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Synthesis and Characterization of Syzygium Cumini Leaves Mediated Nickel Oxide Nanoparticles

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Abstract

Nickel oxide (NiO) nanoparticles possess unique properties that make them highly valuable for applications in energy storage, catalysis, sensors, and biomedicine. Conventional synthesis routes often involve toxic chemicals and high energy requirements, posing environmental concerns. In this study, NiO nanoparticles were successfully synthesized via a green synthesis route using Syzygium cumini leaf extract as both a natural reducing agent and capping agent. The optical properties were monitored through UV-Vis spectroscopy, revealing the transformation from nickel chloride to NiO nanoparticles with a distinct absorption shift from ~394 nm to ~286 nm, confirming nanoparticle formation and semiconducting behavior. Phytochemicals screening results and Fourier Transform Infrared (FTIR) analysis identified functional groups in the extract, including phenols, flavonoids, and carboxylic groups, suggesting their role in reduction and stabilization. Scanning Electron Microscopy (SEM) images showed aggregated nanoparticles with irregular crystalline morphology, while X-ray diffraction (XRD) confirmed a face-centered cubic phase with an average crystallite size of 15.5 nm. The study demonstrates the effectiveness of S. cumini extract in producing NiO nanoparticles sustainably and highlights the potential of plant-mediated routes for environmentally friendly nanomaterial synthesis.

Keywords: Green synthesis; NiONPs; Syzygium cumini; Phytochemicals; Nanotechnology.

I. INTRODUCTION

Nanotechnology has emerged as a transformative area in materials science, offering new solutions in diverse fields ranging from electronics and energy storage to catalysis and biomedicine [1-5]. Among the types of nanomaterials, nickel oxide (NiO) nanoparticles have attracted much attention due to their unique physicochemical properties, including high thermal stability, a wide range of densities, electrochemical activity, and high efficiency [1-3, 5-12]. These properties make NiO nanoparticles valuable in various applications such as supercapacitors, sensors, fuel cells, photocatalysis, and antimicrobial agents [1-3, 5, 9, 12-16]. Conventional synthesis methods for NiO nanoparticles often involve high energy consumption and the use of hazardous chemicals, raising concerns about environmental impact and sustainability [3, 16]. In recent years, green synthesis approaches have garnered significant attention as environmentally friendly, cost-effective, and sustainable alternatives to conventional methods [3-10, 11-18]. These methods use natural substances such as plant extracts, bacteria, or enzymes as reducing and stabilizing agents, thereby reducing the environmental footprint associated with nanoparticle production [3-10, 11-18].

Syzygium cumini (commonly known as Java plum or black plum) is a plant with a wide range of medicinal properties, rich in bioactive phytochemicals including flavonoids, tannins, terpenoids, and polyphenols [10]. These phytoconstituents not only provide antioxidant, anti-inflammatory, and antimicrobial activities but also act as effective agents in the biosynthesis of metal and metal oxide nanoparticles [10, 12-19]. Although Syzygium cumini has been extensively studied within the context of traditional medicine, its application in green nanotechnology, particularly in the biosynthesis of nickel oxide (NiO) nanoparticles, remains relatively underexplored [10, 20]. This study aimed to synthesize nickel oxide nanoparticles using Syzygium cumini leaf extract as a natural reducing and capping agent. The synthesized nanoparticles were systematically characterized using a range of analytical techniques to elucidate their structural, morphological, and functional properties. The project not only contributes to the advancement of sustainable nanomaterial development but also demonstrates the usefulness of medicinal plants in developing green nanotechnological applications.

II. MATERIALS AND METHODS

A. Materials

All chemicals used in this study were of analytical grade. The Syzygium cumini leaves were collected from Adamawa State University, Mubi, Adamawa State, Nigeria, and were taxonomically identified at the Department of Botany, Adamawa State University, Mubi, Adamawa State, Nigeria. All solutions were prepared using distilled water.

B. Sample Collection

The Syzygium tree is shown in Plate 1(a), while a close picture of the collected leaves is depicted in Plate 1(b).

