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Surface Modification of TiO_2-Al_2O_3 Nanoparticles for the Enhancement of the Rheological Properties of Lubricating Oil

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Abstract: Nanoparticles can enhance various properties of lubricants. The surface modification was conducted for inorganic nanoparticles (NPs) TiO_2-Al_2O_3 with oleic acid (OA). The OA renders the TiO_2-Al_2O_3 surface hydrophobic. The OA-treated NPs show very stable dispersion in lubricating oil even after more than one month, compared with the unmodified NPs. The surface modification of NPs was characterized by Fourier transform infrared spectroscopy (FTIR). treated TiO2-Al_2O_3 are added to raw Iraqi base oil SN150 of low viscosity as a hybrid Nanofluid to study the rheological properties such as kinematic viscosity, viscosity index, and the stability of nano-fluid. Four different concentrations were prepared with a range of 0.2% to 1% by weight. Pour point and flashpoint was also identified. The treated TiO_2-Al_2O_3 lubricating oil indicates a significant improvement in the viscosity index by 5.3–9.1 %, while the flashpoint increased by 11-27 °C comparing with base oil.

Keywords: nano-lubricating oil, TiO_2-Al_2O_3 nanoparticles, surface modification, rheology

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1. Introduction

For many decades' researchers have focused on developing base oil additives to overcome friction, oxidation, corrosion, and abrasion (Al-Janabi & Hussin, 2020). Lubricants have important functions that include lubricating, cooling, and protecting metal surfaces against corrosion harm. Lubricants consists of base oil and an additive package; the base fluid's primary function is to lubricate and act as a carrier of additives. Lubricants additives are added to base oil with few percentage weights to impart specific properties to the finished oils (Deepika, 2020; Shahnazar et al., 2016). For example, zinc dialkyldithiophosphate is used as an antioxidant to enhance oxidation and degradation stability. Phosphorus and Sulphur are used to improve extreme pressure and anti-wear property (Kaviyarasu & Vasanthan, 2015). Nowadays, additives play a major role in improving the tribological and rheological properties. Nano lubricants often describe the lubricants in which the NPs are dispersed by using the appropriate dispersion techniques. Metal oxide nanoparticles with sizes 10-100 nm are among the lubricating additives used as an effective lubricating for mechanical application due to high diffusion point, reduced melting, and easy sintering. Furthermore, the main advantage of NPs is that they are insensitive to temperature and that tribo-chemical reaction are limited, compared to regular additives (Abdullah et al., 2014; Ali & Xianjun, 2020; Kamel et al., 2020). Researchers have recently used different nanoparticles of metal oxides as additives, which can be used as an efficient lubricant improver for the rheological and tribology properties. Kamel et al. (2020) investigated the influence of hybrid nano additives of CNTs/GNs on the tribological and rheological characterization of oil 15W-50. They found that the CNTs/GNs with concentration (1.5 % CNTs – 0.5 % GNs) decreased the wear and friction coefficient of oil. The same concentration of NPs enhanced the flashpoint and pour point, also increased thermal conductivity. Abdullah et al. (2014) reported that addition of hBN/Al_2O_3 nanoparticles to SAE 15W-40 diesel engine oil could enhance the viscosity index, flash point, and TBN of engine oil.

Alves et al. (2016) investigated the tribological properties by different additive concentrations (0.1 wt%, 0.25 wt%, and 0.5 wt %.) of CuO NPs diffused in synthetic oil PAO. The results showed that the 0.1 wt % CuO gave a better anti-wear performance. Concentrations of 0.25% and 0.5 % demonstrated the same friction-reduction activity. However, small CuO nanoparticles are more efficient in reducing wear than in friction reduction. Taha-Tijerina et al. (2018) worked on enhancing extreme pressure EP of synthetic fluid by using ZnO nanoparticles with a size of less than 50 nm. The result shows a greater improvement by 0.1wt % of ZnO. Cortes and Ortega (2019) studied the impact of SiO_2 /CuO on the rheological and tribological properties of coconut oil as Nanofluid. The viscosity of fluid increases with an increase in the concentration of SiO_2. However, viscosity decreased by increasing CuO in coconut base oil. On the other hand, the addition of SiO_2/CuO enhances the friction reduction of Nanofluid compared with base oil.

