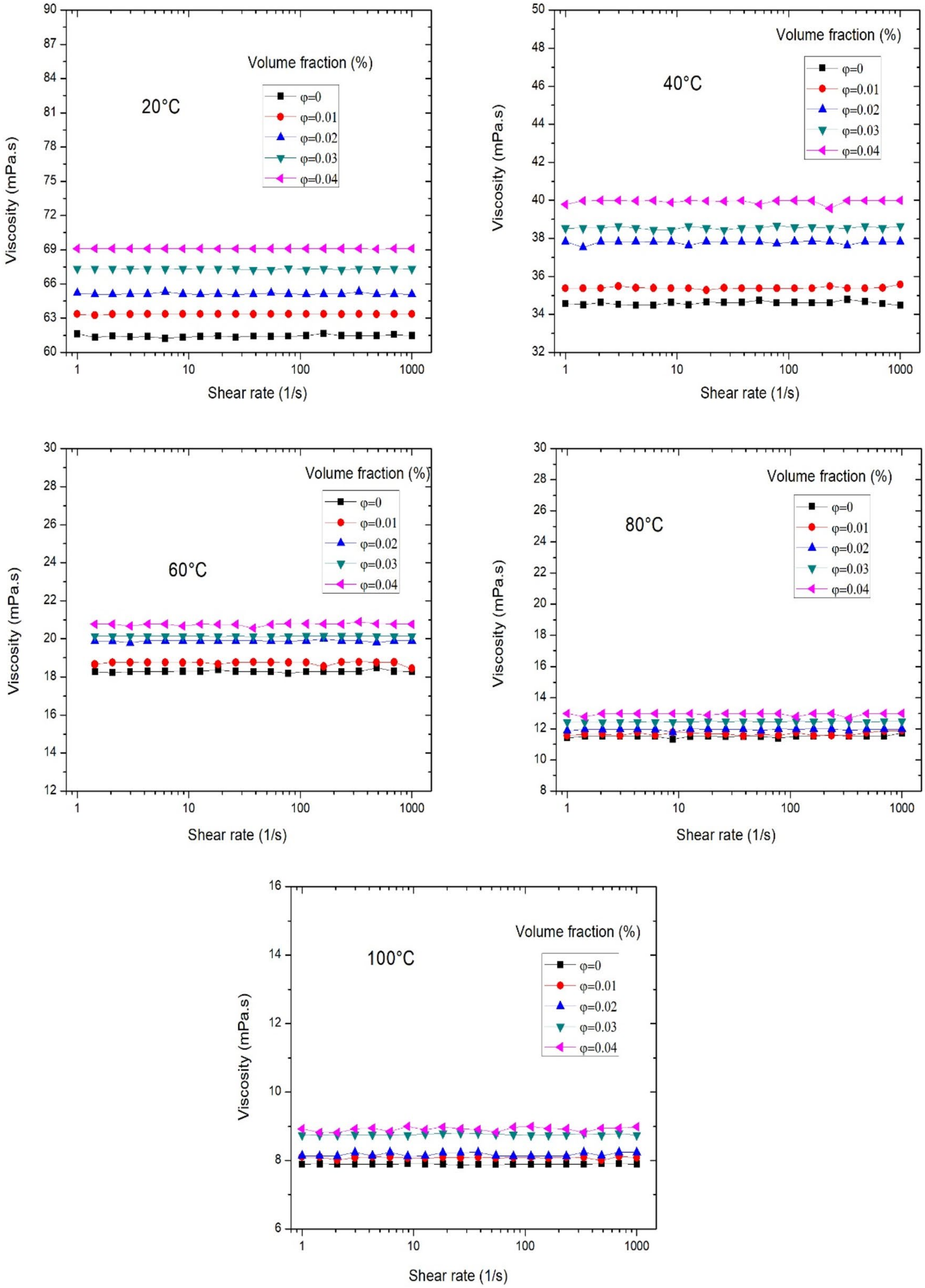


Fig. 15 Variation of viscosity with shear rate ranging from 1 to 1000 s^{-1} for different volume fractions of nanoparticles at temperature of 20 °C, 40 °C, 60 °C, 80 °C and 100 °C

