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Short communication

Green synthesis of cotton flower shaped nickel oxide nanoparticles: Anti-bacterial and tribological studies

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ABSTRACT

The goal of this study was to synthesize nickel oxide nanoparticles (NiO-NPs) by the sol–gel method, which involved the use of Cassia Fistula is a herbal and medicinal plant. The Cassia Fistula (CF) plant extract acts as a stabilizing and capping agent. Further, NiO-NPs were characterized using UV–Visible spectroscopy (UV–Vis), Powder X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Scanning electron microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX). According to the results of UV–Visible, the absorption spectroscopy has displayed that the band gap is 3.5 eV. The biological properties were studied for prepared NiO-NPs. The antibacterial testes were examined using different bacterial strains such as *Escherichia coli, Staphylococcus aureus* and *Staphylococcus Epidermis* and dose-dependent inhibition response was reported. The tribological tests are carried out using four ball testers in accordance with ASTM D4172-18 requirements. NiO-NPs are utilised as lubricating additives in poly alpha olefin (PAO4) base oil. NiO-NPs in PAO4 at a concentration of 0.5 wt% improved the coefficient of friction, and positive results are observed.

1. Introduction

In recent years, the area of nanotechnology has made enormous strides in the production of nanoparticles with exact size and morphology that are considered for certain applications. The study of nanotechnology is currently a major subject of research in all fields, from environmental protection to medicine. Nanoparticles (NPs) have certain features including size, distribution and shape. Nanoparticles offer a variety of applications because of their numerous distinctive, remarkable, and intriguing characteristics that distinguish them from their bulk counterparts. [1–3]. The main task is to improve specific approaches to synthesize NPs of definite size, specific shape, desired composition, and well-ordered dispersity that influence their physical, chemical, catalytic, optical, magnetic, electronic, and electrical properties making them ideal candidates for environmental, biomedical and biotechnological applications [4]. Nanoparticles are used in a variety of industries, including medicine, agriculture, environment industries, and as catalysts, with addition of lubricating additives. The synthesis of bio functional nanoparticles is extremely vital, and it has recently caught the attention of diverse analysis teams, creating a perpetually evolving space [5]. To address the growing demand for eco-friendly nanoparticles, researchers have utilized cost effective strategies for the synthesis of varies metal oxide nanoparticles for pharmaceutical applications [6]. Usually, the physical and chemical techniques are applied extensively but the physical techniques to synthesize NPs are highly expensive; however, the chemical techniques are detrimental to the environment and living organisms [7]. Therefore, it is necessary to add some capping agent to avoid aggregation and to achieve the desired morphology of product. Size reduction may also lead to increased reactivity and toxicity of synthesized material. Hence, priorate the wide scale implementation of these reactions, it is essential to predict the prospective hazards to the eco-system by making an allowance for the entire chemical process relating to all the species involved during the reaction mechanism. There are several options to attain the goals presented in previous reports cite the usage of microorganism, such as bacteria [8]. Algae [9]. Yeast [10] and Fungi [11]. for the biosynthesis of NPs. Lately, numerous plant extracts [12,13]. During the green synthesis of NPs, products from nature or those imitative of natural products

