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Original Article

BIOSYNTHESIS AND CHARACTERIZATION OF NiO THIN FILM USING LEAF EXTRACTION OF LEEK PLANT BY USING THE SPRAY PYROLYSIS TECHNIQUE

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ABSTRACT

Nickel oxide (NiO) nanoparticles were successfully synthesized by a green method using aqueous extract of Fresh Leek leaves. A simple modified spray pyrolysis method has been employed to deposit a NiO seed layer on a glass substrate using a nickel acetate precursor. The effects of different pH values (9-11) of the growth solution on the properties of NiO nanoparticles (NPs) have been determined. The other characterization techniques were used to study the green synthesis and the effect of pH on the elemental, structural, morphological, and optical properties of the biosynthesized NiO NPs: FTIR and UV-Vis. Results for fresh leaves indicate that the plant is highly promising for the fabrication of NiO NPs. The results show that the pH value has a large and significant effect on the quality, shape, morphologies, structural, functional group, and optical properties of synthesized NiO NPs. The XRD results confirm that the NiO NPs have a cubic crystal structure, the UV-Vis. Results for NiO NPs at different pH values show the absorption peak in the range of (360-370) nm with an energy band gap in the range of (3.35-3.65) eV. Also, the energy-dispersive X-ray (EDX) analysis confirmed that the NPs have high purity and stoichiometry. In addition, the results revealed that plant-assisted spray pyrolysis is a promising, environmentally friendly approach for fabricating high-quality NiO NPs with potential applications across diverse technological arenas.

Keywords: Green synthesis, Modified Spray pyrolysis, NiO NPs, growth process.

1. INTRODUCTION

There is growing interest in the production of nanomaterials due to their unique features absent in bulk materials. The morphology and dimensions of nanoparticles significantly influence nanomaterial properties, making these factors crucial for their applications (Fardood *et al*., 2017). Because nanoparticles are currently used across many fields, such as the chemical, food, health, feed, and cosmetic sectors, their production must be environmentally friendly and sustainable. Besides, metal oxides such as Cu, Au, NiO, Pt, ZnO, and TiO2, nanoparticles can also be produced from other metals (Karam & Abdulrahman, 2022). Among the various biosynthetic methods (enzymes, fungi, bacteria, etc.) that have emerged, the most promising green approach is green synthesis using natural strains and plant extracts. Therefore, due to the novel phytochemicals they synthesized, Nickel Oxide (NiO) nanoparticles (NPs) were derived from various plant parts, including seeds, fruits, leaves, stems, and roots. All plant parts can be harvested naturally, which is a very simple, inexpensive, and environmentally beneficial process that eliminates the need for any intermediate base groups. (Karam & Abdulrahman, 2022).

On the other hand, NiO is a ferromagnetic crystalline solid. NiO has unique electrical, magnetic, and optical properties and has therefore been heavily studied. NiO is a wide band gap (3.6–4.0 eV) p-type semiconductor and is as chemically stable as possible. Due to its low cost and strong ion-storage capacity, it was considered a promising research subject (Bonomo, 2018). Various physical and chemical deposition methods have also been reported for the preparation of NiO thin films, including sol–gel, sputtering, pulsed spray pyrolysis, spin coating, and pulsed laser deposition. Spray pyrolysis (SP) is advantageous due to its low cost, simple process operation, no vacuum requirements, and the ability to produce multistate films (amorphous, crystalline, porous, or compact) with excellent adhesion, enabling large-scale production over large areas (Khizir & Abbas, 2022; Obaida *et al*., 2022). The simplest, non-vacuum, and cost-effective method for preparing thin films at the thin-film scale on a large area is Spray Pyrolysis (Obaida *et al*., 2020). The characteristics of the films prepared by the spray pyrolysis technique mainly depend on parameters like the type of gas (nitrogen or air) used as a carrier, pressure, molarity of precursor solution, deposition rate, substrate temperature (T Sub), time of deposition (t d), nozzle-to-substrate distance, and duration of spraying (Khizir & Abbas, 2022). The peculiarities of the disk form and dimensions are determined by the droplet's momentum and size, as well as the substrate temperature. Thus, the film forms as an overlapped disk of metal salt that converts to oxides upon contact with the substrate (Perednis & Gauckler, 2005). All parameters are kept at optimal values, with only the substrate temperature and deposition duration independently tuned, as they largely affect layer process characteristics (Obaida *et al*., 2022; Obaida *et al*., 2020).

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In this study, NiO nanoparticles were synthesized utilizing the extract of the Fresh Leek plant, nickel acetate as the nickel source, and water as the solvent. The produced NiO nanoparticles on a thin-film glass substrate by spraying a NiO nanoparticle solution and subsequently growing these seed layers in a NiO nanoparticle solution prepared from nickel nitrate, using the same extract without any surfactant or reducing agent, as a cost-effective and environmentally friendly method. Among these methods, spray pyrolysis (SP) offers several advantages: it is a low-cost, easy-to-operate process that allows the deposition of multi-state (amorphous, crystalline, porous/compact) and highly adherent films, and is well scalable to large areas. This, crudely speaking, is one of the most special features of this system: the control of substrate temperature during spraying. For example, this is almost impossible to achieve with systems built using spray pyrolysis technology. The prepared film composition is significantly affected by precursor molarity, substrate temperature, nozzle-to-substrate distance, and spray time. Blow time is the most influential factor in the development of coating properties (Obaida *et al.*, 2022). In recent years, green-chemistry-based nanoparticle synthesis has shown great promise as an affordable, ecologically friendly alternative to chemically produced nanoparticles. Interest in researching the application of novel materials for environmental contamination issues has grown in recent years (Barzinjy & Azeez, 2020; Koteeswari *et al.*, 2022).