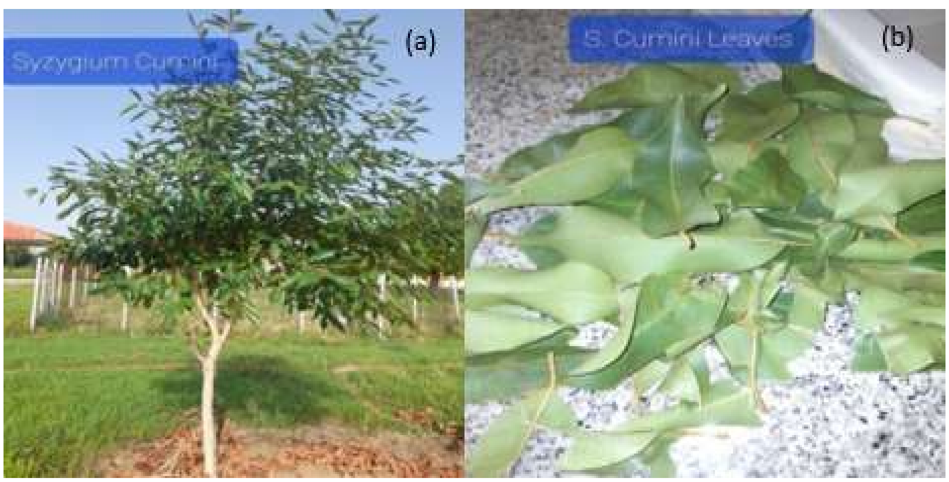

Plate 1. Syzygium cumini (a) tree and (b) leaves.

C. Preparation of Plant Extracts

The fresh Syzygium cumini leaves were finely chopped, thoroughly washed, and air-dried at ambient room temperature for twelve (12) hours. A total of 200 g of the dried leaf material was measured using an analytical balance. The leaves are put in a beaker containing 200 mL of distilled water and heated at a temperature of 200 °C on a hotplate for 30 minutes. The extract is allowed to cool and filtered using a Whatman number 1 filter paper.

D. Preparation of solutions

1) FNiCl$_2$.6H$_2$O solution

A quantity of 1.88 g of nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$) was accurately weighed and dissolved in 250 cm³ of distilled water in a beaker and stirred continuously to obtain a 2 M solution.

E. Synthesis of nickel oxide nanoparticles

A volume of 50 mL of 0.02 M nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$) solution was transferred into a beaker. The pH of the plant extract and the $NiCl_2 \cdot 6H_2O$ solution were measured to be 4.11 and 3.44, respectively. The beaker equipped with a magnetic stirrer was placed on a hot plate set to 600 °C. Subsequently, 10 mL of the plant extract was added dropwise to the salt solution stirred continuously. The mixture was heated and stirred for 30 minutes, then allowed to cool to room temperature. The resulting suspension was centrifuged at 3000 rpm for 10 minutes and washed repeatedly with deionized water to remove impurities. The synthesized nickel oxide nanoparticles are shown in Plate 2.

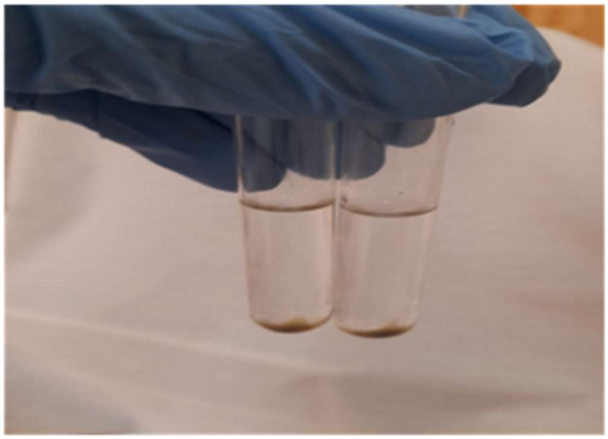

Plate 2. Synthesized nickel oxide nanoparticles.