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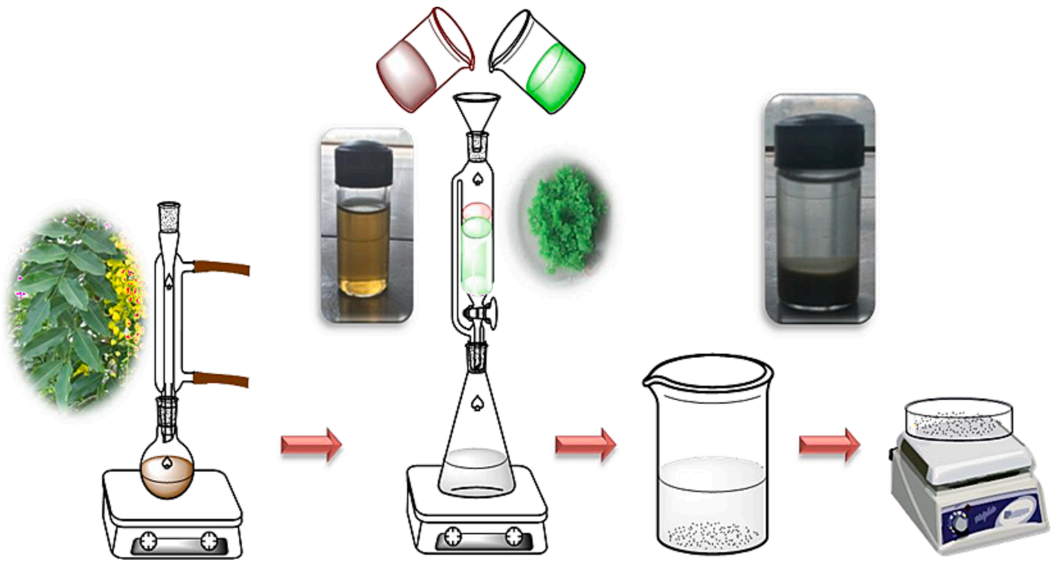

Fig. 1. Schematic diagram of biosynthesis NiO-NPs.

have been used as reducing and capping agents. The methods involved are typically simple, environmentally friendly and naturally compatible one-pot processes. It has been proved by various studies that the reductive abilities of the proteins and metabolites that are present in these biological systems can change inorganic metal ions into metal NPs. Techniques with an emphasis on recent advancements of green synthesis to fabricate metal and other important NPs; followed by a discussion of formation mechanisms and the conditions used to control the surface morphology, disparity and other properties of these biosynthesized NPs.

In this regard, green synthesis or bio materials innovation is cost effective, economical, most preferable and environmentally friendly to chemical and physical methods. Day by day several systematic examinations and improvements have been carried out of the quality of nanoparticles, their size, shape distribution and surface characterizing of these bio-inspired nano particles [14,15]. The developing green way materials enabling for medicinal application in everywhere viz., drug synthesis, drug delivery for cancer treatment, imaging, biological study, and multifunctional medicinal usages etc., In addition to in recent days researchers are focused on mechanical properties of nanoparticles such as tribological property. Nanoparticles are using as additives in automobile industry lubricants to increase the performance [16]. In the resent report's researchers are focused on study of NiO nanoparticles and their biological and mechanical properties.

2. Materials and methods

2.1. Collection of materials and preparation of plant extract

The fresh leaves of Cassia fistula are collected from Tirupathi, Andhra Pradesh, India. The collected leaves were washed with running tap water and twice with distilled water to remove dust particles and dried by using dried absorbent paper. Cassia fistula leaves (25 g) were cut into small pieces and 250 mL of distilled water was added to it. This was boiled at 80 °C for 30 min as shown in Fig. 1. The leaf extract was filtered by Whatman No. 1 filter paper to obtain the pure extract. The extract was yellow in color [25].

2.2. Biosynthesis of NiO-NPs

Bio perspective method was strictly followed for synthesis of NiO-NPs. Nickel chloride purchased from e-Merck (India) Ltd. Absolutely 20 mL of Cassia fistula leaf extract were taken in to 250 mL of round bottom flask and to them 80 mL of 0.1 M $NiCl_2$ are added drop wise separately with constant stirring at ambient temperature. Further 30 mints 2 mL of ammonia solution is added drop wise after completion of addition the solution was heating at 90 °C for 1 h. Then the reaction mixture colour change and (brown) precipitate was observed and adjust