NiO NPs have been synthesized using a variety of techniques. The best approach is green synthesis, as it is easy to use, inexpensive, environmentally benign, non-toxic, and offers quick response times. NiO NPs were recently prepared using extracts from various plants (Prabhu *et al.*, 2022). "Specifically, they prepared nickel oxide nanoparticles (NiO NPs) from *Clitoria ternatea* medicinal flower extract and evaluated their antibacterial and photocatalytic dye degradation activities.. Characterization of the synthesized NPs was performed using various techniques, and their efficiency in degrading specific dyes under sunlight and their antibacterial activity against a few bacterial strains were discussed. UV-visible spectroscopy, XRD, FTIR, TEM, and EDX are the important tools used for the analysis (Barzinjy & Azeez, 2020; Prabhu *et al.*, 2022).

The fabrication of NiO nanoparticles by environmentally friendly approaches is attracting recent attention due to their environmentally benign nature and diverse applications. Several studies, including that of Messai *et al* (2023), have also emphasized the significant effect of pH on the size, morphology, and stability of NiO nanoparticles. The study by Messai *et al.* highlights that changes in pH during synthesis dramatically alter particle size, crystal structure, and surface properties. Likewise, Zakaria and Osman (2020) studied the influence of pH on the synthesis of NiO nanoparticles via a sol–gel approach, finding that particle size decreases and structural quality improves as pH increases. Rafique *et al.* (2021) also support this conclusion and demonstrate that pH within an optimal range plays an important role in removing industrial dyes during catalysis, indicating that controlling pH is essential for achieving high-performance materials. Nisah *et al.* (2025) also reported that a specific pH (e.g., 11) led to higher efficiency in NiO synthesis, enhancing the material's performance in energy storage applications. Further, the review by Istrate *et al.* (2025) recently stressed that pH modification is not only a means to manipulate particle size but also to tune the material's antibacterial properties, rendering it an adjustable tool for various industrial applications. All these studies collectively demonstrate that pH adjustment is crucial for optimizing the properties and functionality of NiO NPs in green processing techniques, thereby making it an important parameter for greener AgNPs synthesis.

This study deals with the green synthesis of NiO NPs from fresh Leek plant extract using a modified simple spray pyrolysis method. The NiO seed layer is deposited on a glass substrate using the green technique. The effect of various pH values on the properties of NiO NPs has been systematically studied.

2. MATERIALS AND METHODS

In this study, all the chemical materials used, such as nickel nitrate(II), hexahydrate ($Ni(NO2)_2 \cdot 6H_2O$) and nickel acetate tetrahydrate ($Ni(CH3COO)_2 \cdot 4H_2O$) were obtained from Sigma-Aldrich without further purification.

NiO Seed Layer Preparations:

The microscope glass substrates have been selected for growing NiO NPs. The glass substrates were cleaned for 15 minutes in an ultrasonic bath using ethanol, acetone, and deionized water, and then dried. NiO NPs were prepared from plant leaf extract. Kurdish (Fresh Leek) leaves are available at local food stores throughout Iraq. The plant leaf is cut and added to deionized water, which is then heated to approximately 60 °C for 40 minutes. After the extract is ready, add it gradually to 0.02 mol of nickel acetate solution while stirring to obtain a homogeneous solution. The seed-layer for NiO deposition is prepared, and the steps are shown in Figure 1.

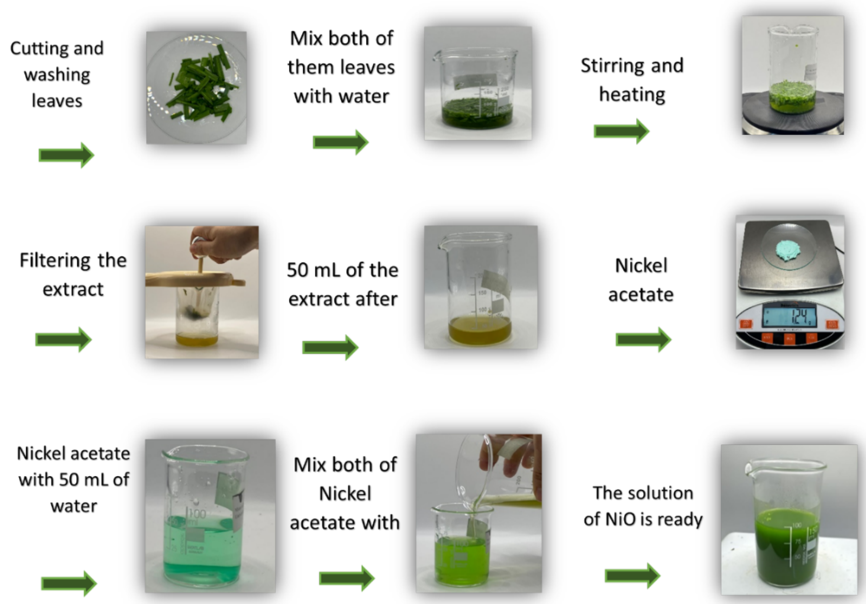

Figure 1: Schematic illustration of the NiO NPs for Biosynthesis by Fresh Leek leaf extract with nickel acetate

Using modified green spray pyrolysis, as shown in Figure 2(a, b), the solution of seed-layer extract was deposited on the glass substrate twice at an angle of 90 degrees for 5 seconds, at a distance of 20 cm. The spray angle (spray cone angle) is the angle formed between the nozzle outlet's centerline and the spray edges on both sides. After the liquid exits the nozzle, it cannot continue in a tangential direction due to the effects of gravitational acceleration and air resistance; therefore, it is done by mopping. The last step is taken before the seed-layer substrate is ready. Please put it in the furnace at 450 °C for about 2 hours.