(Hemmat Esfe et al., 2019) in their investigation, used 30% MWCNT–70%TiO_2/SAE50 as a hybrid nano lubricant. They observed that Nanofluid has a non-Newtonian behavior of shear stress versus shear rate, and the best sliding of fluid layers reducing the friction of the layers was achieved by the addition of MWCNT/TiO_2 to SAE50. Ali and Xianjun (2020), investigated the efficiency of Al_2O_3/TiO_2 hybrid nanomaterials' efficiency as nano lubricants to increase the heat transfer performance. They noticed that oxidation temperature and the burning temperature of Al_2O_3/TiO_2 hybrid NPs could be increased by 54.9 °C and 38.7 °C, respectively. This indicated that the thermal stability of Al_2O_3/TiO_2 NPs permits a higher temperature range for oil (5W-30). The dispersion of inorganic nanoparticles in lubricating oils is still the main problem for applying nanoparticle additives where Nanoparticles in lubricating oil tend to adhere together and form larger agglomerates, and thus it will be precipitated down due to gravity. Because of Van der Waal's forces and Brownian motion, the particles attract each other and agglomerate when attractive forces are larger than repulsive forces (Ahmed Ali et al., 2019; Kango et al., 2013; Kumar & Arasu, 2016). There have been significant efforts to improve nano lubricant stability via the physical ways by using surfactants and surface modification of nanoparticles to solve the problem. Some studies investigated nanoparticles' surface modification with different surfactants or silane coupling agents to enhance inorganic particles' stability in oil base suspension. Modifying agents are usually organic materials consisting of polar groups and long alkyl chains that adsorbed onto inorganic nanoparticles and helped inorganic particles soluble in the organic solvent (Azman & Samion, 2019).

Hong et al. (2009) carried out a surface modification of ZnO nanoparticles by grafting polymethyl methacrylate (PMMA) onto the particles through a free radical polymerization method. Soleimani et al. (2017) used two modifiers such as oleic acid (OA), trimethoxyvinylsilane (TMVS), to modify the surface of alumina NPs and changed them to hydrophobic. The results showed that modified Al_2O_3 NPs with oleic acid (OA) a good dispersion in MMA and BuA monomers. Zhang et al. (2011) enhanced the stability of TiO_2 nanoparticles in liquid paraffin by capped its surfaces with stearic acid (SA). The stability of the TiO_2 anatase coated in liquid paraffin has been improved without any signs of aggregation. The SA was successfully ligand on the surface of the TiO_2 nanoparticle anatase by Fourier transform infrared spectroscope (FTIR) analysis. Luo et al. (2014) found the KH-560 agent modified the good dispersed Al_2O_3 NPs. The zeta potential value shows the static repulsion of the treated Al_2O_3 is greater than that of the as-prepared nanoparticles. The agglomerate of Al_2O_3 nanoparticles is successfully stopped, and the surface of Al_2O_3 nanoparticles changes from hydrophilic to hydrophobic after surface modification.

Hong et al. (2009)(γ methacryloxypropyltrimethoxysilan) was used plasticizers as a surfactant to surface modification of ZnO NPs to increase dispersion organic media.

This study's key features are handling the issue of oil-solubility by the combined effect of surface modification of NPs and mixing methods of lubricating oil. It can be useful for using modified NPs TiO_2 and Al_2O_3 as hybrid nanoparticles as additives in the base oil SN150. The current work investigation focuses on enhancing lubricating base oil properties (SN150) such as viscosity index, flash point, and pour point.

2. Materials and methods

2.1. Materials

Titanium (IV) oxide TiO_2 and gamma-alumina γAl_2O_3 from Hongwu International Group Ltd. (Guangdong, China). Oleic acid, required for the surface coating of the TiO_2-Al_2O_3 nanoparticles, was purchased from (Alpha Chemika, India).

The base oil was attended by (Al-Dura Refinery, Middle refineries Company, Baghdad, Iraq) and met viscometric requirements for SN150 The specifications of nanoparticles are shown in Table 1. The parent oil properties are presented in Table 2.

Table 1. Specifications of nanoparticles.

Specification	TiO_2	γAl_2O_3
Average diameter(nm)	71.14	61.91
Color	White	White
Purity (%)	>99.9	>99.9
surface area (m2/g)	119.9	245.11
Pore volume (cm^3/g)	0.237	0.1606

Table 2. Properties of base oil SN150.