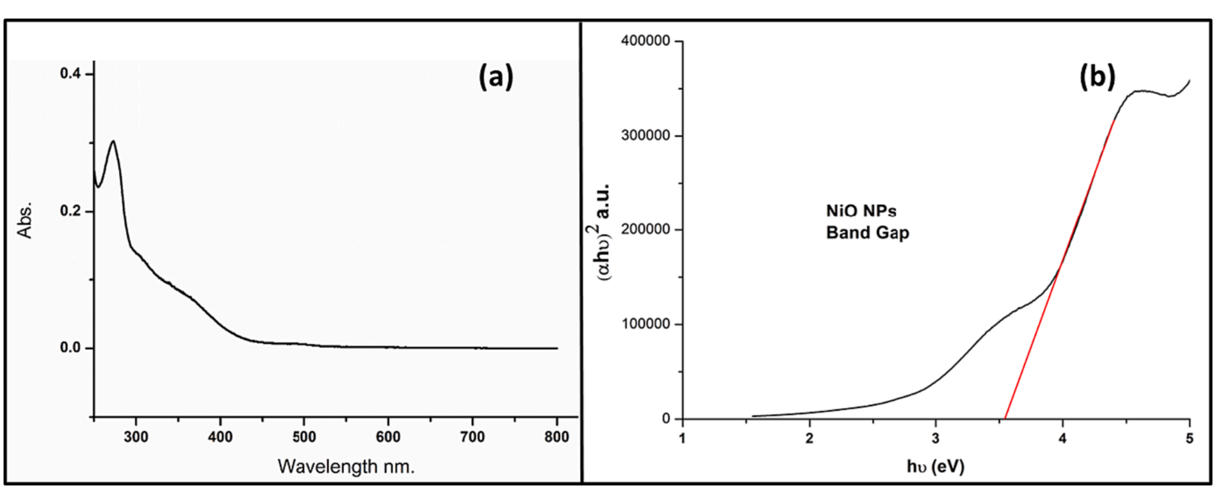

Fig. 2. (a) UV–Visible spectrum of NiO-NPs, (b) Band Gap.

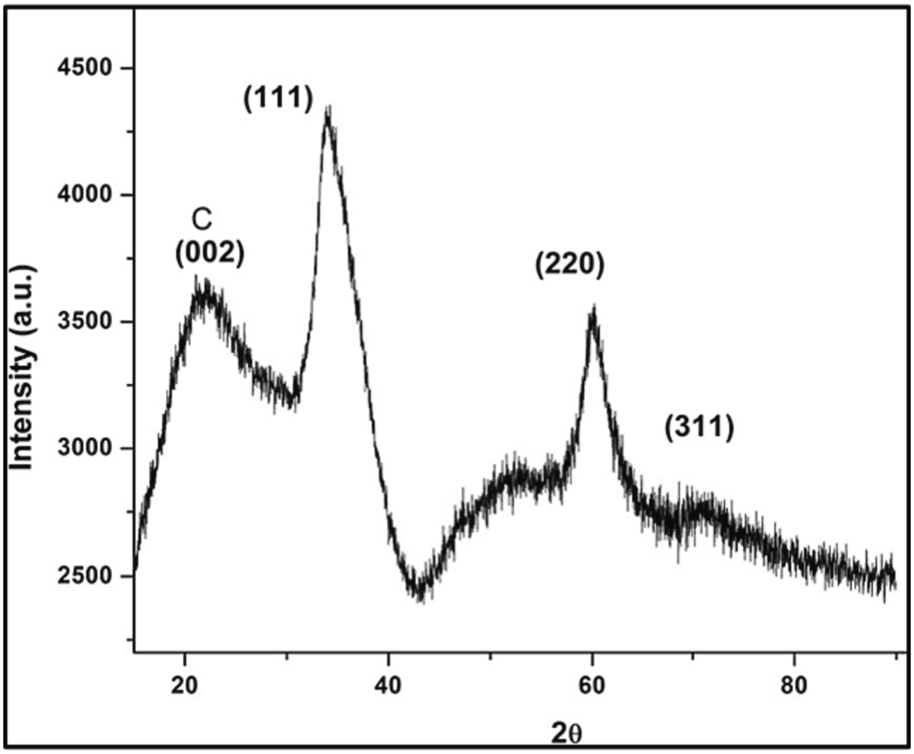

Fig. 3. XRD pattern of NiO-NPs.

p^H at 7. Then the reaction mixture was filtered. The NiO-NPs are washed with distilled water for 2–3 times to remove unbound phyto constituents or impurities. Then Nano particles dried at 100 °C in a hot air oven and stored at 4 °C for further use.