Growth Green Synthesis Process:

FHigh-quality vertically aligned NiO nanoparticles were synthesized using the growth of NiO NPs of a seed layer, as shown in Figure 3.

Deionized water was used as the solvent, with nickel nitrate as the nickel source, and the extract of the Fresh Leek plant was used to synthesize a nickel oxide nanoparticle solution. Equimolar 1:1 of both $(Ni(NO2)_2 \cdot 6H_2O)$ and the plant extract, we separately dissolved 0.02 mol of $(Ni(NO2)_2 \cdot 6H_2O)$ in deionized water. The dissolved solutions are mixed by using a magnetic stirrer to obtain a homogeneous growth solution. In this study, we investigate the effect of different growth concentrations and PH. The growth pH values used were 9, 10, and 11. The growth Concentration used (0.02). Glass substrates covered with an annealed NiO seed layer were placed within a beaker set at 70 °C. The growing solution was stirred for 4 hours. After the growth period, the produced NiO NPs were washed with deionized water. The final procedure was post-annealing for 2 hours at 450 °C in a furnace: the structures and optical properties of the deposited thin films. FTIR, FESEM, XRD, and UV-Vis were used to analyze the movie.

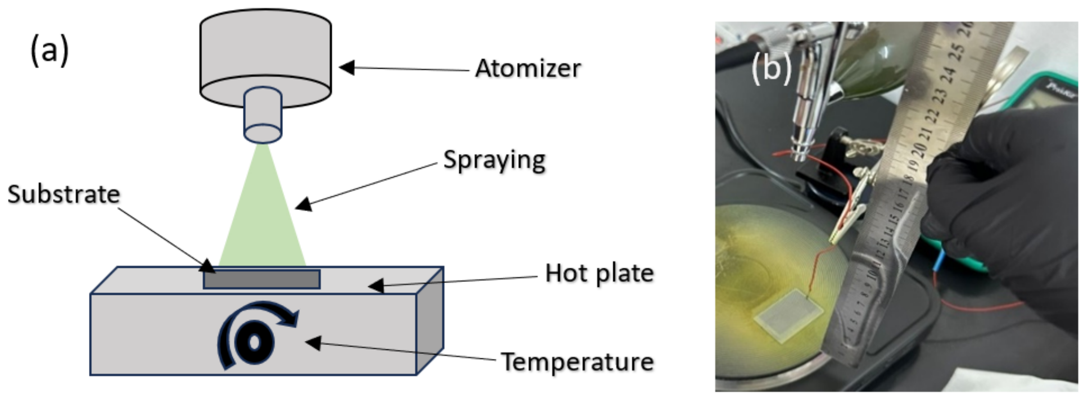

Figure 2: (a)Schematic diagram of spray pyrolysis equipment (b) Spray pyrolysis equipment.

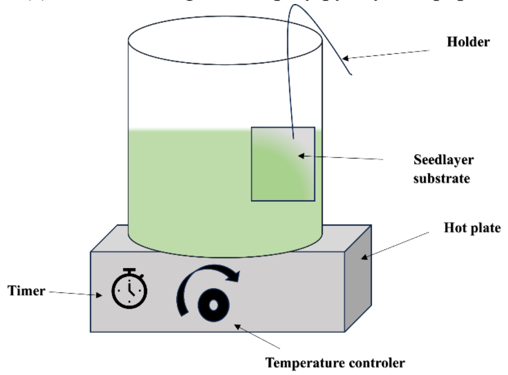

Figure 3: Growth of the seed layer of nickel oxide in nickel oxide solution.

Characterization techniques of NiO Nanoparticles:

Optical properties of the substrate. The fresh Leek extract and as-synthesized NiO NPs glass substrates were analyzed by a Model Lambda 365 Perkin Elmer UV-Visible double-beam spectrophotometer with a Spectrum (190–1100 nm) range, double beam. It was used to study the optical properties of NiO and covers the wavelength range from 230 to 800 nm. The specific functional groups of the fresh Leek extract of plant seed coat layer, as well as NiO NPs fabricated at different pH solutions, were detected and traced out by means of FTIR with Model Spectrum TWO PerkinElmer, Spectral range: 4000–450 cm^{-1}. The size, surface condition, chemical composition, morphologies, and orientation of the prepared NiO NPs were determined by energy-dispersive X-ray spectroscopy (EDX) and field-emission scanning electron microscopy (FE-SEM; ZEISS Sigma VP). The X-ray diffraction (XRD) of the model (2700BH, Co Instrument Haoyuan Company. DX, Ltd) at various pH solutions to define the size and crystal structure, epitaxial growth quality/strain of the synthesized NiO NPs. The scan angle range for CuK radiation (λ = 0.154 nm) was 5–80° (2θ).

3. RESULTS

Characterization of Fresh Leek Leaf Extract:

FTIR spectroscopy is used to study the biomolecules that reduce metal salts to metal nanoparticles and subsequently inhibit their growth by capping them. This is accomplished by recording the FTIR spectra of metal nanoparticles and plant extract. The absorption band that corresponds to the biomolecules that cause bio reduction should only show up in the extract's spectrum; in the NPs' spectrum, it should vanish. This helps both suggest the reaction mechanism and identify the biomolecules that drive bio-reduction. To discover the biomolecules responsible for the bio-reduction of Ni(II) ions into Ni(0) by recording the FTIR spectrum of both leaf extracts of Fresh Leek and Ni NPs (Imran Din & Rani, 2016)The spectrum of FTIR for the Fresh Leek leaf extract is shown in Figure 4.

