# Nano zinc oxide additive for the enhancement of lubricant properties

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**Abstract:** Zinc oxide (ZnO) nano additives were successfully synthesized in laboratory by precipitation technique. The XRD showed crystalline nature of ZnO with the average crystallite size of 16 nm. The stretching band of ZnO was found at around 400 $cm^{-1}$ to 600 $cm^{-1}$ wave number in FTIR. The prepared nano particle have been used as nano additive and sodium lauryl sulphate (SLS) as surfactant, in base oil to improve physio-chemical parameter of lubricants. The result revealed that the additive blended base oil (lubricant) has shown excellent lubrication properties. The higher kinematic viscosity of 90.72 and 10.40 were obtained at 40°C and 100° C respectively. Similarly, viscosity index were found to be 96 which was improved indicating the use of lubricant in slightly high temperature is possible. The pour point was found to be decreased to -9.2°C which was quite significant and could be used in cold environment. The flash point was also found to be increased from 225° C to 230°C which indicated that the prepared nano additive ZnO acts as flash point enhancer. The corrosion test done by copper strip comparative method and was found to be 1b for additive indicating the non-corrosive nature. The absence of moisture and $p^H$ around the neutral range 6.65 showed that the additive blended lubricant is not harmful for machinery devices.

**Keywords**: ZnO; Additive lubricant; Kinematic viscosity; Viscosity index; Flash point; Pour point.

**Introduction**

Highly viscous lubricants are commonly applied in every single engine to operate it effortlessly. It enhances the life time of engine by preventing sliding surfaces of engine from friction, wear, tear and corrosion. However, use of lubricant could not control anti wear (AW) in extreme pressure (EP) properties. The lubricant along with some additives plays an outstanding role not only to avoid engine deterioration but also enhances the lubricant properties like kinematic viscosity, viscosity index, flash point, pour point as well. These properties depend mainly upon the composition of base oil in lubricant as well as possessions of additives used. The base oils are a mixture of hydrocarbon, including alkanes (paraffins), alkenes (olefins), alicyclic (naphthenes), aromatic and some mixed hydrocarbons. Base oil occupies more than 90 % by weight of total lubricants formulation and rest part may be some additives[1]. Different base oils have been used to prepare different types of lubricants. These oils are symbolized as SN and BS. The SN symbolises for solvent neutral whereas BS denotes for bright stock. Solvent neutral are mainly used to manufacture engine oil and are entitled as: SN-100, SN-150, SN-350, SN-500, SN-650 whereas bright stock are used in manufacture of gear oil and are named as; BS-150, BS-300, BS-500 and so on[2]. As described earlier, only simple base oil cannot prevent anti wear in high pressure, hence it couldn't fulfil the current demand of heavy

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machinery devices in the lubricating field. So, chemical additives are added to the base oil in order to meet the quality requirements for the final products in terms of friction wear, tear, life span and efficiency[3]. The key functions of additives are: antioxidants, viscosity modifiers, pour point depressants, detergents, dispersants, and antifoam agents, anti wears, friction modifiers, and antirust. Molybdenum disulphide, phosphorus, sulphur, and nitrogen, zinc, and chlorine compounds are examples of additives that have been formulated and added to the base mineral oil to increase their anti-wear (AW) in extreme pressure (EP) properties[4].

In recent years, the macro sized additives have been replaced by nano-sized additives like Cu, CuO, ZnO, $TiO_2$, $CeO_2$, $Al_2O_3$, $MoS_2$, nano graphite, nano diamond, carbon nanotube, ZrO, $LaF_3$, $CaCO_3$[5-21].These nanoparticles act as third body which can act as nano-bearings on the sliding surfaces of engine where it deposits and improves the tribological properties of base oils. The major advantage of using nano-sized additives over macro-sized are: extreme pressure resistance performance, excellent anti-wear resistance and better lubrication properties[4].

