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Synthesis, characterization and electrochemical activity of *Syzygium cumini* leaves mediated Nickel Oxide nanoparticles

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Abstract
Nickel oxide nanoparticles were synthesized from $NiCl_2.6H_2O$ solution using aqueous extract of SyzygiumCumini at 60^0C and formation of nanoparticles carried out with precipitation method, which were centrifuged, washed, and dried. It was characterized by microscopic and spectroscopic techniques, UV-visible spectroscopy, Fourier transform infrared spectroscopy, scanning electron microscope, X-ray diffraction and electrochemical impedance spectroscopy. The UV-visible spectra showed nickel nanoparticles maximum peak at 286nm, with the reported range for nickel oxide nanoparticles as 265-297nm. The FTIR spectra showed bands corresponding to ethers, phenols and polyphenols which stabilized and cap the NiO nanoparticles. The scanning electron microscope image of synthesized NiO nanoparticles showed the nanoparticles are dense irregular structure and shapeless on the surface. The XRD showed patterns characteristics of NiO nanomaterial. The replication can be indexed to face-centered cubic phase NiO. The average crystallite size is about15.5nm. The cyclic voltametry showed the modified electrode as being electroactive.

Keywords: Green synthesis, NiONPs, *Syzygium cumini*, Phytochemicals, Nanotechnology

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 methods have gained momentum as environmentally friendly, cost-effective, and sustainable alternatives [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].

Syzygiumcumini (commonly known as Java plum or black plum), a plant with a wide range of medicinal properties, is 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]. Despite the widespread research of Syzygiumcumini in traditional medicine, its potential in green nanotechnology, especially in the synthesis of NiO nanoparticles, remains relatively underexplored [10, 20]. This study aimed to synthesize nickel oxide nanoparticles using Syzygiumcumini 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.

Materials and Methods

a. Materials

All the chemicals used were of analytical grade. The plant *Syzygium cumini* leaves were obtained from Adamawa State University, Mubi, Adamawa State and were identified at the Department of Botany, Adamawa State University, Mubi. The solutions were prepared using distilled water.

b. Sample Collection

The leaves were collected from the tree as seen from plate (a), washed, and dried at room temperature for 12hours as seen in plate (b). The leaves were cut into little pieces.

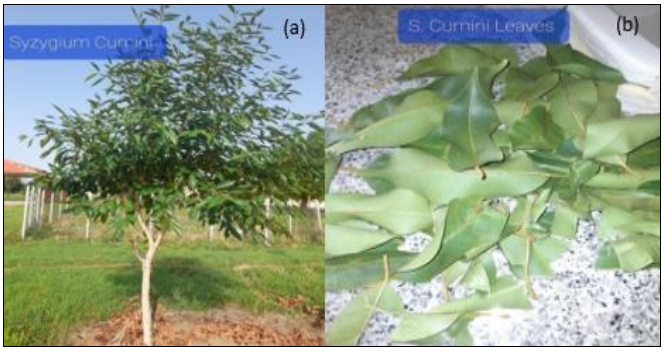

Plate 1: Syzygiumcumini:(a) three and (b)leaves

c. Preparation of Plant Extracts

The fresh Syzygiumcumini leaves 200 g were cut into pieces, washed, dried at room temperature and weighed on an analytical balance. The leaves are put in a beaker containing 200ml of distilled water and heated at a temperature of 2000C 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

F$NiCl_2.6H_2O$ solution

1.88 g of $NiCl_2$.6H2O was weighed into a beaker, and 250cm^3 of distilled water was poured and stirred. The solution prepared was 2M.

e. Synthesis of nickel oxide nanoparticles

50ml of 0.02 M $NiCl_2.6H_2O$ solution was poured into a beaker. The pH of the plant extract was 4.11, while that of the $NiCl_2.6H_2O$ solution was 3.44. The beaker containing the solution was placed on a hot plate at 600 °C with a magnetic stirrer, then 10ml of the plant extract was added to the salt solution in drops. The solution was heated and stirred for 30minutes. The solution was allowed to cool and centrifuged at 3000 rpm for 10 minutes and washed several times with deionized water. Plate 2 depicts the synthesized nickel oxide nanoparticles.

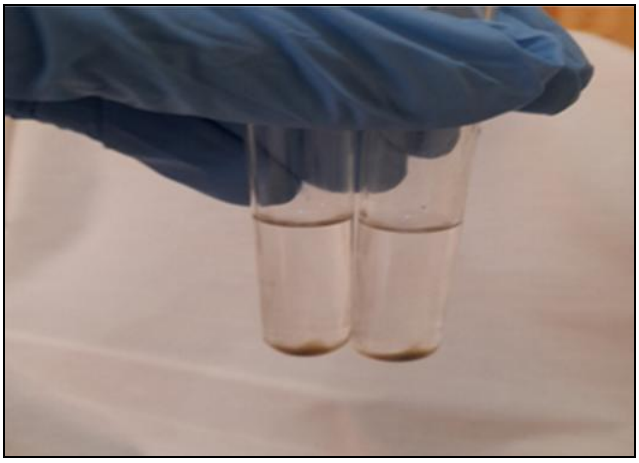

Plate 2: Synthesized nickel oxide nanoparticles

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**
   0.5 ml of the extract was dissolved in 10 ml of water, filtered, and subsequently treated with a 1% ferric chloride solution. A blue-black colouration indicates the presence of phenols [22-23].