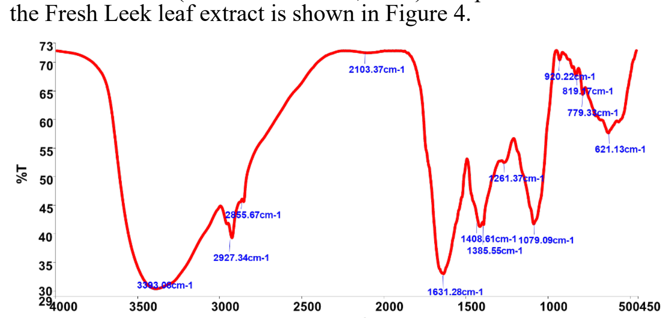

Figure 4: The FTIR Analysis Spectra of the Fresh Leek leaf extract.

The solution's color changed from light green to dark green, as seen in Figure 5, indicating that nickel nitrate was reduced into nickel oxide nanoparticles using the Fresh Leek plant extract. The UV-V spectrophotometer was used to record the absorbance spectra of NiO NPs, as shown in Figure 6. In the case of Fresh Leek extract, the highest and most noticeable peaks in the spectrum are found at 303 nm and 330 nm, respectively.

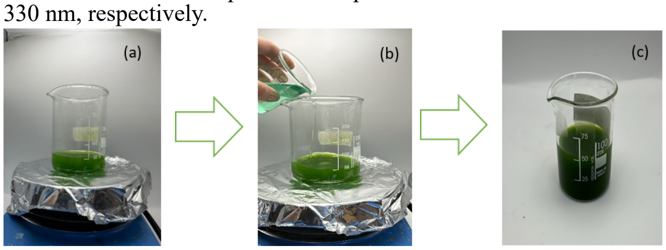

Figure 5: (a)Nickel nitrate, (b) added to Fresh Leek extract, (c)Nickel oxide nanoparticles

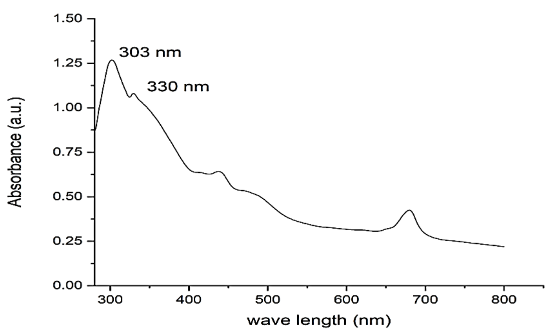

Figure 6: UV-Visible spectrum of the Fresh Leek leaf extract.

Characterization of Nickel Oxide Nanoparticles:

Using the leaf plant extraction method, this study employed various characterization techniques to determine the size, shape, morphology, particle size distribution, crystal structure, chemical composition, optical properties, and energy band gap of NiO NPs on a glass substrate.

The FESEM Analysis:

The top-view morphology, shape, size, orientation, and distribution of biosynthesized growth of NiO NPs on glass substrate (seed layer of NiO) with different PH of the growth solution from nickel nitrate with plant extract, are displayed in Figure 7 (Emission Scanning Electron Microscopy) (FESEM). The NiO NPs prepared from nickel nitrate hexahydrate exhibited a spherical shape, a homogeneous distribution on a glass substrate, and a nano-sized size range. According to FESEM data, the different pHs used altered the nanoparticles' size.

The FTIR Analysis:

The purity and composition of the NiO NPs obtained by biosynthesis are presented in Figure 8. From the above Figure, no distinct peaks were observed in the purity monitoring range for Ni-NPs biosynthesized using Fresh Leek Leaf Extract. The broadband centered at 620 cm^{-1} disappeared on the formation of NiO nanoparticles.

Optical Properties of NiO Nanoparticle Thin Films:

To study the optical characteristics of NiO thin-film nanoparticles prepared from nickel salt, UV-visible spectrometers were used to examine the optical absorption spectra of NiO NPs. This work explores how spray pyrolysis deposition of NiO NPs on a glass substrate affects the optical properties and band gap. The figure illustrates the optical absorption spectra of NiO NPs, fitted using Fresh Leek leaf extract for nickel salts, over a wavelength range of 300 nm to 600 nm, obtained after exposure at various pH levels via spray pyrolysis (Obaida *et al.*, 2022). The UV–vis spectrum in Figure 9 shows an absorption peak around 360-370 nm.

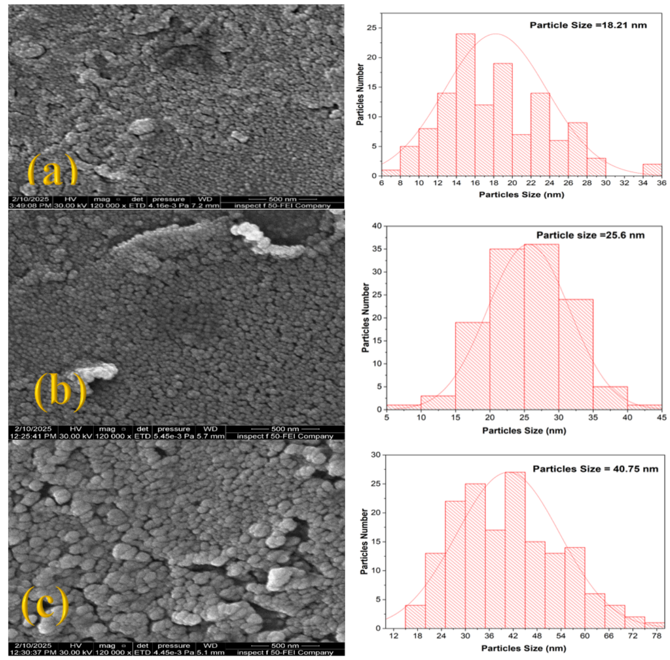

Figure 7: Top View FESEM Images of NiO Nanoparticles (seed layer) deposited on substrates that were synthesized by growing the Green Method using Nickel nitrate with different pH: (a) NiO solution with PH 9, (b) NiO solution with PH 10, and (c) NiO solution with pH 11. Frequency Histogram of NiO NPs Distribution for Different pH.