**The mechanism of additives in lubricants**

In between the sliding surfaces of engine, there exist numerous peaks and valleys (cracks and crevices).These leads to friction, wear and tear on the rubbing surfaces. When nano additives incorporated with lubricants are kept in between these rubbing surfaces, due to mutual rubbing additive molecules and base oil starts to interact with each other and there is a formation of tribofilm as shown in Figure 1. Consequently, this film creates smooth layer during the sliding process and reduces friction wear and tear which significantly extends life span and efficiency of machinery devices.

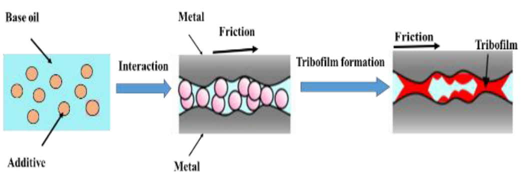


**Figure 1: Mechanism of formation of tribofilm**

In fact, bare inorganic nano additive in liquid media like lubricant is thermodynamically unstable with respect to growth and it tend to spontaneous coalescence as a consequence of their high surface energy. Therefore, surface of nanoparticle is controlled by suitable capping agents or surfactants such as polyethylene glycol, lauryl sodium sulphate, triton X-100. The polar head of the surfactant surrounds the nanoparticles which prevent the spontaneous coalescence and non-polar tail lies outwards which are responsible for easy and smooth dispersion in oil. It is clearly shown in the schematic diagram (Figure 2).Then it provides stabilization of nano-sized objects in viscous liquid or semi-liquid and corrosion inhibitors for several decades.

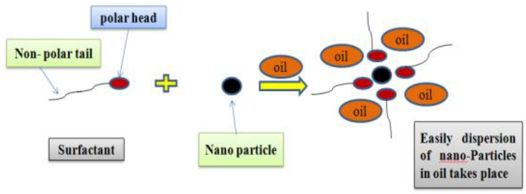


**Figure 2: Schematic diagram of working mechanism of surfactant**

In the year of 2014, synthetic macro additives are normally mixed with engine oil[4]. In recent years, the macro sized additives have been replaced by nano-sized additives which have been reported in literatures. Some of them are nano sized Cu, CuO, $TiO_2$, $CeO_2$, $Al_2O_3$, SiC, nano-diomond graphite, molybdenum disulphide etc [5-23]. In recent years, nano zinc oxide has found wide ranging applications in various areas due to its unique and superior physical and chemical properties like large surface area, high surface energy, strong adsorption, high diffusion, easy sintering, low melting point and other outstanding characteristics[24]. The zinc oxide nanoparticles were generally prepared by different methods such as sol-gel method, precipitation method and microwave irradiation technique [25,26]. A research reported on the synthesis of ZnO by precipitation method and studied the anti-wear properties, coefficient of friction and wear scar diameter of ZnO nanoparticles blended in base oil (SN-500) at different concentration (0.5,0.1and 2.0% by wt)[5]. It was found that the 1.0% by wt of ZnO nanoparticles suspension in lubricant exhibited the better tribological behavior. However, the effect of zinc oxide on the physio-chemical properties of the lubricant has not been studied. Hence, in present study, we try to synthesize ZnO nanoparticles by precipitation method. The prepared materials were characterized by different techniques like XRD, FT-IR, and SEM and was blended with base oil (lubricant) as additive. Further, physio-chemical properties of additive blended lubricant was investigated and compared with base oil (SN-500).

## Materials and methods

Analytical grade (AR) zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$) and (LR) grade sodium lauryl sulphate ($C_{21}H_{25}O_4SNa$) was obtained from SD fine-chemical limited, India. Pure urea ($NH_2CONH_2$) was obtained from Hi Media Laboratories Pvt. Ltd, India. (LR) grade sodium hydroxide was obtained from Thermo Fisher Scientific Pvt. Ltd, India. Dehydrated ethyl alcohol was obtained from Bengal Chemicals and Pharmaceuticals Ltd., India. Laboratory grade base oil (SN 500) was obtained from Sun Oil Company Pvt. Ltd, India.