Table 6 Proposed models for viscosity measurement of nanofluids

Model	Expression
Krieger and Dougherty (K-D) et al. (1959)	$\mu_{nf} = \mu_{bf}\left[1-\frac{\varphi}{\varphi_m}\right]^{-2.5\varphi_m}$, $0.495<\varphi_m<0.54$
Batchelor (1977)	$\mu_{nf} = \mu_{bf}\left(1+2.5\varphi+6.5\varphi^2\right)$
Wang et al. (1999)	$\mu_{nf} = \mu_{bf}\left(1+7.3\varphi+123\varphi^2\right)$
Esfe et al. (2014)	$\mu_{nf} = \mu_{bf}\left(1+11.61\varphi+109\varphi^2\right)$

Theoretical predictions of viscosity of canola oil-based nanolubricants

To evaluate the viscosity of nanofluids/nanolubricants theoretically, various viscosity models have been offered. In the present study, the experimental results of viscosity of canola oil-based nanolubricants have been validated and compared with four different models, viz. Krieger and Dougherty (K-D) et al. model (Xue et al. 1997), Batchelor model (1977), Wang et al. model (1999) and Esfe et al. model (2014). The expression of these models is given in Table 6. Figure 16 illustrates the comparison of experimental results of viscosity of canola oil-based nanolubricants with the theoretical models for different temperatures. The figure reveals that all the three models are not able to predict the viscosity accurately. However, the values evaluated from K-D et al. model and Batchelor model are much closer to the experimental values and the closeness increases with the rise of temperature. The recorded variation between the theoretical and experimental results is observed due to the morphology and aggregation effects of the nanoparticles. In addition, the percentage deviation between the theoretical and experimental data is shown in Table 7. To minimize the deviation and predict the viscosity more accurately, a non-linear regression model has been proposed in reference to the experimental values which follows quadratic polynomial equation and is expressed as:

$$\mu_{nf} = \mu_{bf}\left(a_o + a_1\varphi + a_2\varphi^2\right). \tag{2}$$

The values of correlation coefficients a_o, a_1 and a_2 and R-squared (R^2) values for the temperature, 20 °C, 40 °C, 60 °C, 80 °C and 100 °C, have been calculated from the respective experimental results and are shown in Table 8. This proposed theoretical model is able to predict the viscosity of canola oil-based nanolubricants with the limitations of volume fraction of nanoparticles ranging from 0 to 0.04% and temperature ranging from 20 to 100 °C.

To evaluate the accurateness of the proposed regression model, the margin of deviation is calculated as:

$$\text{Margin of deviation} = \frac{\left(\mu_{nf}\right)_{\text{Experimental}} - \left(\mu_{nf}\right)_{\text{Proposed}}}{\left(\mu_{nf}\right)_{\text{Proposed}}} \times 100. \tag{3}$$

The margin of deviation of varying range of nanoparticles' concentration at different temperatures is presented in Fig. 17. The figure demonstrates that the proposed model perfectly correlates with the experimental results, as the deviation is not beyond 1.38%. The maximum deviation was reported to be 1.38% for the lubricant containing 0.02 volume fraction of nanoparticles at 40 °C.

Conclusions

In the present study, the dispersion stability of the prepared samples of canola oil-based nanolubricants with added different surfactants and different particle to surfactant mass ratios was investigated on the basis of visual monitoring and experimentally using UV–Vis–NIR spectrometer over a period of 30 days. Moreover, the rheological analysis of canola oil by dispersing TiO_2 nanoparticles of different concentrations for different temperatures and different shear rates was also investigated experimentally and theoretically. On the basis of experimental results, the following key highlights can be pointed out.

1. On the basis of experimental results of stability, the optimum particle to surfactant mass ratios of 1:3, 1:2, 1:4 and 1:2 was observed for TiO_2: Triton X-100, TiO_2:oleic acid, TiO_2:SDS and TiO_2:Tween-20, respectively. However, the maximum absorbance between them was recorded for the TiO_2:Triton X-100 mass ratio of 1:3 and the absorbance was decreased by only 5.74% over the duration of 30 days.
2. The flow behaviour of canola oil and canola oil-based nanolubricants with different volume fractions of TiO_2 nanoparticles was Newtonian at all temperatures under the shear rate ranging from 1 to 1000 s^{-1}.
3. The viscosity of nanolubricants increased with increase of nanoparticles concentration, while it decreased with the rise in temperature.