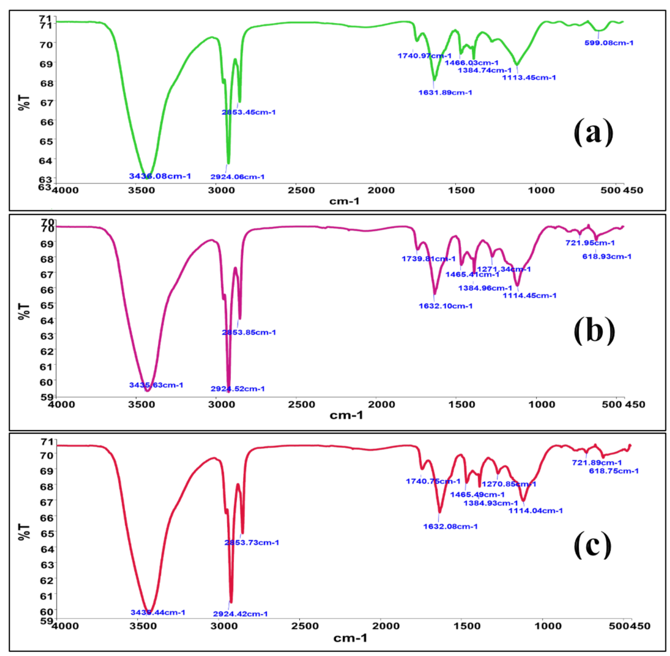

Figure 8: The FTIR Analysis Spectra of the synthesized NiO NPs for different pH: (a) pH 9, (b) pH 10, and (c) pH 11.

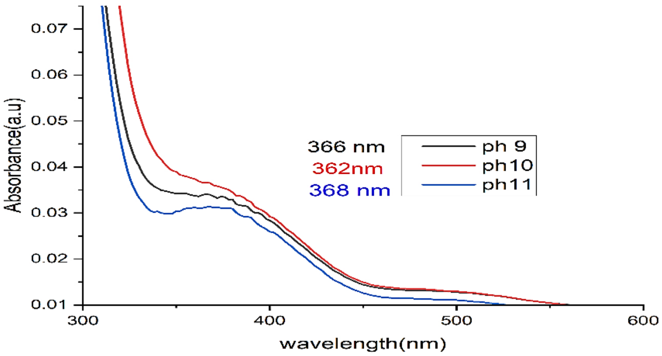

Figure 9: The UV–vis spectroscopy that was obtained reveals an absorption peak for different pH levels.

Spray pyrolysis is a scalable, cost-effective method for creating NiO thin films on glass substrates. It provides excellent control over the substrate temperature, deposition rate, film thickness, and molarity of the precursor solution, all of which can be used to adjust the final film's characteristics (Khizir & Abbas, 2022). The following equation for a direct allowed transition was used to calculate the energy band gaps (E_g) of nanocrystalline NiO from optical absorption spectra (Khizir & Abbas, 2022). The Tauc plot (from UV-Vis absorbance data) is commonly used to estimate the band gap using the equation:

$$\alpha(h\nu) = A(h\nu - E_g)^{1/2} \quad (1)$$

Where α is the absorption coefficient, $h\nu$ is the photon energy, and A is a constant.

The band gap energy was found from the plot of $\alpha(h\nu)^2$ as a function of $(h\nu)$. The linear fit to the linear region on the energy axis $(h\nu)$ extrapolates to an absorption coefficient of zero ($\alpha = 0$). NiO is a highly stable chemical compound, and has a broad band gap of 3.0-4.3eV as a good case of p-type metallic-oxide semiconductors film (Obaida *et al*., 2022). The band gap is slightly higher in nanoparticles synthesized from a plant extract. The blue shift in the band gap energy and the small particle sizes of nanoparticles, as revealed, indicated the occurrence of size quantization effects. As the particle size is scaled down to nanosized, the width of the energy band increases due to a decrease in the overlapping of nearest neighboring energy levels (Selvanathan *et al*., 2021). As presented in Figure 10, the band gap energies are determined, and the values were found to be 3.39 eV, 3.54 eV, and 3.65 eV for different pH values (9,10,11), respectively.