**Synthesis of zinc oxide nanoparticles:** The homogeneous mixture of zinc nitrate and urea solution was obtained by mixing 100 mL of each solution in a flask. Then, it was heated maintaining the temperature of $95^0C$. The surfactant laureal sulphate was also added to it and heating was continued for 3 hours. A white precipitate were obtained which was filtered and washed several times with water and finally with ethanol. Thus obtained precipitate was then calcined at $400^0C$ for 3 hours.

**Characterization of material:** The crystal structure and average crystalline size of as prepared material was measured by Bruker D8 advance x-ray Diffractometer using monochromatized CuKα (λ=1.54060°A). The crystallite size was calculated by Debye Scherer's equation. SHIMADZU Fourier Transform Infrared Spectrometer was used to observe the functionality of the prepared material. The surface morphology was determined by SEM (KEYENCE REAL 3D System, VE-series, Japan).

**Physiochemical characterization of additive blended lubricant**: Kinematic viscometer (STANHOPE SETA LIMITED) has been used to investigate viscosity. To investigate flash point, cleveland open cup has been employed. In the same way, digital refrigerator (EIE instrument PVT.LTD) was applied to study the pour point of oil. Constant temperature bath (EIE Instrument PVT.LTD) has been used to test the corrosion. Test methods employed for the study of physio-chemical parameters are shown in Table 1.

**Table 1**. **Test methods employed for the study of physio-chemical parameters.**

| S.N | Test Parameter | ASTM Method |
|---|---|---|
| 1 | Kinematic Viscosity at 40°C(cSt) | D 445 |
| 2 | Kinematic Viscosity at 100°C(cSt) | D 445 |
| 3 | Viscosity Index | D 2270 |
| 4 | Pour Point (°C) | D 97 |
| 5 | Flash Point (°C) | D 92 |
| 6 | Density at 30 °C (g/mL) | D 1298 |
| 7 | Emulsion test at 54 °C | D 1401 |
| 8 | Corrosion test (copper strip test) at 50 °C | D 130 |
| 9 | Moisture content test (crackle test) | – |
| 10 | pH | – |

## Results and discussion

**X-ray analysis:** The XRD pattern of as prepared material is shown in Figure 3. In Figure 3, the sharp diffraction peaks could be seen clearly at 21.7, 25.2, 32.2, 33.5, 35.0, 36.9, 48.1, 57.0, 59.0,63.3,68.4 and 69.4 at $2\theta^{\circ}$ which are assigned for zincite structure of ZnO according to JCPDS card No. 36-1451. The sharp diffraction peaks are also indication of the crystallinity of the materials.

The crystalline size of the material was also calculated from the most intense peak using Debye-Scherer's equation and was found to be 16 nm. The XRD pattern and crystallite size are quite comparable with reported value[27].

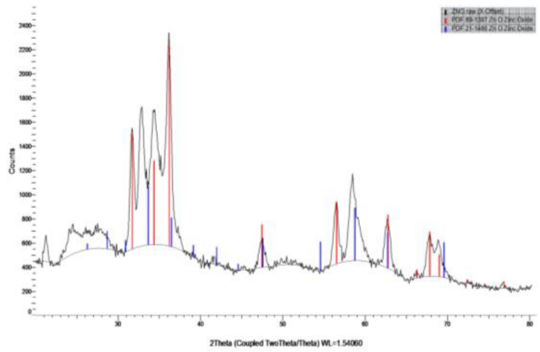

**Figure 3**: **XRD pattern of as prepared material**

**Fourier Transform Infrared Spectroscopy (FT-IR):** The FTIR spectrum of as prepared nano ZnO material was obtained and is shown in Figure 4. The weak and broad bands at around 3250- 3550 $cm^{-1}$ was observed which indicates the stretching band of hydroxyl groups. Similarly, weak vibration band at 1625 $cm^{-1}$ could be seen which indicates the presence of little water absorbed on the surface of metal oxide. A peak around the 1112 $cm^{-1}$ was obvious which revealed the stretching band of C-H vibration. The stretching band of ZnO could be seen at around 400 $cm^{-1}$ to 600 $cm^{-1}$ which is in good agreement with literature value [26]. Here, a sharp peak at 587 $cm^{-1}$could be clearly seen which was assigned for ZnO peak.