2. **Test for Terpenes/Steroids Using Salkowski Test**
   2 ml of chloroform and 5 drops of concentrated sulphuric acid were added to 2ml of the extract. A reddish-brown colour at the interphase indicates the presence of terpenoids [21-22].

3. **Test for Saponins Using Frothing Test**
   A 2 ml of the extract was dissolved in 10 ml of water, and it was shaken vigorously for 30 seconds. It was then allowed to stand for 30 minutes. A persistent honey-comb froth indicates the presence of saponins.

4. **Test of Alkaloids Using Wagner's Test**
   3 ml of the extract was stirred with 1 ml of aqueous hydrochloric acid on a steam bath and filtered. A solution of iodine in potassium iodide was then added to the filtrate. The formation of precipitate indicates the presence of Alkaloids [23].

5. **Test for Tanins Using Lead Sub-Acetate Test**
   3 drops of lead sub-acetate were added to the 2 ml of the extract; a brown precipitate indicates the presence of tannins [23].

6. **Test for Flavonoids Using (i) Lead Acetate Test, and(ii) Shinoda Test**
   5 drops of 10% lead acetate solution were added to the 5ml of the extract; a grey precipitate indicates the presence of flavonoids [23]. Also, a piece of magnesium filings and 3 drops of concentrated hydrochloric acid were addedto the 2ml of the extract. An orange or a pink-red colouration indicates the presence of flavonoids [23].

7. **Test for Glycoside**
   The extract 0.1g was dissolved in 1.0cm^3 of glacial acetic acid containing one drop of ferric chloride solution. Concentrated sulphuric acid 1.0cm^3 was added gently to the side of the test tube. A brown ring formed at the interphase indicates the presence of deoxy sugar, 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. 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, Fuorier Transform Infrared Spectroscopy (FTIR) was used. Electrochemical measurements of cyclic voltametry and electrochemical impedance spectroscopy was carried out using Autolab potentiostat PGSTAT driven by Anova 1.8 Software

Results and Discussion

a. Phytochemical Analysis

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

Table 1: Qualitative phytochemical analysis of *Syzygium cumini*aqueous leaf

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 Plate3(a) and (b), the plant extract was golden brown, while the nickel chloride solution was green. When 10ml of Syzygiumcumini extract was added to 50ml of nickel chloride solution, the solution turned light green and then light yellow after about 30 minutes, indicating the formation of nickel nanoparticles (see Plate 3(c)) [1-3, 5]. During biosynthesis of metal nanoparticles, a change in coloured was observed as seen in Plate3(d), which could be ascribed to the localized surface plasmon resonance (LSPR) effects that arise from the NiONPs for enhanced absorptions and scattering of light in different ways [1, 3, 16]. When light interacts with metal oxide nanoparticles, it can cause the electrons on the surface of the particles to vibrate, creating a type of wave called a surface plasmon resonance (SPR) [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, which leads 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].

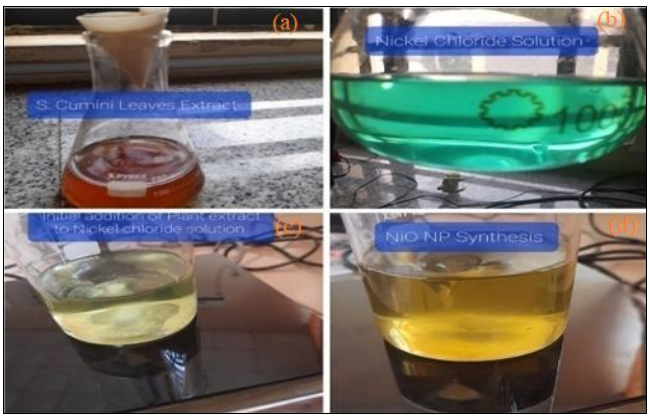

Plate 3: Colour changes for the synthesis of nickel oxide nanoparticles.

c. Optical Study

Fig. 1 shows the absorption spectra of unsynthesized ($NiCl_2.6H_2O$) and synthesized NiOnanoparticles (NiONPs) using Syzygiumcumini 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 394nm 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.