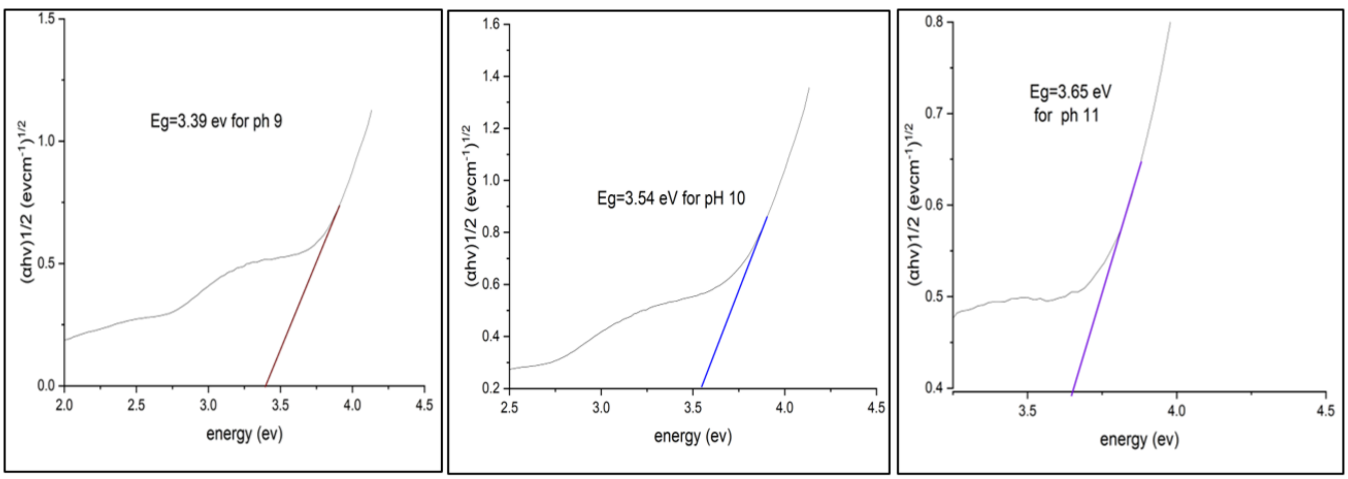

Figure 10: The Tauc-plot Versus Energy Band Gap of biosynthesized NiO NPs by using Leaf extract of Fresh Leek at Different pH:-(9,10,11)

Energy-Dispersive X-Ray Spectroscopy (EDX):

The Fresh Leek plant leaf extract from nickel acetate seed-layer thin films grown in a nickel nitrate solution with varying pH values has been used to study the elemental chemical composition of biosynthesized NiO nanoparticles using EDX spectroscopy. From Figure 11, the EDX technique shows the presence of O and Ni.

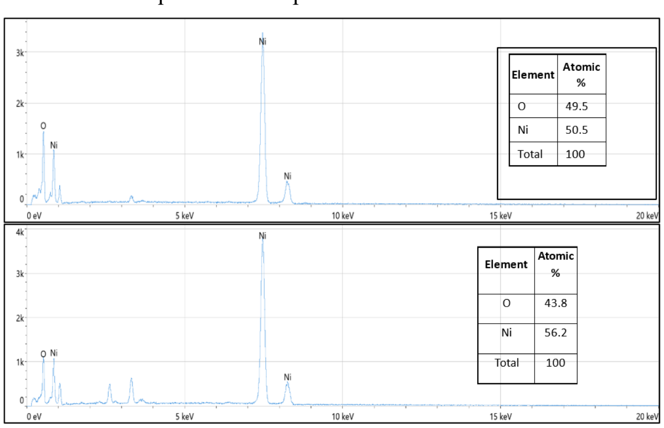

Figure 11: EDX analysis of NiO NRs for pH growth.

X-Ray Diffraction (XRD) Analysis:

The XRD-sprayed NiO samples were prepared to grow in different pH values. The substrates were annealed at 450 °C in the furnace. XRD was used to study the structural information and crystalline size of NiO NP. From Figure 12, diffraction peaks of NiO are found at 2θ values of (37.2, 43.3, 62.9, 75.5, and 79.5) and correspond to the planes (111), (200), (220), (311), and (222), respectively.

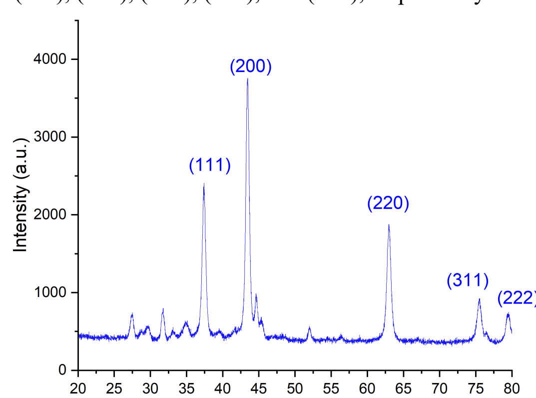

Figure 12: The pattern of XRD of the synthesized NiO NPs.

4. DISCUSSION

From Figure 4, Top View FESEM pictures of NiO NPs (seed layer) Grown on Green Method-Synthesized Substrates as Nickel Nitrate with Various pH, the average particle size of the NiO NPs, as synthesized, was determined from the associated histogram data using a Gaussian distribution. The average particle size of biosynthesized NiO NPs was 18.21 nm, 25.6 nm, and 40.75 nm, respectively, at targeted pH levels of 9, 10, and 11, as indicated by the frequency histogram of NiO NPs distributions. The NiO NPs synthesized from diverse pH levels exhibit homogeneous, high-quality, varied sizes, and uniform spherical shapes. Since electrostatic interactions between particles change as pH rises from 9 to 11, aggregation increases. Weak interactions between particles may be the cause of the apparent aggregations at pH 9. The higher alkalinity may favor the formation of larger clusters by altering the nanoparticles' surface charge and reducing repulsive interactions, thereby explaining the increase in aggregation at higher pH values.

FTIR spectroscopy is used to study the biomolecules that reduce metal salts to metal nanoparticles (NPs) and subsequently inhibit their growth by capping them. This is accomplished by recording the FTIR spectra of metal nanoparticles (NiO NPs) and a plant extract (fresh Leek).