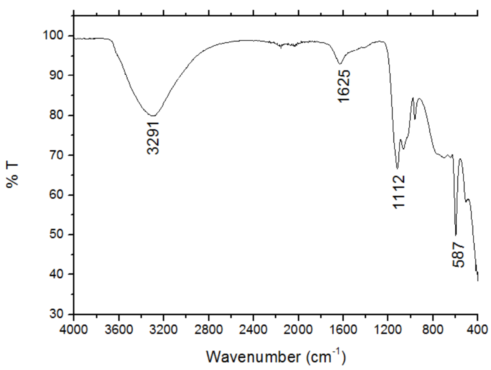


**Figure 4: FT-IR spectrum of as prepared ZnO nano material**

**Scanning Electron Microscopy (SEM):** The surface morphologies of as prepared ZnO was studied by SEM. Figure 5 shows the SEM image of ZnO at 10 µm. In SEM image, one can observe the agglomerated type of ZnO nano materials. Some are smaller agglomerates and some larger agglomerates. It was also observed that the surface is rough, irregular and narrow flake like structure.

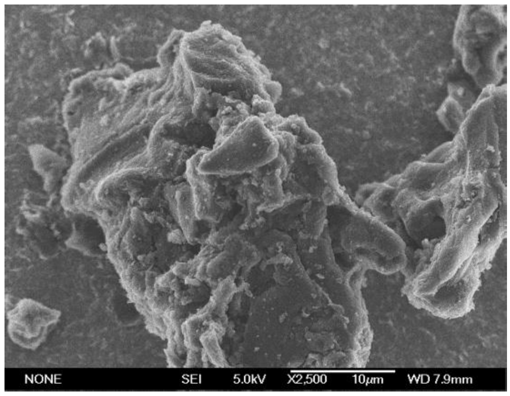


**Figure 5**: **SEM image of as prepared nano ZnO materil at 10 µm magnification**

**Physio-chemical parameters of base oil (SN 500) and additive blended lubricant**

**Kinematic viscosity:** The kinematic viscosity of bare base oil (SN-500) was obtained at 40 ˚C and 100 ˚C. Similarly, additive blended base oil (SN-500 + 1% ZnO) at 40 ˚C and 100 ˚C were also noted and are given in the Table 2. The results were then compared and are presented in the bar graph (Figure 6**).** From the bar diagram**,** it can be perceived that in both cases, the kinematic viscosity was found to be decreased with increase in temperature to 100˚C. This might be due to the weakening of inter-particle and intermolecular adhesion forces. Furthermore, it can also be seen that the kinematic viscosity was found to be slightly higher in case of additive blended base oil than that of bare base oil. This may be due to the internal shear stress of the lubricant rises after addition of nano ZnO additives.

**Table 2. Kinematic viscosity of base oil (SN-500) and additive blended base oil (SN-500 + 1% ZnO) at 40 °C and 100 °C.**

| S.N. | Sample | K.V. at 40°C (cSt) | K.V. at 100°C (cSt) |
|---|---|---|---|
| 1 | Base oil (SN-500) | 90.47 | 10.19 |
| 2 | Additive blended base oil (SN-500 + 1% ZnO) | 90.72 | 10.40 |