Specification	Base oil(SN150)
Kinematic viscosity @ 40°C (cSt)	15.493
Kinematic viscosity @ 100° C (cSt)	3.4706
Viscosity index	98.7
Density @15(̊C) (g/cm^3)	0.8551
Flash Point (̊C)	188
Pour point (̊C)	-12

2.2. Surface modification of NPs

TiO_2 and Al_2O_3 nanoparticles were separately added into 100 mL of anhydrous ethanol solution containing 1 g of oleic acid (OA). The resultant sample will be heated at 75°C for two hours needed to complete the reaction using a magnetic stirrer. The sample is put in an oven furnace for four hours until ethanol evaporates completely. Then, the residue is washed with methanol to remove unreacted OA. The sample is dried at 80 °C for six hours to yield OA surface-modified TiO_2 nanoparticles (denoted as OA/TiO_2) and OA surface-modified Al_2O_3 nanoparticles (marked as OA/Al_2O_3).

2.3. Preparation of Nano-lubricating oil

The modified NPs TiO_2 and Al_2O_3 were added at a weight ratio of 50:50 to base oil. The total NPs concentrations of 0.2, 0.5, 0.8, or1 % wt. in the base oil were reached. The required amount of modified TiO_2-Al_2O_3 was weighed carefully using an accurate electronic balance and mixed with the base oil using a magnetic stirrer for 2 h (Asadauskas et al., 2015), followed by exposing to ultrasonic waves for 30 minutes in Sonicator type VCX 750 (Sonics & Materials Inc., USA) operating at 20 kHz and 750 W maximum, fitted out with a 13 mm diameter Ti-6Al-4V alloy tip. Sonication was carried out of each pulsed irradiation, alternating 5 s of stagnation (not sonicated) and 5 s of sonication at 60% power. Usage of Sonicator aims to achieve a more uniform dispersion and break down the agglomeration of nanoparticles.

2.4. Characterization of Nano-Lubricant

Fourier transform infrared spectroscopy (FTIR) (type Shimadzu, Japan) was used to characterize OA/TiO_2 and OA/Al_2O_3 NPs using KBr tablets with the scanning area 500 – 4000 cm^{-1} and the resolution of 1 cm^{-1} for each spectrum. The nano-lubricating oil was examined in terms of kinematic viscosity, Viscosity Index (VI), flashpoint and pour point. The VI was calculated from kinematic viscosity at 40 °C and 100 °C using a viscometer (Anton Paar) according to ASTM D-7042. Flashpoint and the pour point of base and nano-oil were measured according to ASTM D-92 and ASTM D-97 respectively. The stability formulations were also observed by measuring the absorption over time using UV spectrometer in which the conventional base oil was set as a background.

3. Results and discussion

3.1. Characterization of OA-TiO_2 and Al_2O_3 nanoparticle

FTIR analysis was used to analyze OA/Al_2O_3 and OA/TiO_2 nanoparticles' chemical characterization, as seen in Figure 1 and Figure 2, respectively. in Figure 1a the observed band at 789 cm^{-1} of FT-IR of NPs is related to the stretching frequency of Al-O in alumina. The adsorption peaks at around 3471 and 1637 cm^{-1} are recognized to the stretching of O-H groups on alumina's surface (Soleimani et al., 2017). In Figure 2a, the adsorption peaks in 1635 and 1437 cm^{-1} are dedicated to Ti–OH and Ti-O groups in TiO_2 nanoparticles, respectively (Parvizian et al., 2020). In Figure 2b, the adsorption peaks of 2924.09 and 2852.72 cm^{-1} are attributed to OA's long alkyl groups. Furthermore, carbonyl groups of oleic acid in OA/TiO_2 were characterized in the peaks of 1710.86 cm^{-1} (Hu et al., 2002). The peaks in 3423.65 cm^{-1} and 3471 cm^{-1} in Figure 1a and Figure 2a are associated with −OH groups on the surface of Al_2O_3 and TiO_2 NPs decreased in OA/TiO_2 and OA/Al_2O_3 nanoparticles. Meanwhile, OA groups of carboxyl (COOH) reacted with hydroxyl (OH) groups to produce carboxylate in TiO_2 and Al_2O_3 nanoparticles. OA functional groups' presence on the surface of NPs in OA/TiO_2 and OA/Al_2O_3 nanoparticles confirmed these groups' adsorption.