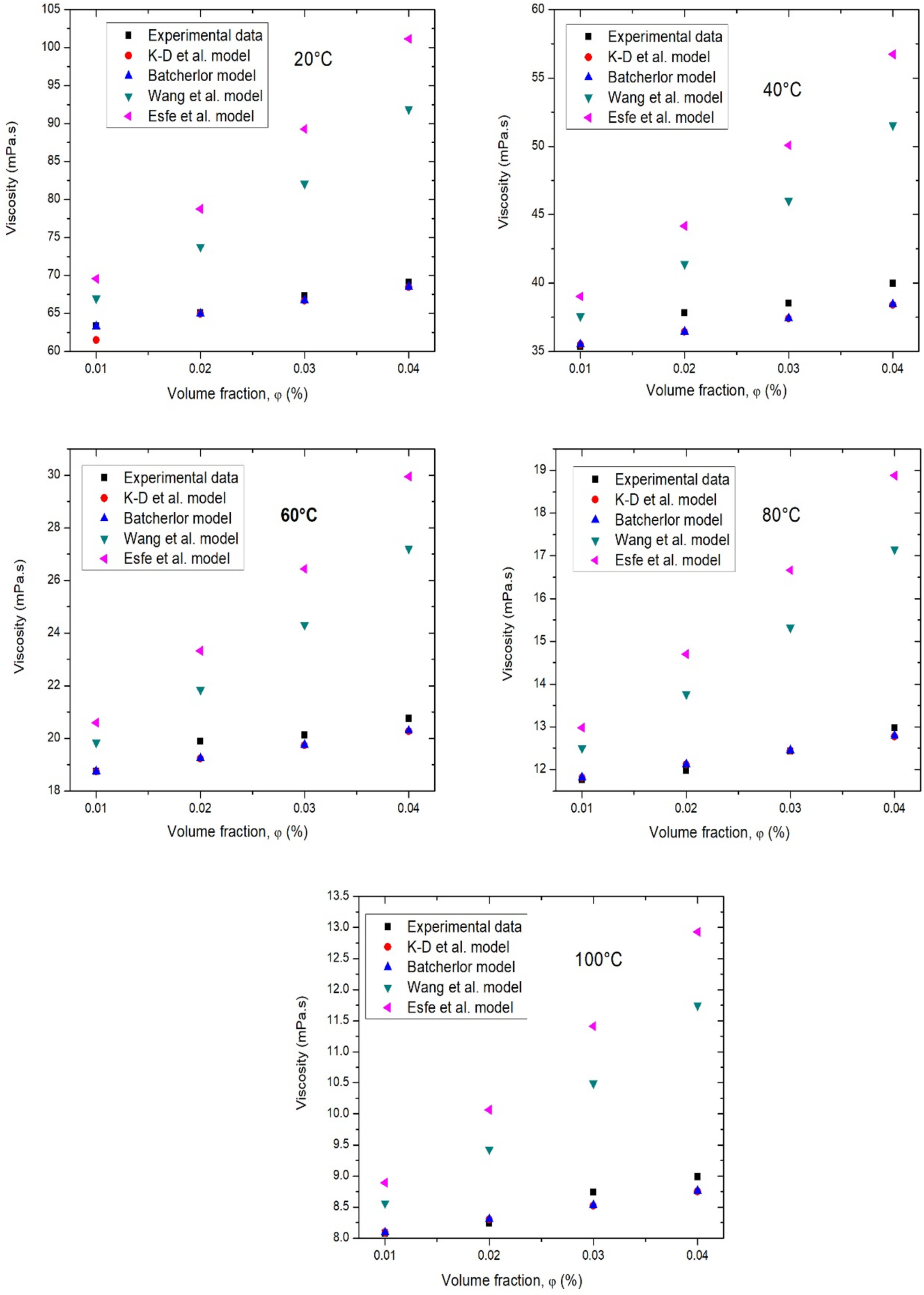


Fig. 16 Comparison of experimental viscosity of canola oil-based nanolubricants with theoretical viscosity models for different temperatures

