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GREEN SYNTHESIS AND CHARACTERIZATION OF NICKEL OXIDE NANOPARTICLES USING SALVIA OFFICINALIS LEAF EXTRACT AND EVALUATION OF THEIR CATALYTIC ACTIVITY IN HYDROGEN PEROXIDE DECOMPOSITION

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ABSTRACT

This study aims to synthesize nickel oxide nanoparticles (NiO NPs) via a green method using aqueous *Salvia officinalis* (sage) leaf extract as a natural reducing and capping agent without the addition of any toxic chemicals. The synthesized nanoparticles were characterized using Fourier Transform Infrared Spectroscopy (FTIR) to identify the functional groups involved in the reduction and stabilization processes. The FTIR spectra showed characteristic peaks at 450.3 cm^{-1} and 418.5 cm^{-1}, which are attributed to Ni-O bond vibrations, confirming the successful formation of NiO nanoparticles capped with plant phytochemicals. Furthermore, the catalytic activity of the prepared nanoparticles was evaluated in the decomposition of hydrogen peroxide (H_2O_2). The results showed that the green NiO NPs exhibited outstanding catalytic performance, with the decomposition efficiency progressively increasing over time to reach a maximum of 61.11% after 60 minutes. Kinetic modeling revealed that the catalytic degradation strictly followed pseudo-first-order kinetics, with a calculated reaction rate constant (k) of 0.014 min^{-1} and a strong coefficient of determination (R^2 = 0.953). The findings indicate that sage extract provides an effective, low-cost, and sustainable route for the synthesis of NiO nanoparticles with excellent catalytic activity, making them promising candidates for future environmental and catalytic applications.

KEYWORDS: Green synthesis; Nickel oxide nanoparticles; *Salvia officinalis*; Pseudo-first-order kinetics; Hydrogen peroxide decomposition.

1. INTRODUCTION

In the last decade, nanoparticles have gained significant importance due to their unique physical and chemical properties resulting from their small size (approximately 1 to 100 nm) and their high surface area-to-volume ratio. This has made them effective tools in numerous fields. In environmental applications, they have become essential materials for the treatment of water contaminated with heavy metal ions, the degradation of dyes, and the removal of organic pollutants from water. In the biomedical field, numerous studies have investigated their ability to interact with cells and biomolecules, and their capability to damage bacterial cell walls has been confirmed, thereby inhibiting bacterial growth and antibiotic resistance.[1,5]

However, conventional methods for nanoparticle synthesis rely on toxic and expensive solvents and reducing agents, which limits their biological applications and increases their environmental and toxicological risks. Therefore, scientific research has shifted toward “green chemistry” as a sustainable and environmentally friendly alternative. This approach utilizes plant extracts and microorganisms as natural reducing and capping agents. Such a method minimizes the use of toxic chemicals, reduces pollution, prevents nanoparticle agglomeration, and enhances biocompatibility, making it a safe and effective choice for future applications.[6]

Plant extracts represent one of the best approaches for the green synthesis of nanoparticles, as they serve as

natural sources of reducing and stabilizing agents. This provides a safe and eco-friendly synthesis route that reduces the possibility of contamination and accelerates the reaction, while also helping to maintain the stability of the nanoparticles. This is attributed to the high content of active compounds such as phenols and flavonoids in these extracts.[7,8]

Nanoparticles are generally classified into two main types: organic and inorganic. Inorganic nanoparticles are further divided into magnetic nanoparticles (such as cobalt, iron, and nickel), noble metal nanoparticles (such as gold and silver), and semiconductors (such as nickel oxide and zinc oxide). Metal oxide nanoparticles, in particular, have attracted extensive research interest due to their combination of high catalytic activity, chemical and thermal stability, distinctive electromagnetic properties, and low toxicity.[9]

Based on the above, nickel oxide (NiO) was selected for this study. In addition to the aforementioned properties, NiO possesses a wide band gap of 3.6–4.0 eV, which enables its use in a broad range of applications including catalysis, gas sensing, and energy storage.

For the synthesis of NiO nanoparticles, sage (*Salvia officinalis*) was chosen due to its richness in phenolic compounds such as rosmarinic acid, flavonoids, and phenolic diterpenes. These compounds act as effective natural reducing and capping agents in the green synthesis of nanoparticles.

The aim of this study is to synthesize and characterize nickel oxide nanoparticles using sage leaf extract as a reducing and stabilizing agent, and to evaluate their potential applications in the catalytic decomposition of hydrogen peroxide H_2O_2 via volumetric titration.[10]

2. MATERIALS AND METHODS

2-1- Preparation of sage leaves extract

Sage leaves (Salvia officinalis) were collected from Salanfa – Syria and washed thoroughly with distilled water to remove dust and impurities. The leaves were dried in the shade at room temperature for 7 days.

After drying, the leaves were ground to obtain a fine powder. Then, 10 g of sage leaf powder was weighed and added to 100 mL of distilled water in a glass beaker. The mixture was heated on a magnetic stirrer with continuous stirring at 50 °C for 2.5 hours.

After that, the extract was cooled to room temperature and filtered using Whatman No.1 filter paper to obtain a clear extract free of impurities. The aqueous extract was stored in the refrigerator at 4 °C and used within one week of preparation.

2-2- Preparation of nickel nitrate solution

A 0.2 M solution of nickel(II) nitrate hexahydrate, $Ni(NO_3)_2 \cdot 6H_2O$, was prepared by dissolving 2.91 g of the salt in distilled water and transferring it to a 50 mL volumetric flask. The volume was then made up to the mark with distilled water. The solution was stirred using a magnetic stirrer until complete dissolution was achieved, resulting in a clear light green solution. The solution was stored at room temperature until use.