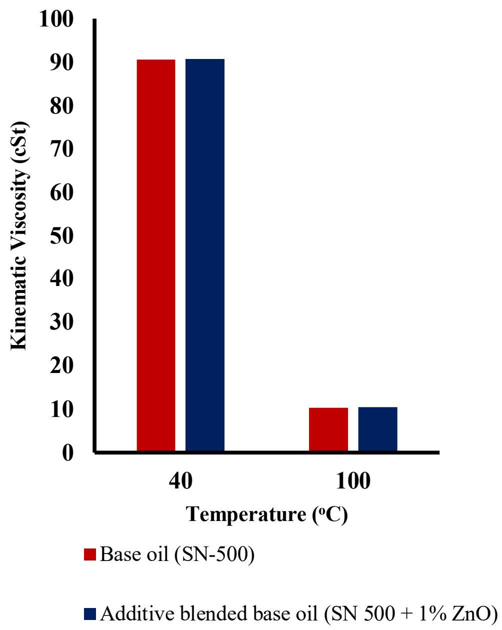


**Figure 6: A bar diagram showing the comparison of kinematic viscosity of base oil (SN-500) and additive blended base oil (SN-500 + 1% ZnO) at 40 °C and 100 °C.**

**Viscocity index**: The viscosity index of bare base oil (SN-500) and additive blended base oil (SN 500 + 1% ZnO) are determined on the basis of ASTM viscosity index table[27] and was found to be 92 and 96 respectively which was presented in bar graph (Figure 7). Figure 7 clearly displayed the increase in viscosity index in additive blended base oil (SN-500 + 1% ZnO). Though the value is lower in comparison to literature value of CuO-ZnO mixed oxide additive [28] and CuO nano additive [29], lubrication property of ZnO blended lubricant oil (SN-500 + 1% ZnO) was enhanced by addition of ZnO nano additive. It is an indication of good property of additive blended lubricant which will not fall its viscosity upon raising of temperature during operating condition of engine. This condition directly protects the tear and wear of sliding surfaces of engine.

**Pour point:** The pour point of base oil was investigated. Then the effect of additive on pour point of base oil was also observed and results are presented in Table 3. As can be seen in Table, the pour point of base oil was found to -6°C while pour point of additive blended base oil was found to be -9.2°C. It showed that pour point is significantly lowered when additive was blended in base oil. This value is the positive indication of suitability of lubricant in cold condition. It prevents the solidification of lubricant and prevents the jamming of engine in cold environment.

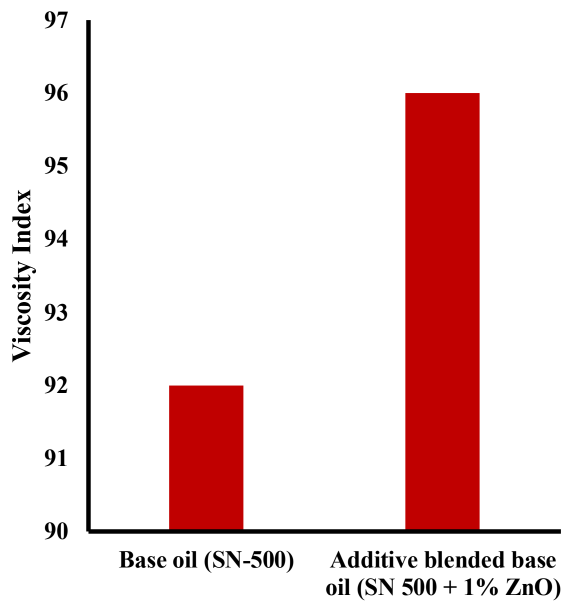


**Figure 7: A bar diagram showing the comparison of viscosity index of base oil (SN-500) and additive blended base oil (SN-500 + 1% ZnO)**

**Table 3. Pour point of base oil (SN-500) and additive blended base oil (SN-500 + 1% ZnO)**

| S.N. | Sample | Pour Point (°C) |
|---|---|---|
| 1 | Base oil (SN-500) | -6 |
| 2 | Additive blended base oil (SN-500 + 1% MO) | -9.2 |

**Flash point:** The flash point of base oil and additive base oil were obtained by careful investigation and results are shown in bar diagram (Figure 8**)**. It was found that the addition of 1% ZnO nanoparticles to the base oil causes an increase in flash point by 2.22% in comparison to base oil (Table 4). It may be due to increase thermal conductivity as reported in literature[23]. It was due to presence of extra material or additive on base oil which consequently resist against the fire. In addition to this, this extra material or additive which is nonvolatile leads to less amount of vapors get evaporated during flash point examination. This result is significant as it reduces a risk of fire during the use of lubricant.