The spectrum of FTIR for the Fresh Leek leaf extract is shown in Figure 5 and contains several peaks visible throughout the entire range. The peak at 3393.08 cm^{-1} is due to the Broad O-H stretch (alcohol and hydroxy compounds). Both peaks are (2927.34 and 2855.67) cm^{-1} Methylene C-H asym, while the triple band region is near 2103.37 cm^{-1}(C≡C stretch). Two absorption bands were detected in 1631.28 cm-1 double bond stretch(amide) carbonyl compound, while 1408.61 cm^{-1} describes the Carboxylate (carbonyl compound). There is a peak around 1385.55 cm^{-1} OH bend (Alcohol) compound. In the aryl-O stretch Ether compound, there is a peak at 1261.37 cm^{-1}. At 1079.09 cm^{-1} , Aliphatic fluoro compounds showed a peak, the C-F stretch of Aliphatic for pH organohalogen compounds. There are four peaks at (920.22,819.17,779.33,621.13) cm^{-1} . The first peak is the Alkyne(C-H) bend, the second and third peaks are noticed as the C-Cl stretch of Aliphatic organohalogen compound, and the last one is at the (S-S stretch) Thiols compounds. These days, researchers use phytochemicals from various plants to biosynthesize nanoparticles, thereby customizing their size and shape. Phytochemicals from the plant extract reduce metal ions to metal nanoparticles. Thus, the plant extract acts as a stabilizing and reducing agent. Due to their antioxidant properties and their freedom from toxic materials, these phytochemicals possess a unique ability to stabilize and minimize metal ions at the nanoscale. Furthermore, they can provide nanoparticles of multiple sizes and shapes (Barzinjy & Azeez, 2020).

FTIR spectra of nickel oxide nanoparticles at different pH levels. Figure 8 displays the composition and purity of the NiO NPs produced during biosynthesis. In three biosynthesized NiO NPs samples, there is a separation of peak broad absorption between 1000 and 4000. To be more specific, the peaks at 3436 cm^{-1} are due to >N-H stretch of Secondary amine: Heterocyclic amine. In addition, two absorption bands were found at 2924.33 and 2853.85. In the three NiO NPs samples synthesized by biosynthesis, there is a series of peaks in absorption between and 4000. More precisely, the peaks that appeared at 3436 are due to Heterocyclic amine, >N-H stretch of Secondary amino. Two absorption bands were found at 2924.33 and 2853.85. To be more precise, three peaks around 1740 cm^{-1} show the Ester of carbonyl compound, while the triple bands nearby 1632 describe the Amide of Carbonyl compound band, in addition, 1466 cm^{-1} . Three peaks describe the C≡C−C Aromatic ring stretch of the Aromatic ring (aryl)bond. However, the three same peaks at 1384 cm^{-1} were termed gem-Dimethyl or "iso"- (doublet) of the Methyl (−CH3) bond. In pH 11 and 9, there were two peaks of bond Aliphatic fluoro compounds, C-F stretch of Aliphatic organohalogen compounds that were (1113 and 1114) cm^{-1} . At 599 cm^{-1} , Aliphatic iodo compounds show a peak; the C-I stretch of Aliphatic for PH is an organohalogen compound. Finally, there are four peaks at (721.95, 721.89, 618.75, 618.93) cm^{-1}. The first and second peaks were noticed as the C-Cl stretch of the Aliphatic organohalogen compound. In contrast, the last two peaks were observed at the C-Br stretch of the Aliphatic organohalogen compound.

Moreover, FTIR at pH 11: the high pH (11) might promote better crystalline order of NiO, but if particle aggregation occurs, as suggested by the FTIR data showing more stable NiO formation, it might indicate better structural alignment and fewer defects at pH 11.XRD at pH 9: XRD analysis often correlates with the degree of crystallinity and particle size. It could be that pH 9 provides a more uniform distribution with less aggregation, which might indicate high-quality crystallinity at this pH despite the absence of the pH-11 advantage. The difference could be attributed to the interaction between the nanoparticle size and morphology rather than just crystallinity.

The Tauc plot (from UV-Vis. absorbance data) is commonly used to estimate the band gap. The Tauc-plot Versus Energy Band Gap of biosynthesized NiO NPs by using Leaf extract of Fresh Leek at Different pH (9,10,11). The band gap energies were determined for the sample oxidized at 400 ˚C for 2 hours (Figure 10). The band gap values were found to be 3.39 eV, 3.54 eV, and 3.65 eV for the samples deposited on substrates grown at pH 9, 10, and 11, respectively. The sample generated at pH 9 had the lowest band gap, 3.39 eV. The enhanced crystal structure of the film is responsible for the low band gap observed in the produced sample at pH 9, and for the optical band gap energy, which was calculated for this spray pyrolysis NiO thin film to be around 3.35-3.7 eV at 400 ˚C. A value in close agreement with 3.65 eV is reported after oxidation at or above 400 ˚C, as described in the literature for NiO films. (Venter & Botha, 2011).

The EDX method shows the presence of O and Ni in Figure 11. The EDX elemental mapping of the Ni and O nanoparticles on the glass substrate for different pH levels, in the absence of any other elements. The NiO NRs have a Ni/O stoichiometry of roughly 1:1. The uniformity of Ni and O presence along the glass substrate is evident. The elemental mapping confirms the formation of high-purity NiO on the glass substrate. The Ni-to-O ratio was nearly identical across all samples.