2.3. Tribiological Studies

The steel balls are cleaned with ethanol and acetone before tribological test. After that, they are dried for 30 min at 70 °C in an ultrasonic dryer. The TR30L four ball tester was manufactured by DUCOM Instruments Peenya manufacturing region for tribology research, Bangalore. Media containing NiO-NPs and PAO4 are applied to contact surfaces. Test conducted according to the ASTM D4172-18 standards.

3. Results and discussion

NiO-NPs have attracted vast interest due to their better optical properties. Colour change during the preparation of nanoparticles useful to control the reaction conditions to get good product. Fig. 1 signifies the green synthesis of the NiO-NPs from the Cassia Fistula leaf extract. Further, addition of nickel chloride solution to the leaf extracts the colour change from dark green to brown colour indicates the formation of NiO-NPs and are confirmed by XRD, SEM and EDX analysis.

3.1. UV Visible spectroscopy

The formation of NiO-NPs was characterized by optical absorption spectroscopy shown in Fig. 2 (a) absorption spectrum of NiO-NPs was observed at 290 nm which is characteristic absorption of NiO nanoparticles. There is another absorption appeared at 360 nm which is attributed to absorption of chromophores in the organic moieties adsorbed on NP s surface.

The optical band gap energy (Eg) was calculated using the Tauc formula given in Eq. (1)

$$(\alpha h\nu)^n = A\left(h\nu - E_g\right) \quad (1)$$

Where α is the absorption coefficient, h is the plank constant, and V is frequency of light [17–21]. Fig. 2 (b) showed the calculated energy band gap of the NiO-NPs is 3.5 eV.

3.2. Fourier transforms infrared spectroscopy

FT-IR spectra of prepared NiO-NPs using Cassia Fistula leaf extract shown in supporting information (Fig.S1) The broad band was observed at 3294 cm^{-1} was attributed to O–H stretching vibrations and a band at 1663 cm^{-1} was assigned to H-O–H bending vibrations [22]. The above two vibrations indicate the presence of H_2O. In FTIR spectra, metal–oxygen band occurs in between 500 and 838 cm^{-1} regions. The band at 691 cm^{-1} is associated with Ni-O–H stretching bond. These result well consistent with other previous reports. The peak cantered at 872 cm^{-1}, 466 cm^{-1} are assigned to NiO bending vibrations of the NiO [23].

3.3. Powder X-ray diffraction

The crystal structures of prepared NiO-NPs were evaluated by XRD. The XRD pattern of the prepared NiO-NPs is shown in Fig. 3. The diffraction patterns of the prepared NiO-NPs confirm the formation of a hexagonal structure. The characteristic peaks were observed in the XRD pattern at 2θ values of 34.040, 60.080 and 72.0 The diffraction peak corresponding to 2θ values of NiO were indexed by the (1 1 1), (2 2 0), and (3 1 1) diffraction patterns (JCPDS card no. 73–1523) [24]. In general, strong and sharp peaks can be seen from XRD, indicating the NiO NPs' crystalline structure [25].However, since we made NiO-NPs from plant extract, we can see broad peaks since the carbon residuals are concentrated more than usual. It is evident that there is a distinction between the two samples of pure NIO NPs and made in the presence of plant extract. It was observed that an extra peak value of 2θ at (0 0 2) indicates the existence of carbon residuals in the prepared NiO-NPs.