3.2. Stability of nano-lubricating oil

The stability of prepared nano-lubricating oil can be investigated using UV spectrum, UV-9200 (Biotech engineering, management Co. LTD, UK). Figure 3 shows that the TiO_2-Al_2O_3/lubricating oil absorbency at the wavelength of (λmax) of 600 nm was observed after preparation. From Figure 4, the nano-lubricating oil shows good stability over a period time of 30 days at room temperature. However, the stability of nano-lubricating oil with unmodified TiO_2-Al_2O_3 nanoparticles decreased sharply with the 22% reduction in absorbency value at 30 days. The aggregation resulted from the Brownian motion when the attractive forces (Van der Wall) of the nanoparticles were more significant than the repulsive forces. Figure 5 shows the samples were kept in glass containers under room temperature for about more than 30 days.

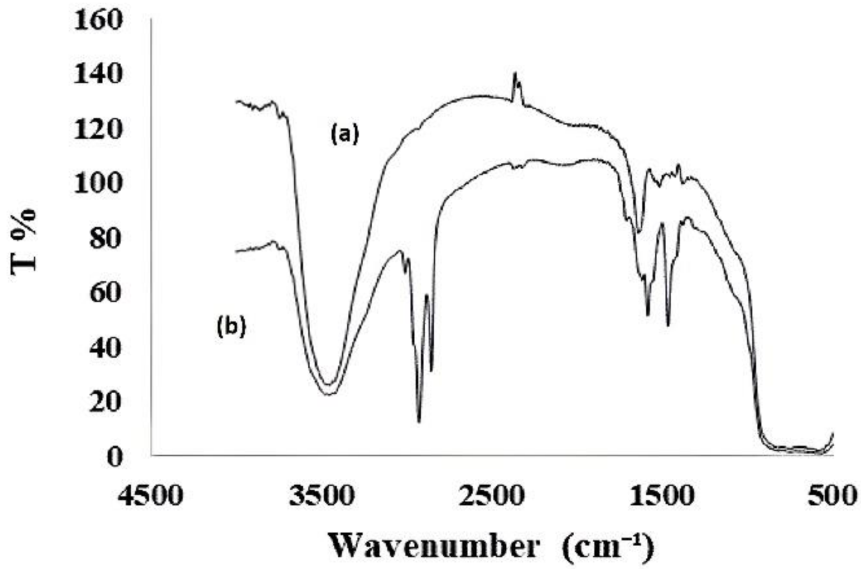

Figure 1. FTIR spectrum of Al_2O_3 (a) and OA/Al_2O_3 (b).

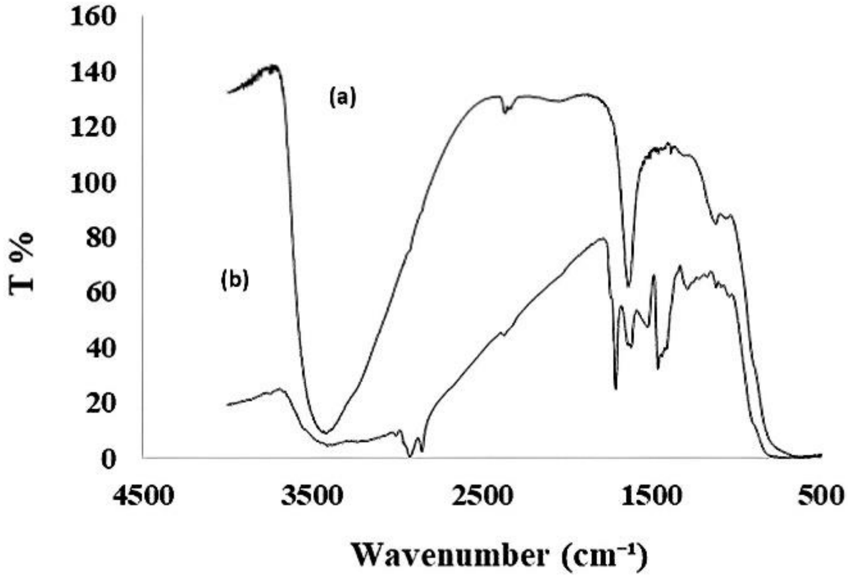

Figure 2. FTIR spectrum of TiO_2 (a) and OA/TiO_2 (b).