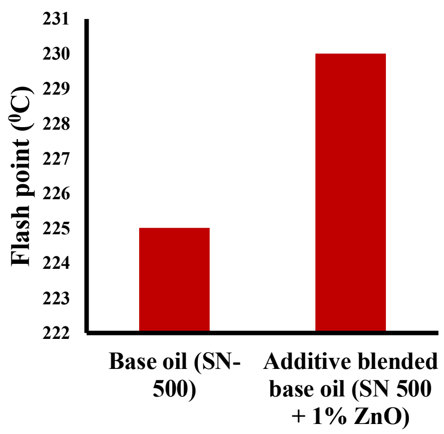


**Figure 8: A bar diagram showing the comparison of flash point of base oil (SN-500) and additive blended base oil (SN-500 + 1% ZnO)**

**Table 4. Flash point of base oil (SN-500) and additive blended base oil (SN -500 + 1% ZnO)**

| S.N. | Sample | Flash Point (°C) |
|---|---|---|
| 1 | Base oil (SN-500) | 225 |
| 2 | Additive blended base oil (SN-500 + 1% ZnO) | 230 |

**Density:** The density of base oil and additive blended base oil was determined. The results obtained are compared with density of base oil. Then comparative results are shown in Table 5. The value showed that the density of the base oil was increased slightly as it contains additive.

**Table 5. Density of base oil (SN-500) and additive blended base oil (SN -500 + 1% ZnO) at 29.5°C.**

| S.N. | Sample | Density at 29.5°C (g/mL) |
|---|---|---|
| 1 | Base oil (SN-500) | 0.882 |
| 2 | Additive blended base oil (SN 500 + 1% ZnO) | 0.885 |

**Emulsion test:** After emulsion test, it was observed that there is a complete separation of water and additive blended base oil (SN -500+1% ZnO) at 54°C after the thirty minutes.

**Corrosion test (Copper Strip Test):** The copper strip corrosion test results are shown in Table 6. The results showed that the corrosiveness of base oil and additive blended base oil both reached a rating of 1b in ASTM standard D 130-12 [30]. It is an indication of slight tarnish but not corrosion.

**Table 6. Copper strip corrosion tests of base oil (SN-500) and additive blended base oil (SN -500 + 1% ZnO)**

| S.N. | Sample | Copper strip corrosion, rating |
|---|---|---|
| 1 | Base oil (SN-500) | 1b |
| 2 | Additive blended base oil (SN 500 + 1% ZnO) | 1b |

**Moisture content test (Crackle test):** In moisture content test, water vapors were not found around the mouth of the test tube. In addition to this crackle sound was not produced during heating process. It means that the additive blended base oil (SN-500 + 1% ZnO) does not content noticeable amount of water. The result also support for corrosion test. Then further pH measurement also carried out which was found to be 6.65. The absence of water content and neutral pH also the indication of non-corrosive nature.

## Conclusion

ZnO can be synthesized by precipitation method. XRD pattern of ZnO shows sharp diffraction peaks indicating zincite structure. The crystallite size was found to be 16 nm, indicates the nanomaterials. Furthermore, in FT-IR spectrum showed the presence of stretch peak of ZnO at 587 $cm^{-1}$. The 1% wt/wt ZnO blended base oil were investigated to examine the lubricating properties. All the physio-chemical parameters; kinematic viscosity, viscosity index, flash point, pour point of the additive blended lubricant was found to be significantly improved than base oil without addition of the additive.


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