F. Phytochemical Screening

The phytochemical screening of Syzygium cumini leaves extract was carried out using the following prescribed standard methods.

1) Test for Phenols Using Ferric Chloride Test

A 0.5 mL aliquot of the plant extract was diluted with 10 mL of distilled water, filtered, and then treated with a 1% ferric chloride ($FeCl_3$) solution. The appearance of a blue-black coloration indicated the presence of phenolic compounds [22-23].

2) Test for Terpenes/Steroids Using Salkowski Test

To the 2 mL of the plant extract, 2 mL of chloroform and five (5) drops of concentrated sulfuric acid (H_2SO_4) were added. The formation of a reddish-brown coloration at the interface indicated the presence of terpenoids [21-22].

3) Test for Saponins Using the Frothing Test

A 2 mL portion of the plant extract was diluted with 10 mL of distilled water and vigorously shaken for 30 seconds. The mixture was then allowed to stand undisturbed for 30 minutes. The formation of a persistent honeycomb-like froth indicated the presence of saponins.

4) Test for Alkaloids Using Wagner's Test

A 3 mL aliquot of the plant extract was stirred with 1 mL of aqueous hydrochloric acid (HCl) on a steam bath and subsequently filtered. A few drops of iodine solution in potassium iodide (Wagner's reagent) were added to the filtrate. The formation of a precipitate indicated the presence of alkaloids [23].

5) Test for Tanins Using Lead Sub-acetate Test

Three (3) drops of lead subacetate solution were added to 2 mL of the plant extract. The formation of a brown precipitate indicated the presence of tannins [23].

6) Test for Flavonoids Using (i) Lead Acetate Test, and (ii) Shinoda Test

To the 5 mL of the plant extract, five (5) drops of 10% lead acetate solution were added. The formation of a grey precipitate indicated the presence of flavonoids [23]. Additionally, 2 mL of the extract was treated with a small piece of magnesium ribbon, followed by the addition of three (3) drops of concentrated hydrochloric acid. The development of an orange or pink-red coloration further confirmed the presence of flavonoids [23].

7) Test for Glycoside

A 0.1 g portion of the plant extract was dissolved in 1.0 cm^3 of glacial acetic acid containing one drop of ferric chloride solution. Subsequently, 1.0 cm^3 of concentrated sulfuric acid was carefully added along the side of the test tube. The formation of a brown ring at the interface indicated the presence of deoxy sugars, characteristic of cardenolides [23].

G. Characterization and Measurement

The optical study of NiONPs was recorded using an Axiom Medicals (UV752 UV-Vis-NIR spectrophotometer). Scanning Electron Microscopy (SEM) images were obtained using Phenom pro-X, with an acceleration voltage of 10 kV, while structural analysis of the TiO_2 film was performed using an X-ray diffractometer (Rigaku D, Max). To determine the biomolecules present in the leaf extract, which were responsible for the bio-reduction of NiO nanoparticles, Fourier Transform Infrared Spectroscopy(FTIR) was used.

III. RESULTS AND DISCUSSION

A. Phytochemical Analysis

The result of phytochemical screening of Syzygium cumini leaves extract is presented in Table I.

Table I. Qualitative phytochemical analysis of Syzygium cumini aqueous leaf extract

Phytochemical	Result
Alkaloids	+
Flavonoids	+++
Glycosides	+++
Steroids	+
Phenols	++
Tanins	++
Terpenoids	?
Resins	+
Proteins	-

Key: + present ++ Moderately present +++ Appreciable amount - absent

Previous studies have demonstrated that flavonoids function as both bioreducing agents and electrostatic stabilizers in the green synthesis of metal nanoparticles, while phytochemicals such as glycosides, steroids, alkaloids, phenols, and tannins have also been identified as potential reducing, capping, and stabilizing agents in plant-mediated nanoparticle synthesis [21–25].