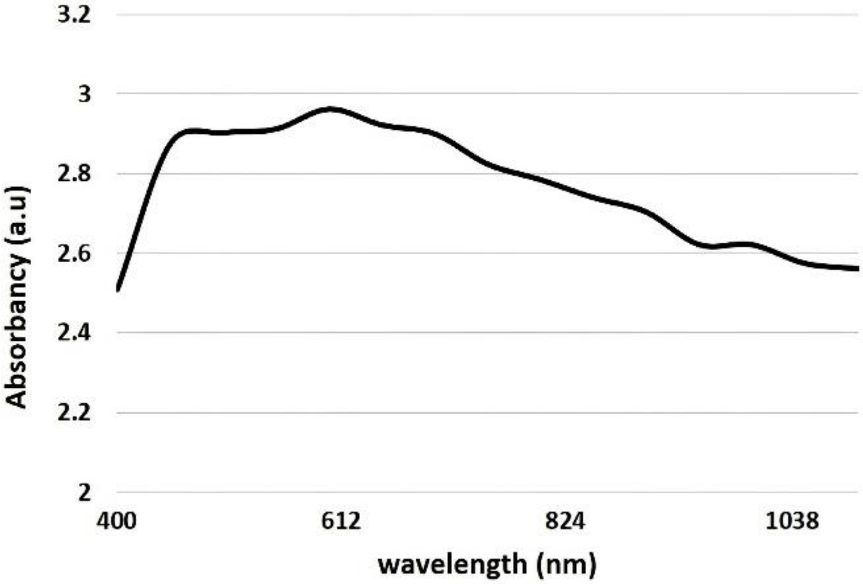

Figure 3. UV for TiO_2-Al_2O_3/lubricating base oil after preparation.

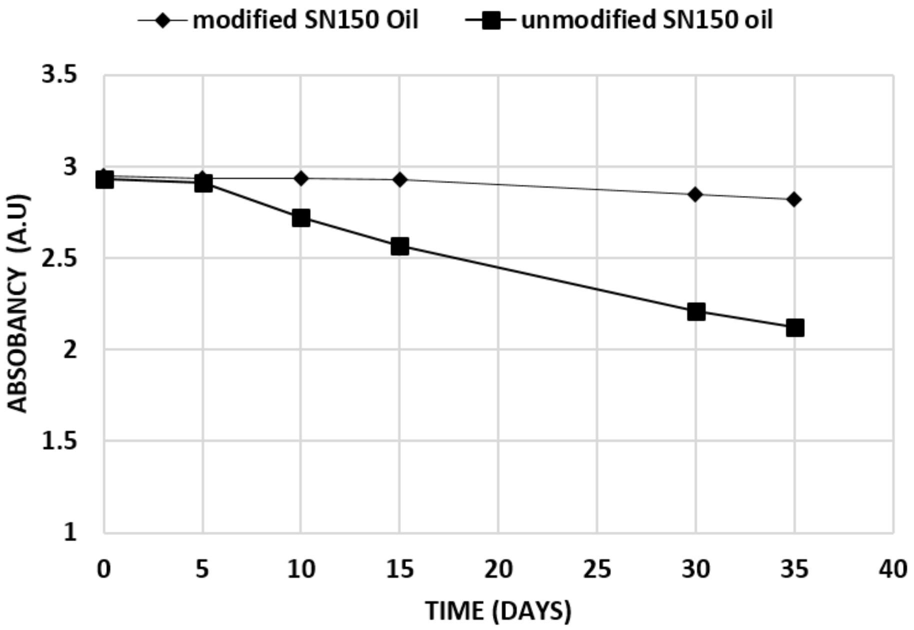

Figure 4. The absorbency curve of modified nano-oil and unmodified nano-oil as a function of time.