The XRD was used to examine the crystalline size and structural details of NiO-NP. From Figure 12, diffraction peaks of NiO are found at 2θ values of (37.2, 43.3, 62.9, 75.5, and 79.5) and correspond to the planes (111), (200), (220), (311), and (222), respectively. These numbers closely match the cubic NiO phase reported in reference code 96-101-0382. It can be inferred that the diffraction peaks along the (200) plane are stronger and more prominent. The patterns of the deposited thin films show the crystalline structure of NiO-NP, which is cubic with the space group Fm-3m (COD 96-101-0096), and the obtained XRD results are in good agreement with previous studies (Nashim *et al*., 2024; Wardani *et al*., 2019). The full width at half maximum (FWHM) of an XRD spectrum can be used to characterize the crystal quality and the phase distribution of the NiO NPs formed. The Scherrer equation was used to calculate the average crystallite size from the XRD patterns (Abdulqudos & Abdulrahman, 2022):

$$D = \frac{k\lambda}{\beta cos\theta} \tag{2}$$

where D: Crystallite size (in nm), K: Shape factor (typically 0.9), β: FWHM in radians, θ: Bragg angle (in radians).

Moreover, the dislocation density(L) was determined using the relation below (Abdulqudos & Abdulrahman, 2022):

$$L = \frac{1}{D^2} \tag{3}$$

Where L: Dislocation denisty (in m^{-2}), D: Crystalite sieze (in nm).

The average crystallite sizes along the strong diffraction peaks (111), (200), (220), (311), and (222) for NiO thin films were determined to be 15.2 nm, 1.51 nm, 13.6 nm, 13.4nm, and 14.6 nm, respectively. The obtained XRD results (Table 1), the average crystalline size of 14.3 nm, are in good agreement with previous studies. The Debye Scherrer equation was used to determine that the average crystalline size of NiO-NP was 11.5 nm (Wardani *et al*., 2019).

Table 1: Particle sizes of ZnO nanoparticles before and after calcined at various temperatures, calculated using Debye–Scherrer's equation.

Pos. [°2θ]	d-spacing [Å]	Height [cts]	FWHM Left [°2θ]	Crystallite Size only [Å]	Micro Strain only [%]	Dislocation Density [m^{-2}]
37.069	2.42327	1518.74	0.5055	152	0.79834	4.33×10^{15}
43.1032	2.09697	2721.44	0.494	151	0.69504	4.39×10^{15}
62.7083	1.48041	1245.02	0.5974	136	0.54284	5.41×10^{15}
75.2498	1.26177	437.95	0.6453	134	0.47152	5.57×10^{15}
79.2618	1.20767	341.36	0.5771	146	0.41414	4.69×10^{15}

However, if we look at figures 11 and 12 some prominent noisy peaks in the X-ray diffraction (XRD) reflection profile can be rationalized as due to impurities, amorphous materials, or scattering artifacts so that there are residual compounds left over associated with reactant components or solvents dried incompletely from during synthesis. These peaks may or may not be associated with the material of interest, then become noise in the data. Analysis with Energy-Dispersive X-ray (EDX) suggests that the other elements may be impurities in the synthesis. Green synthesis techniques, for instance those with plant extracts could involve elements from the plant material (e.g., sulfur or potassium) that can be detected by EDX. These can be a harmless level of radiation from another part of the instrument, but which shows up as spurious features in the spectrum. Thus, the noisy peaks (in XRD) and extra elements (in EDX) originate from impurities or complicated synthesis method, further purifying may solve this problem.

5. CONCLUSION

In the present study, NiO nanoparticles were successfully prepared by a clean, inexpensive, and environmentally friendly method using an aqueous extract of Fresh Leek leaves as both reducing and stabilizing reagents. The nanoparticles were directly grown on glass substrates using a modified spray pyrolysis method, and further growth in nickel nitrate solutions with different pH (9, 10, and 11). The microstructure, morphology, optical property, and composition of the NiO nanoparticles were extensively characterized by FTIR, FESEM, XRD, and UV-Vis spectroscopy, and EDX analysis.

The results showed that the pH of the growth solution plays a crucial role in controlling the size, distribution, and optical properties of NiO nanoparticles. FESEM images showed well-dispersed spherical nanoparticles with average particle sizes of 18.21 nm, 25.6 nm, and 40.75 nm at pH 9, 10, and 11, respectively. XRD analysis confirmed the formation of a cubic crystalline structure with an average crystallite size of approximately 14.3 nm, consistent with previous reports. FTIR spectra indicated the involvement of phytochemicals from the leek extract in the reduction and stabilization processes.

The UV-Vis spectroscopy showed absorption peaks between 360–370 nm, and Tauc plot analysis revealed band gap energies of 3.39 eV, 3.54 eV, and 3.65 eV for samples grown at pH 9, 10, and 11, respectively. These values are in close agreement with the literature and confirm the quantum confinement effect in nanoscale NiO. EDX analysis confirmed the high purity and stoichiometric composition (Ni:O ≈ 1:1) of the synthesized nanoparticles. In conclusion, the green synthesis method using Fresh Leek leaf extract combined with modified spray pyrolysis offers a sustainable, efficient, and scalable route for producing high-quality NiO nanoparticles with tunable properties, suitable for various applications in catalysis, optoelectronics, and energy storage.

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This article does not include any studies involving human participants or animals performed by the authors; therefore, ethical approval was not required.

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Author Contributions:

L.N.A. performed formal analysis and was responsible for writing the original draft, as well as review and editing. A.F.A. contributed to the conceptualization and visualization of the study, and was responsible for supervision, resources, data curation, statistics, validation, and software. Both authors have read and agreed to the published version of the manuscript.

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