3.4. Scanning Electron Microscopy

The SEM examination is extensively used to study the morphology of

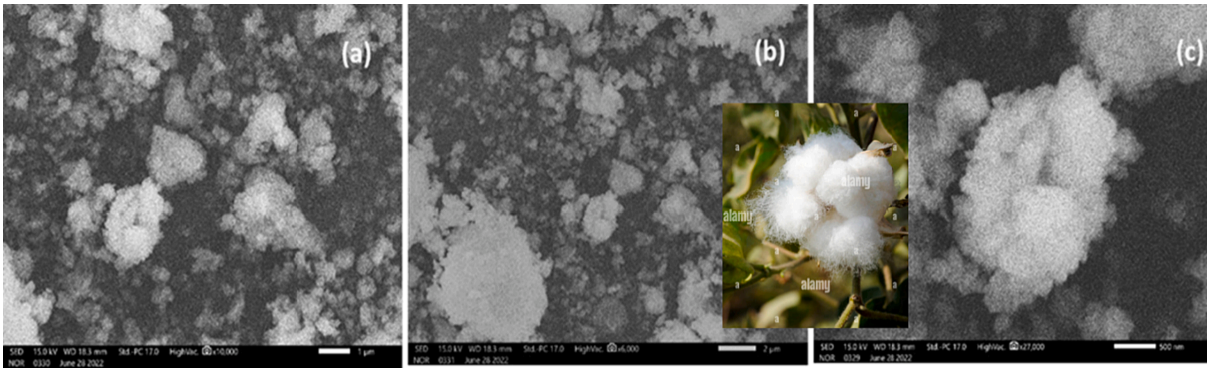

Fig. 4. SEM Morphological behaviour of NiO-NPs.