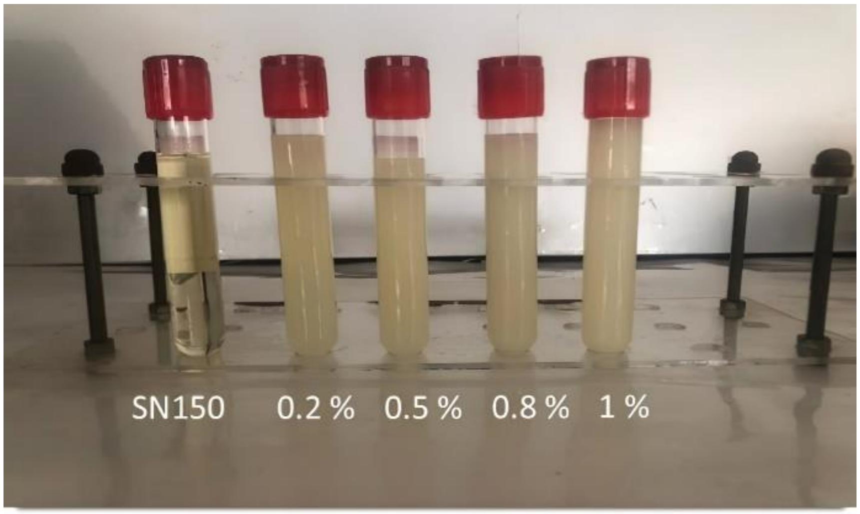

Figure 5. Photograph of prepared modified TiO_2 Al_2O_3/SN150 oi l with different concentrations after 30 days.

3.3. Effect of TiO_2-Al_2O_3 nano-lubricating oil on kinematic viscosity and viscosity index

Viscosity is one of the essential properties of any lubricant. It determines the film formation tendency of the lubricant. The kinematic viscosity for nano-lubricating oil existed in the current study compared with base oil at 40 °C and 100 °C temperatures using different concentrations (0.2-1 wt.%) of NPs. Figure 6 shows that all lubricant oil samples' viscosity decreases significantly from 40°C to 100°C temperature, respective Viscosity increased slightly on increasing modified TiO_2-Al_2O_3 nanoparticle concentration at both temperatures. The rate of viscosity variations was much lower at a lower concentration than that of the higher concentration. At high concentrations, the presence of larger, spherical particles within oil layers, which prevents movement, might explain increased viscosity (Ettefaghi et al., 2013). Moreover, another important parameter in the properties determination of lubricants derived from kinematic viscosity at 40 °C and 100 °C is the viscosity index (VI). Figure 7 shows the viscosity index increased with nanoparticles' addition to base oil by 9 % with modified nanoparticles' addition at 1% wt. compared with the base oil without NPs additives. The higher viscosity index indicates more stable kinematic viscosity with varying temperatures.

3.4. Flashpoint and pour point

The flashpoint can be described as the lowest temperature at which a flammable mixture in the air can be formed. In Figure 8 can see the addition of modified TiO_2 and Al_2O_3 as hybrid NPs enhancement the flashpoint of base oil. Also, the flashpoint directly relates to the concentration of nanoparticles, but this relationship is not linear, and the changes at lower concentrations are more significant than changes in higher concentrations. The increase in the value of flashpoint at 0.2 wt. % with respect to the base oil is 11°C, and the highest value is related to 1 % of NPs, which was 27 °C. This improvement is attributed to an increase in nano-lubricating oil's thermal conductivity, and it can be said that it allows the oil to better resist ignition.

The pourpoint is the lowest temperature at which the lubricating oils can flow easily. The highest wear occurs at start-up due to oil not reaching all engine parts. To solve this issue, it is necessary for the oil to be pumped and to be able to flow freely and with high speed enter all parts of the engine. The trend changes in pour point as a function of the modified TiO_2-Al_2O_3 NPs concentrations are shown in Figure 9. The results show decreasing pour point from -12 °C to -15 °C with all concentrations of modified NPs