B. Optical Activity

As depicted in Plates 3(a) and (b), the plant extract was golden brown, while the nickel chloride solution was green. Upon the addition of 10 mL of Syzygium cumini extract to 50 mL of nickel chloride solution, the solution turned light green and then light yellow after approximately 30 minutes, indicating the formation of nickel nanoparticles (see Plate 3(c)) [1-3, 5]. During the biosynthesis of metal nanoparticles, a colour change was observed as seen in Plate 3(d), which could be attributed to the localized surface plasmon resonance (LSPR) effects associated with the NiONPs. These effects enhance the absorption and scattering of light due to the collective oscillations of surface electrons [1, 3, 16]. When light interacts with metal oxide nanoparticles, it induces surface electron vibrations, leading to surface plasmon resonance (SPR) phenomena [1-5]. The frequency of this surface SPR wave determines the colour of the metal nanoparticles that we observe [1-5, 9, 12]. An observed change in colour indicating the formation of nanoparticles in synthesis involving biological agents was attributed to the excitation of surface Plasmon vibrations in the metal nanoparticles [24].

As the size, shape, and composition of the nanoparticles change during biosynthesis, so does the frequency of the surface plasmon resonance wave, leading to a change in the observed colour. This effect is especially noticeable when the size of the nanoparticles changes from larger to smaller sizes, leading to a shift from longer (red-shift) to shorter (blue-shift) optical path lengths of light being absorbed and scattered, resulting in a colour change [25].

Plate 3. Colour changes for the synthesis of nickel oxide nanoparticles.

C. Optical Study

Fig. 1 shows the absorption spectra of unsynthesized ($NiCl_2.6H_2O$) and synthesized NiO nanoparticles (NiONPs) using Syzygium cumini plant extract in a wavelength range of 200 nm to 800 nm. The unsythesized $NiCl_2.6H_2O$ is a molecular compound where nickel exists in an ionic form, Ni^{2+}, surrounded by water molecules in a crystal lattice [1-3, 5, 9, 12]. The sharp and strong absorption peak in the ultraviolet region at approximately 394 nm corresponds to d-d transitions of Ni^{2+} ions – in Ni^{2+}, electrons in 3d orbitals can be excited between split energy levels under the influence of the octahedral ligand field of water molecules [1-3, 5-10]. These transitions have discrete energies, giving sharp, well-defined peaks in the UV spectrum and exponentially decreased as they skewed to the visible region of the solar spectrum of the electromagnetic radiation.

When $NiCl_2.6H_2O$ was synthesized into NiO nanoparticles by Syzygium cumini plant extract, the UV-Vis spectra help confirm successful synthesis through the disappearance of the d-d peak in NiONPs, and a bluer shift with strong absorption in the UV-region at ~ 286 nm proves transformation to oxide [1-5, 8, 9, 12]. Broad absorption confirms semiconducting behavior of NiONPs [1-5, 8, 9, 12]. The shift to broader absorption is a hallmark of nanostructured semiconductors.

In summary, $NiCl_2.6H_2O$ shows a sharp and strong in absorption UV-region due to Ni^{2+} d-d transitions in its hydrated ionic structure while NiONPs show broad absorption because of semiconductor band gap transitions, quantum confinement, surface defect and size dispersion, leading to loss of sharp features and a broad, shifted spectrum[1-5, 8, 9, 12].

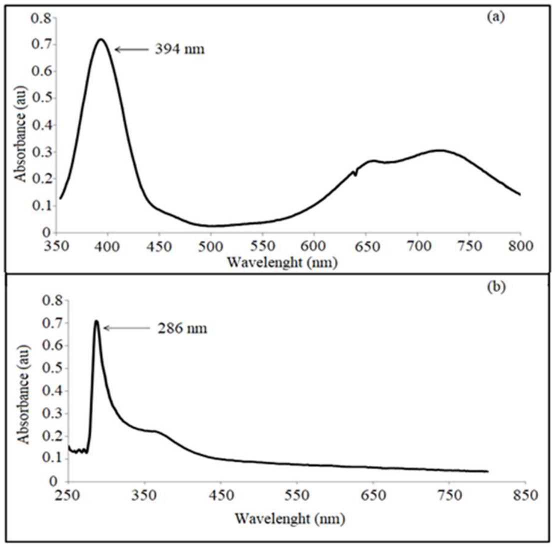

Fig. 1. UV-visible spectra of (a) $NiCl_2.6H_2O$ and (b) NiO nanoparticles

D. Fourier Transform Infrared Spectroscopy (FTIR) Study

Fourier transform infrared spectroscopy was used to identify functional groups of the phytochemicals present in the plant extract. Fig. 2 presents a comparative FTIR spectra of S. cumini and nickel oxide nanoparticles.