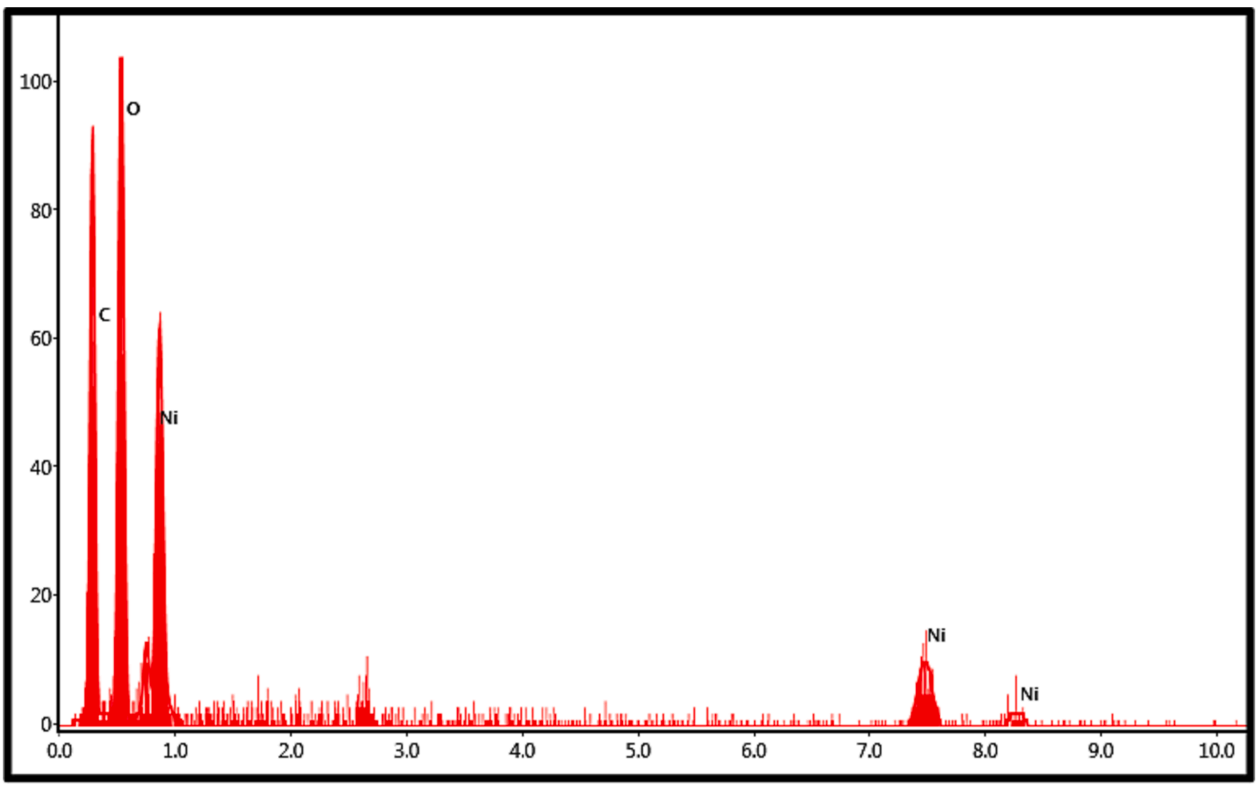

Fig. 5. Energy Dispersive X-Ray Spectroscopy of NiO-NPs.

nanomaterials. Fig. 4 displays the morphology of synthesized NiO-NPs, from the attained results, its appearances resembling nanoparticles aggregated and looks cotton flower like structures. Fig. 4 shows the particles are aligned in the range of 60–80 nm.

3.5. Energy Dispersive X-ray Spectroscopy

Energy Dispersive X-Ray Spectroscopy of prepared NiO-NPs using Cassia Fistula leaf extract shown in Fig. 5. It is a one of the good analytical tools for the chemical analysis of materials. From the figure, it is clearly identified that the presence of C, O and Ni from the prepared NiO-NPs were confirmed by EDX. The carbon is formed on the surface of the NPs due to presence of organic moieties, which are prepared using green synthesis.

3.6. Anti-bacterial Activity Assay

The antibacterial activity of leaf extracts (Aqueous and Ethanolic) of the plant was confirmed using Gram-positive bacterial strains (*Staphylococcus aureus* and *Staphylococcus epidermis*) and Gram-negative bacterial strains (*Klebsiella pneumonia* and *Escherichia coli*), which was determined by Disc Diffusion Method in the presence of nutrient agar medium [19]. Sterile Whatmann filter discs (6 mm diameter) were placed on nutrient agar plates and its column containing 106 CF U/ml of bacteria were spread on the solid plates with a sterile swab moistened bacterial suspension. Then 50 μL each of all aqueous and solvent extracts were placed in the discs made in inoculated plates. The treatments also included 50 μL of solvents served as control and Antibiotics (Augmentin, Amoxicillin, Tetracycline, Ciprofloxacin, Ceftriaxone and Cefotaxime) as a standard control. Then were incubated for 24 h at 37 °C and zone of inhibition if any around the wells were measured in mm (millimetre). Each treatment was repeated least twice replicates.

Table 1 showed the results of Antibiotics were used as reference drugs for definition of antimicrobial activities and compared with the activities of the plant crude extracts (aqueous and ethanolic) such as standard drugs (Augmentin, Amoxicillin, and Penicillin etc.) The tested

Table 1
Results of the NiO-NPs extracts against Escherichia coli, Staphylococcus aureus and Staphylococcus Epidermis.

Plant Extracts & Antibiotics	*Klebsiella Pneumoniae*	*Escherichia Coli*	*Staphylococcus Aureus*	*Staphylococcus Epidermis*
NiO-NPs Aqueous Solution	7 (mm)	14 (mm)	12 (mm)	11(mm)
NiO-NPs Ethanolic Solution	8 (mm)	16 (mm)	10 (mm)	9 (mm)
Augmentin	R	R	R	S
Amoxicillin	R	R	R	S
Ceftriaxone	S	S	R	S
Cefotaxime	S	S	R	S
Ciprofloxacin	R	S	S	R
Penicillin	R	S	S	R
Tetracycline	R	S	S	R

R = Resistant, S = Sensitive, (mm) = millimeters.