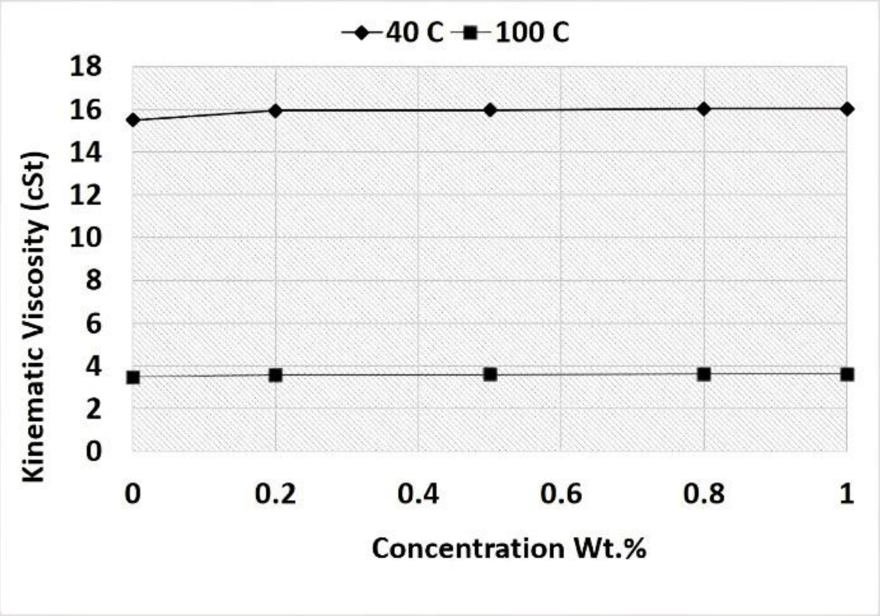

Figure 6. Kinematic viscosity of nano-lubricating oil at 40 °C and 100 °C.

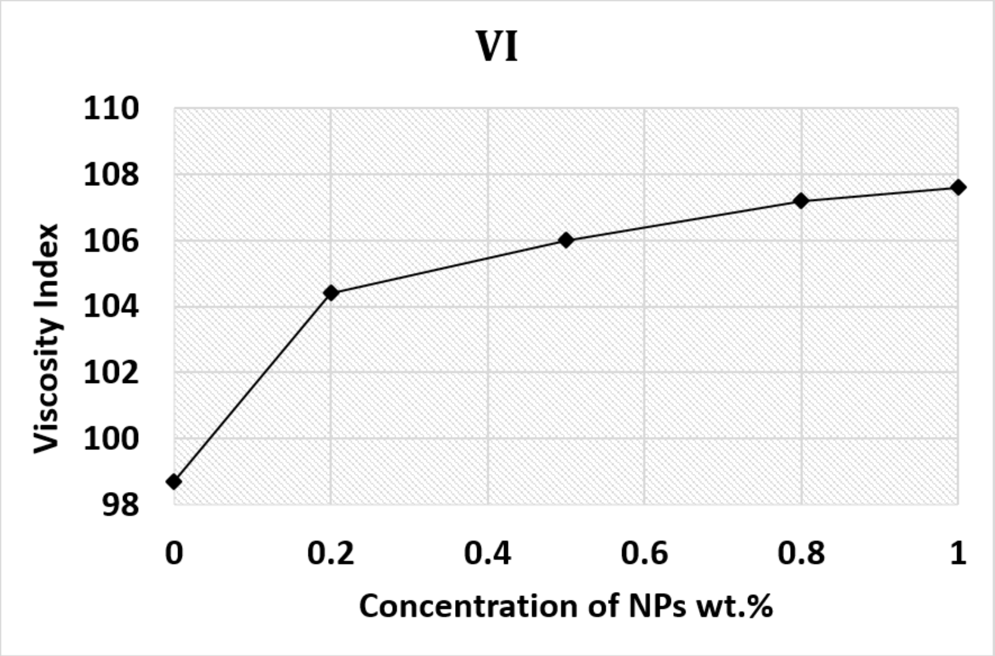

Figure 7. Viscosity index of nano-lubricating oil for different concentrations of nanoparticles.