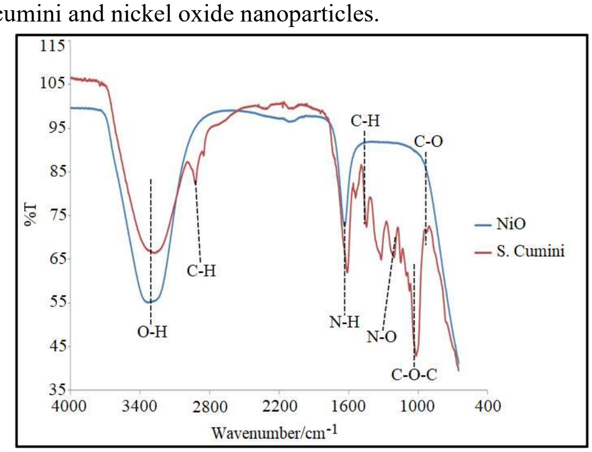

Fig. 2. Comparative FTIR spectra of S. cumini and nickel oxide nanoparticles.

The notable peaks are; 3345 cm^{-1} (-OH carboxylics), 2900 cm^{-1} (–CH stretch alkanes), 1619 cm^{-1} (-NH amines), 1455 cm^{-1} alkanes, 1371 cm^{-1} (-C-O anhydrides) 1353 cm^{-1} (nitro),1265 cm^{-1} (-C-O-C ethers),1000 cm^{-1} (-CH ethers), 957 cm^{-1} (-C-O anhydrides) [23].

The possible biomolecules responsible for the reduction of Ni^{2+} ions and capping to form NiO NPs are OH 3345 cm^{-1} and alcohols, -OH 3345 cm^{-1} and -CO 1371 cm^{-1} carboxylics, -C-O-C 1265 cm^{-1} and -CH 1000 cm^{-1} ethers. The bands obtained from the NiO Nps were 3300 cm^{-1} and 1700 cm^{-1} [23]. Two bands of near equal wave number that were found in NiO NPs and plant extract are 3345 cm^{-1} and 1619 cm^{-1}. The bands at 1353 -1380 cm^{-1}indicate the -OH deformation vibration in the aromatic ring/phenol. The band at 1619 cm^{-1} corresponds to -C=C groups or aromatic rings of polyphenols [23].

E. Scanning Electron Microscopy (SEM)

Fig. 3 illustrates the SEM image of the synthesized NiONPs with plant extracts.

The particles appear as small, irregularly shaped grains that are clustered together into larger groups. This clustering is called agglomeration, which happens because nanoparticles naturally stick together due to attractive forces [1-5]. The particles have irregular, often faceted shapes, suggesting they are crystalline. Individual particles are in the nanometer size range, but they clump together to form larger aggregates visible under the SEM [1-3, 5-10]. The particles are not perfectly separated but group together[5-10]. This is normal for nanoparticles, as their small size and high surface energy cause them to attract each other. The surface of the particle looks rough and uneven. This indicates the presence of surface defects or active sites, which can be useful in applications like catalysis or sensing [1-3, 5-11]. The irregular shapes and facets confirm the crystalline nature of NiONPs [1-3, 5-11]. The agglomeration may reduce the available surface area, but is typical for nanomaterials [1-3, 5-10]. Surface roughness and defects could influence the optical and electronic properties, as seen in the UV-Vis spectra in Fig. 2. In summary, the SEM image shows that NiONPs were successfully synthesized, displaying crystalline, irregular shapes, with some degree of clustering. These features are important for their functional properties in various applications [10-12, 16-21].