solutions of NiO-NPs have a good antibacterial activity against *E. coli* for aqueous solution of NiO-NPs with an inhibition zone of 14 mm and with an inhibition zone of 16 mm for ethanolic solution of NiO-NPs. The Staphylococcus Aureus with an inhibition zone of 10 mm for ethanolic solution of NiO-NPs with an inhibition zone of 12 mm for aqueous extract. The Staphylococcus Epidermis with an inhibition zone of 9 mm for ethanolic solution of NiO- NPs, with an inhibition zone of 11 mm for aqueous extract. The moderate antibacterial activity was observed against *Klebsiella pneumonia* with an inhibition zone of 8 mm for ethanolic solution of NiO-NPs with an inhibition zone of 6 mm for aqueous extract compared with appropriate antibiotics used. This may be deals with the fact that on the site of flavonoids compounds the number of hydroxyl groups determines the toxicity against the microorganisms (Davar F et al 2009). And also, related to the antimicrobial effects of flavonoids to their capacity to form complexes with extracellular and soluble proteins and with the cell wall. The antibacterial activity against strains was achieved by “Cassia Fiestula” conceivably owing to their high content of chemical constituents such as Flavonoids and Phenolics; also, its activity may be to presence of its high content of the Alkaloids.

Further, numerous NPs characteristics, including morphology and size, have an impact on their anti-microbial effectiveness. There are a few methods that have been reported, but no specific mechanism has been found that results in NPs’ antibacterial properties [17]. The produced NiO-NPs were used in the investigation and were predicted to follow the same pathways as other metal oxides. First, when microbes are exposed to NPs, cell membranes may be damaged. Second, there is a possibility that Ni ions may be released, which could have an impact on enzymes function during metabolism. The third method relies on the intrinsic property of NiO, which results in the release of reactive oxygen species (ROS) such superoxide (O2) and H2O2, which harm microbial DNA or RNA [3].

3.7. Tribological studies

There is research being done on a new class of lubricant additives called NPs. To adjust friction properties, NPs are typically combined with other materials in lubricant like oils and surfactants. The compatibility with NPs, as well as their size and morphology, will alter the lubricant quality. As a result of metal oxides stability and ability to endure greater temperatures NPs are promising additives for advanced lubricants.

Tribological studies conducted according to the procedure mentions in the materials and methods section 2.3. According to the ASTM D4172-18, each frictional experiment bears a load of 392 N, a rotational speed of 1200 rpm, temperature maintained at 75 °C, and a sliding period of 60 min. We used steel test balls with an AISI E52100 standard, diameter of 12.7 mm and a hardness ranging between 64 and 66 HRC. [26–27].

The results of each sample, three wear tests on a four-ball tribo tester are averaged after synthesis and experimentation. Additionally, data is gathered by a computer integrated to a four-ball tribo machine. The information that follows is an analysis of nanofluid behaviour with respect to time. NiO-NPs sample compositions in PAO4 base oil are 0.1, 0.2, 0.3, 0.4, 0.5, 0.6 and 0.7 wt%.

Four ball testers were used to test the effectiveness of NiO-NPs as lubricating additives in PAO4 oil for 60 min. The quantity of nano-additives present in PAO4 base oil is shown in Figure. The COF is gradually raised from 0.7 wt% (0.104) after being dropped from zero Wt % (0.136) to 0.5 wt% (0.083). The optimal weight percentage is 0.5, and the COF is 0.083. It exhibits exceptional tribological properties, which can observe from Fig. 6.Fig. 7.

NPs are important when using lubricant in machinery because they change the surface characteristics of metals and reduce friction by creating a ball bearing effect. Numerous mechanisms, including the ball bearing effect, protective coating generation, mending effect, and polishing effect, can be used to explain how the addition of NPs improves the lubricating property.[28].