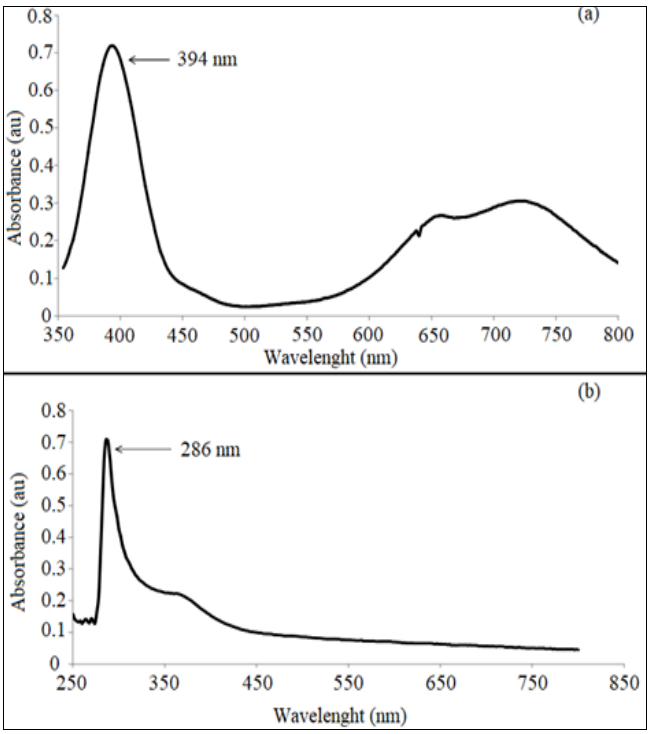

Fig 1: UV-visible spectra of (a) $NiCl_2.6H_2O$ and (b) NiO nanoparticles

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].

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.

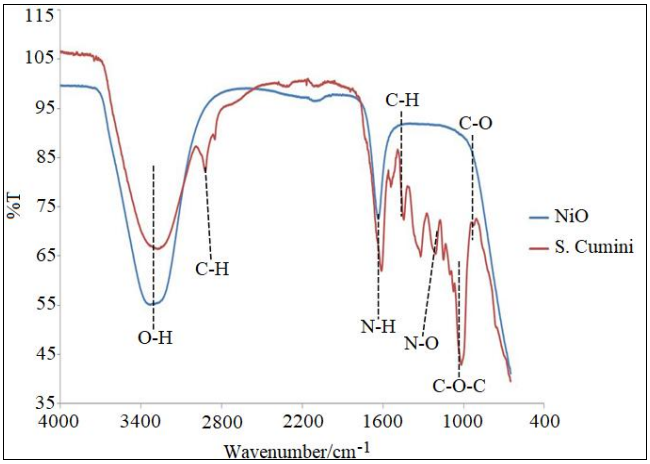

Fig 2. Comparative FTIR spectra of S. cumini and nickel oxide nanoparticles.

The notable peaks are; 3345cm^{-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 -1380cm^{-1}indicate the -OH deformation vibration in the aromatic ring/phenol. The band at 1619cm^{-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 Figure 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].

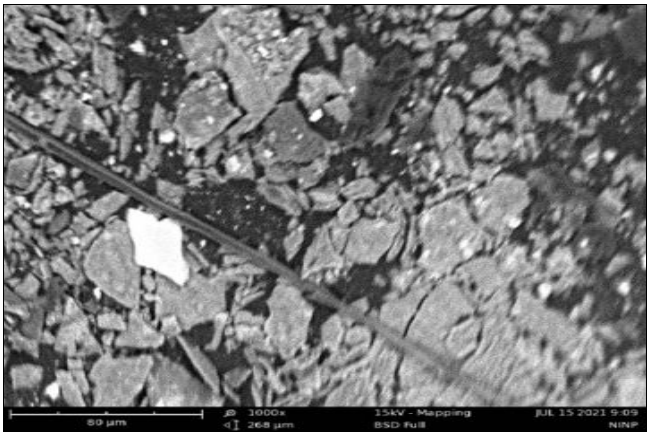

Fig. 1. SEM micrograph of nickel oxide nanoparticles.

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.

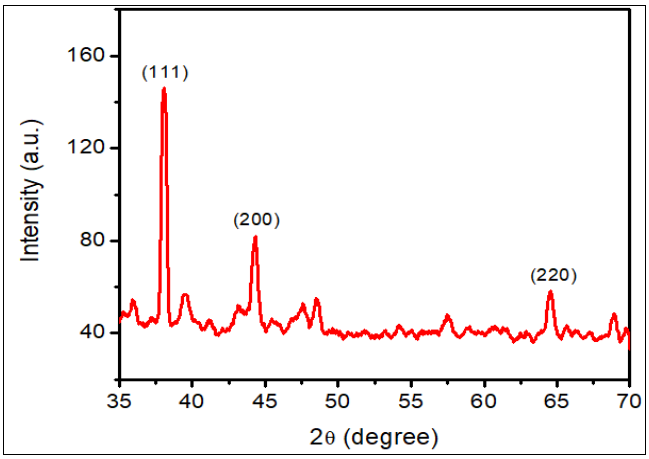

Fig 2: XRD profile of nickel oxide nanoparticle

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.