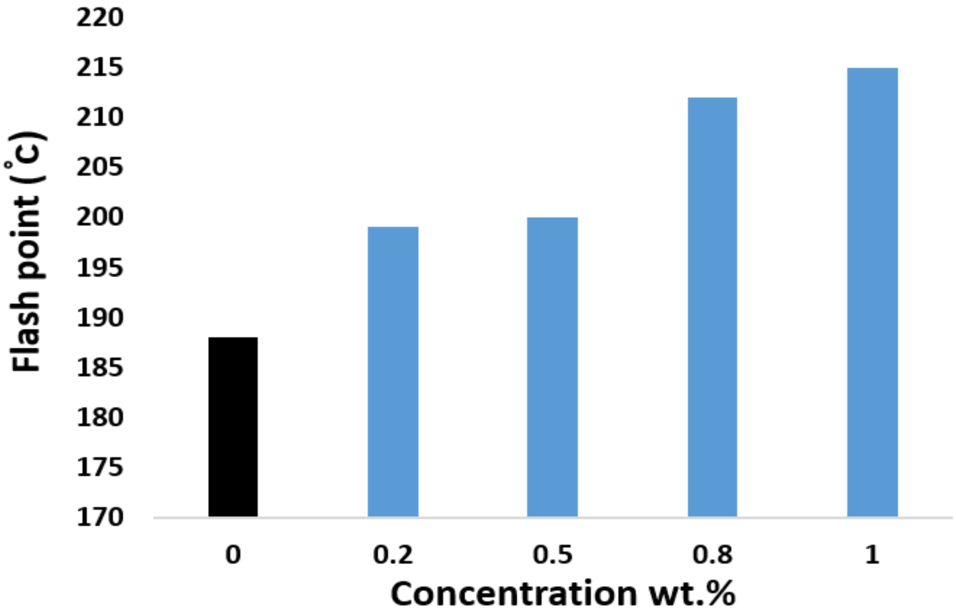

Figure 8. Effect of nano additive modified TiO_2-Al_2O_3 wt. % on flash point of SN150.

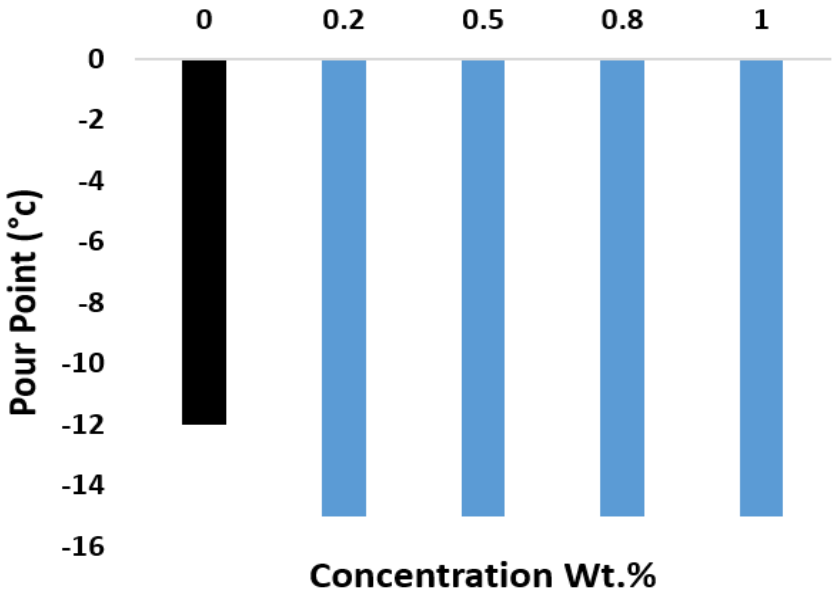

Figure 9. Effect of modified nano additive TiO_2-Al_2O_3 wt. % on pour point of SN150.

4. Conclusions

- In the current study, the surface of TiO_2 and Al_2O_3 nanoparticles has been modified using the oleic acid surfactant to achieve a stable colloidal and homogeneous dispersion in the base oil SN150.
- The modified NPs were characterized by analytical techniques such as FTIR. The UV-visible method was used to test the dispersion stability of NPs.
- The kinematic viscosity of modified nano-lubricating oil at 40°C and 100 °C increased slightly with the addition of modified nanoparticles.
- With 0.2 wt. % of modified TiO_2- Al_2O_3 nano-lubricating oi, the viscosity index increased by 5.3%, and increase was 9.1 % at 1 % concentration.
- The results show that the pour point decreased from -12°C to -15°C when adding 0.2 wt. % of modified NPs to base oil. Also, no improvements in the pour point have been found by adding more than 0.2 wt. % of the modified NPs.
- Adding TiO_2- Al_2O_3 nanoparticles to SN150 oil at varying concentrations improved nano-lubricant oil's flash point compared to parent oil. The flashpoint of nano-lubricants at 1 wt. % increased from 188°C to 213°C
- We conclude that the nano-lubricant produced with a concentration of 0.8 wt. % modified TiO_2-Al_2O_3/ SN150 oil seems to be the optimum because of this sample's kinematic viscosity doesn't increase more, which is required, and other oil parameters have been improved.

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