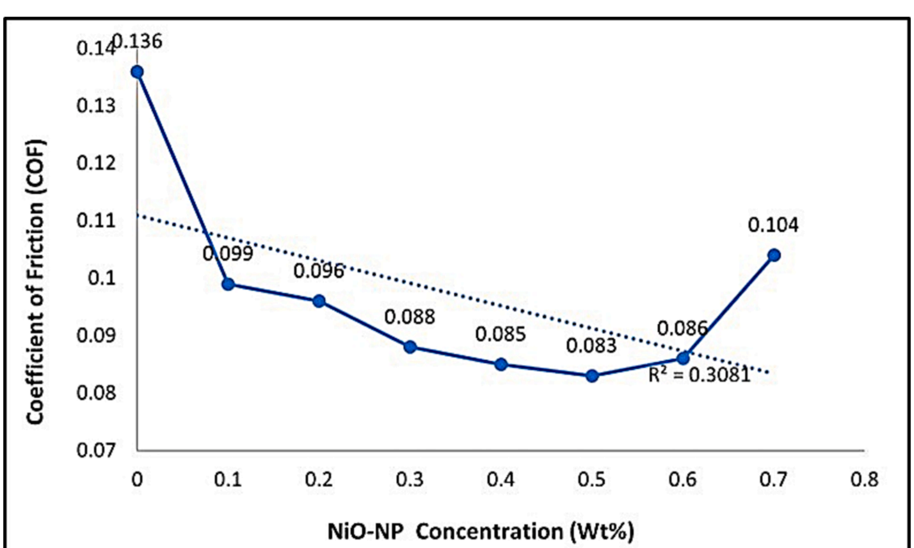

Fig. 6. Analysis of NiO-NPs are nano additives in PAO4 after 60-minutes.

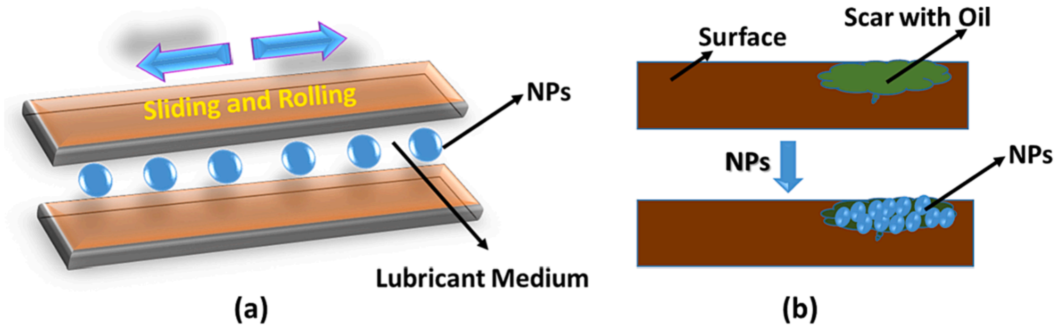

Fig.7. (a) ball bearing effect, (b) mending effect of nanoparticles.

The current results show that NIO-NPs enhance lubricating characteristics, which can be explained by the following methods. As seen in Fig. (a), NIO NPs are stable and roll in between surfaces that are shearing metal, which reduces friction.

Furthermore, because NPs are nanometer-sized, they can fill surface groves and scars to enhance surface characteristics, a process known as the "mending effect." This further lowers the surface's coefficient of friction [29]. The nanoparticles' ability to repair damage is described in Fig. (b).

4. Conclusion

In this study, NiO-NPs were synthesized by the sol–gel method, which involved the use of Cassia Fistula (CF) plant extract acts as a stabilizing and capping agent. The nanoparticles was calcinated at 600 °C. The characterization approaches of UV–Visible spectroscopy (UV–Vis), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Scanning electron microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX). According to the results of UV–Vis, the gap band of nanoparticles was calculated to be in the range of about 2.4–3.5 eV. The antibacterial testes were examined using different bacterial strains such as *Escherichia coli, Staphylococcus aureus, Staphylococcus Epidermis.* The results indicate that the prepared NiO-NPs have a promising candidate for antibacterial activity. The life of the mating parts is increased by a small addition of these substances- NiO-NPs, to the synthetic lubricant. 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, and 0.7 wt% represent most of them. NiO-NPs are good anti-friction and durably additive to oils. When compared to pure base oil, the tribological characteristics of PAO4 were improved by 39% with the addition of 0.5 wt% NiO-NPs. The results indicate that the prepared nanoparticles have good tribological property.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2023.111239.

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