Table 7 Percentage deviation between theoretical and experimental results

Temperature (°C)	Concentration (φ)	Deviation (%)			
		From K-D et al. model	From Batchelor model	From Wang et al. model	From Esfe et al. model
	0.01	0.08	0.07	− 5.42	− 9.92
20	0.02	0.25	0.20	− 11.74	− 17.32
	0.03	0.99	0.91	− 17.97	− 24.58
	0.04	0.94	0.81	− 24.81	− 31.69
40	0.01	− 0.37	− 0.38	− 5.86	− 9.34
	0.02	3.82	3.78	− 8.59	− 14.37
	0.03	3.05	2.96	− 16.30	− 23.05
	0.04	4.13	4.0	− 22.43	− 29.53
60	0.01	− 0.01	− 0.03	− 5.52	− 9.02
	0.02	3.42	3.38	− 8.95	− 14.70
	0.03	1.98	1.89	− 17.17	− 23.85
	0.04	2.40	2.27	− 23.71	− 30.70
80	0.01	− 0.41	− 0.42	− 5.89	− 9.38
	0.02	− 1.16	− 1.20	− 12.99	− 18.48
	0.03	− 0.08	− 0.16	− 18.84	− 25.39
	0.04	1.60	1.47	− 24.31	− 31.24
	0.01	− 0.11	− 0.12	− 5.61	− 9.10
100	0.02	− 0.76	− 0.80	− 12.64	− 18.16
	0.03	2.58	2.49	− 16.68	− 23.40
	0.04	2.69	2.56	− 23.50	− 30.50

Table 8 Correlation coefficients for the proposed model at different temperatures

Temperature (°C)	a_0	a_1	a_2	R^2
20	0.99	3.12	0.81	0.99
40	0.95	7.811	− 72.21	0.97
60	0.9659	7.011	− 71.39	0.96
80	1.014	− 0.07682	72.7	0.99
100	0.99	2.665	28.2	0.96

4. The viscosity of nanolubricants was higher than that of canola oil for all concentrations and temperatures. However, the influence of nanoparticles on viscosity of canola oil was ignificant at lower temperatures. But, the viscosity of nanolubricants was observed to be near the viscosity of canola oil at higher temperatures.
5. A comparative analysis of experimental data of viscosity with the data obtained from available viscosity models was performed and found that models were not able to predict the viscosity of nanolubricants accurately. Therefore, a regression model was proposed with the maximum margin of deviation of 1.38% for future investigations.

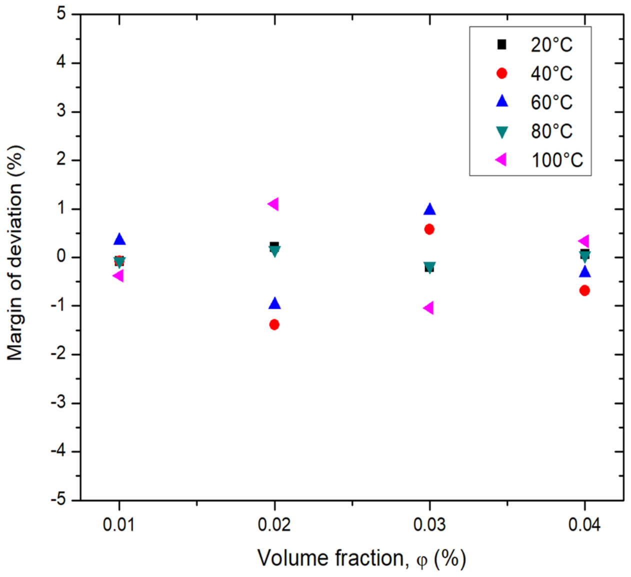


Fig. 17 Margin of deviation of the proposed regression model

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Data availability The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Compliance with ethical standards

Conflict of interest The authors have no competing interests to declare.

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