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

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 θ [1, 2, 3, 4, 5].

g. Cyclic Voltammetry (CV)

Figure 5 compares typical cyclic voltammetric evolutions of bare Pt and Pt/NiO recorded in 0.005 M [Fe $(CN_6)]^{3-}$/ [Fe

$(CN_6)]^{4-}$ solution containing 0.2 M NaOH. It is a well-known phenomenon that $[Fe(CN)_6]^{3-}$/ $[Fe(CN)_6]^{4-}$ is an important redox probe known to exhibit one-electron reversible process. This investigation was carried out in order to ascertain the ability of the modified electrode to permit the transfer of electron of the $[Fe(CN)_6]^{3-}$/ $[Fe(CN)_6]^{4-}$ species to the underlying Pt electrode. In Figure 4.5, the bare Pt electrode (blue line) shows no Faradaic responses while the Pt/NiO modified electrode (red line) shows a Faradaic response at ~420 mV indicating that the NiO is electroactive.

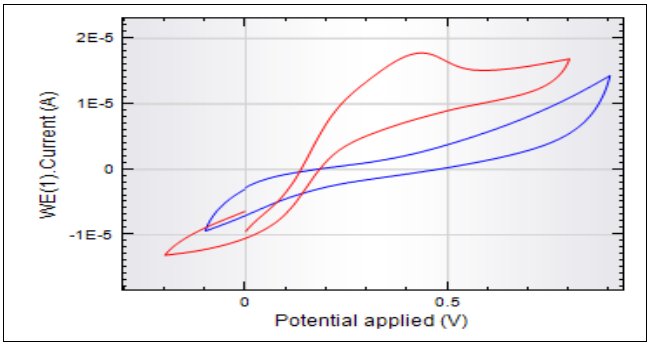

Fig 5: Comparative cyclic voltammetric evolution of bare platinum electrode (blue line) and nickel nanoparticles modified platinum electrode (red line) in 0.005 M $[Fe(CN_6)]^{3-}$/ $[Fe(CN_6)]^{4-}$ solution containing 0.2 M NaOH solution

Electrochemical Impedance Spectroscopy (Eis)

Electrochemical Impedance Spectroscopy (EIS) is a known complementary technique to CV; while CV represents only part of an electrochemical system, the results of the EIS measurements give detail description of the electrochemical system, providing vital information about processes occurring at the electrode/electrolyte interface [16,24]. Figure 6 presents the Nyquist plots for the electrodes studied in 0.2 M NaOH solution containing 0.005 M $[Fe(CN)_6]^{3-}$/ $[Fe(CN)_6]^{4-}$. The impedance spectra of the bare Pt electrode and Pt/NiO electrode were satisfactorily fitted with the proposed equivalent electrical circuits R(RQ)C (Figure 11b) and R(QC[RQ]) R (11c), respectively. The fitting parameters involve the electrolyte resistance (R_s), electron transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and constant phase element (CPE). The platinum bare electrode was fitted with a simple Randles Sevick circuit while the modified Pt/NiO electrode was fitted with a more complex circuit suggesting the modification of the bare electrode and the electroactive nature of the NiO nanoparticles.

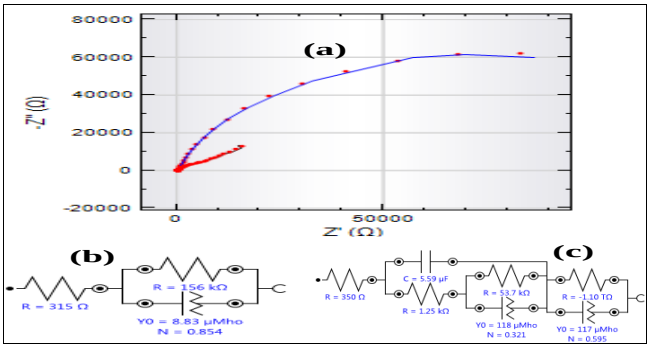

Fig 6: Nyquist plot of bare Pt electrode and Pt/NiONPs electrode (a) in 0.2 M NaOH solution containing 0.005 M $[Fe(CN)_6]^{3-}$/ $[Fe(CN)_6]^{4-}$. Equivalent electrical circuit used in fitting bare Pt electrode (b) and Pt/NiONPs electrode (c)

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. The NiO Nps on a platinum electrode showed faradaic response at 420Mv indicating that is electroactive. The circuit fitting of the modified platinum electrode is more complex compared to the bare platinum electrode showing the electroactive nature of the NiO Nps.

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