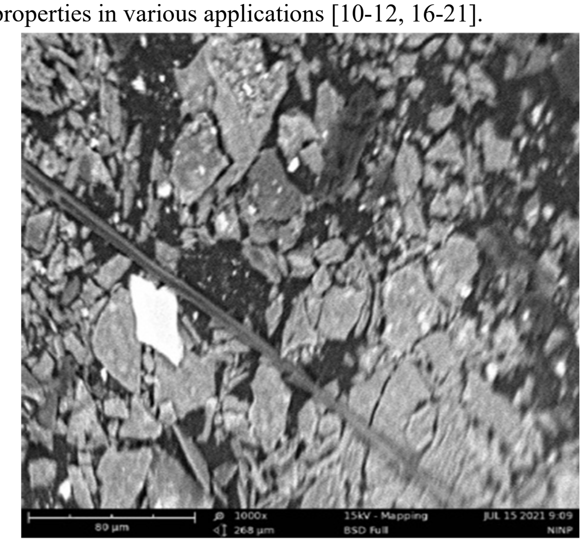

Fig. 3. SEM micrograph of nickel oxide nanoparticles.

F. X-Ray Diffraction (XRD) Study

The XRD technique was used to determine the phase and structure of the sample. Fig. 4 shows the XRD patterns characteristic of NiO nanomaterials.

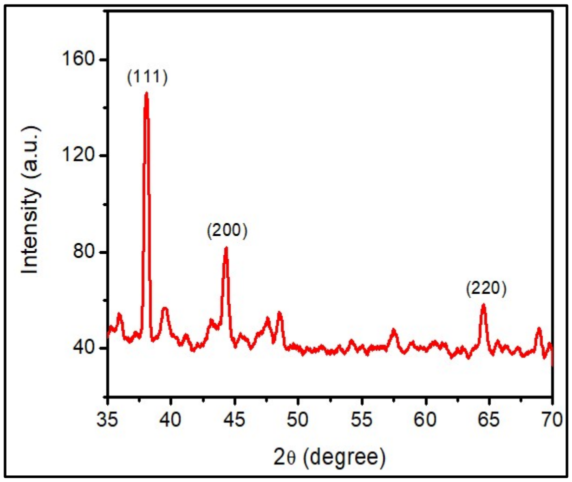

Fig. 4. XRD profile of nickel oxide nanoparticle.

All the replications in the XRD pattern can be indexed to face-centered cubic phase NiO (JCPDS card #47-1049[1-3, 5, 9, 12]. The three characteristic peaks at 38.5°, 44.5°, and 64.5° correspond to the (111), (200), and (220) diffraction planes, respectively. The high peak intensity indicates that the NiO nanomaterial is of high crystallinity. The un-indexed peaks could have possibly resulted from the presence of unwanted impurities during synthesis and deposition processes. Likewise, no peaks from the Ni substrate were detected, suggesting that the NiO nanoparticles are uniformly grown upon the NiNPs spray surface [1-3, 5, 9, 12].

The average crystallite size was calculated using the Scherrer equation in (1), based on the half-width of the (200) peak, which is about 15.5 nm.

$$D = k\lambda(B\cos\theta) \quad (1)$$

Where D is the average crystalline size, k is the geometric factor with value (0.9), λ is the wavelength of X-ray radiation source, and B is the angular full width at half maximum of the XRD peak at the diffraction angle [1-5].

IV. Conclusion

This study successfully demonstrated the green synthesis of nickel oxide nanoparticles using Syzygium cumini leaf extract as both reducing and stabilizing agents. Characterization by UV-Vis spectroscopy, FTIR, SEM, and XRD confirmed the formation of crystalline NiO nanoparticles with nanoscale dimensions and semiconducting properties. The phytochemicals in S. cumini, including phenols and flavonoids, played a critical role in the reduction and stabilization processes. This green approach offers a sustainable, low-toxicity alternative to conventional methods, reinforcing the significant potential of medicinal plants in eco-friendly nanotechnology applications.

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