2-3- Green synthesis of nickel oxide nanoparticles (NiO-NPs)

50 mL of the previously prepared aqueous sage leaf extract was added dropwise to 50 mL of 0.2 M nickel nitrate solution under continuous stirring at room temperature. Subsequently, the mixture was heated on a magnetic stirrer at 60 °C for 1 hour. A color change from light green to dark brown was observed, indicating the reduction of nickel ions and the formation of nanoparticles.

After the reaction was completed, the pH of the solution was adjusted to 11 using a 0.2 M sodium hydroxide (NaOH) solution to precipitate the nanoparticles. The mixture was then left to age for 24 hours. The precipitate was separated by centrifugation at 4000 rpm for 10 minutes. The obtained precipitate was washed several times with distilled water and ethanol to remove impurities, and then dried in an oven at 60 °C for 12 hours.

Finally, the dried powder was calcined in a furnace at 450 °C for 2 hours to obtain black nickel oxide nanoparticles (NiO-NPs).

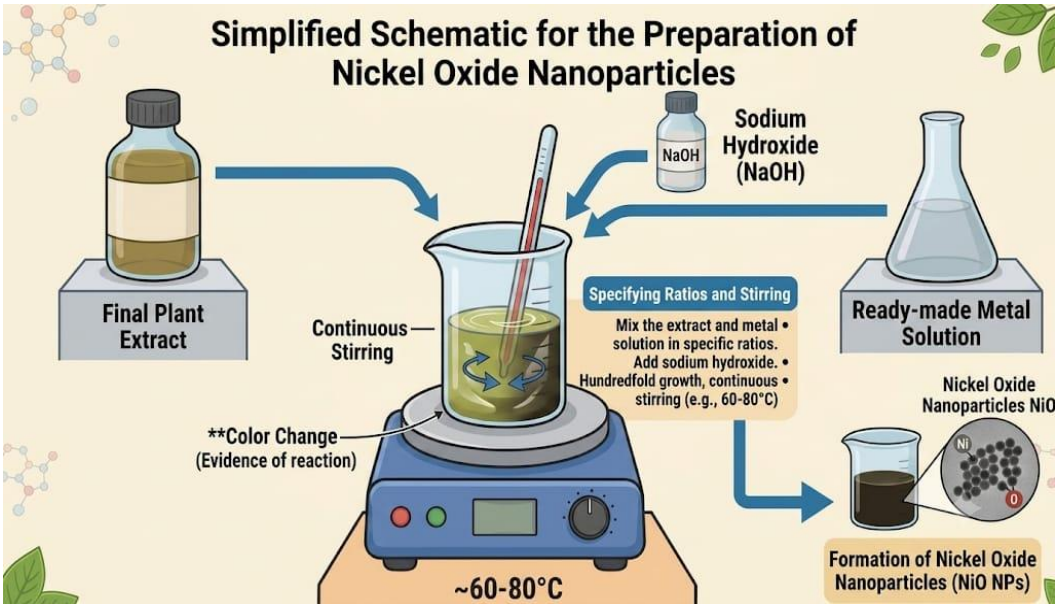


Figure 1. Simplified schematic representation for the green synthesis of nickel oxide nanoparticles (NiO NPs).

Figure 2. As-prepared green-synthesized nickel oxide nanoparticles (NiO NPs).

3. FTIR analysis

The FTIR spectrum of the green synthesized NiO nanoparticles using *Salvia officinalis* leaf extract is shown in Figure 3. The spectrum exhibits several characteristic absorption bands corresponding to both the Ni-O lattice vibrations and the phytochemicals present in the sage extract that act as reducing and capping agents.

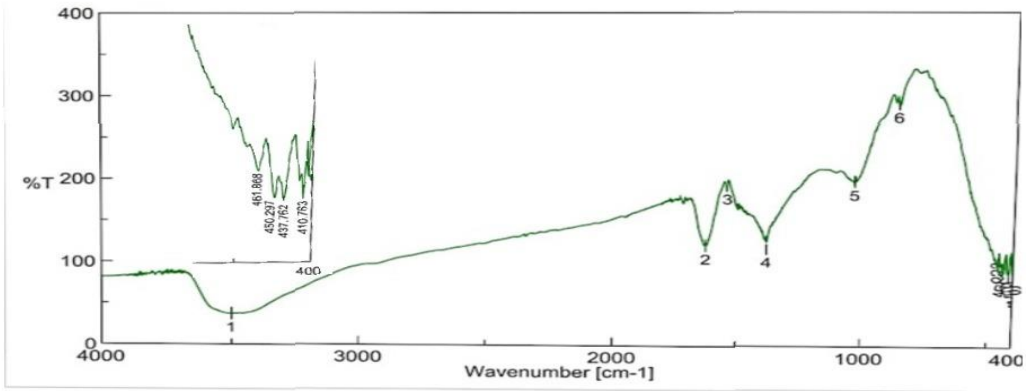
A broad and intense absorption band observed at 3458.3 cm^{-1} is attributed to O-H stretching vibrations of hydroxyl groups due to adsorbed water molecules on the surface of nanoparticles and phenolic compounds from the sage extract. The peaks at 2927.4 and 2855 cm^{-1} correspond to the symmetric and asymmetric stretching vibrations of aliphatic C-H groups of CH_2 and CH_3 present in the organic compounds of the plant extract.

The peak at 1732 cm^{-1} is assigned to the C=O stretching vibration of carbonyl groups present in terpenoids and organic acids of the sage extract. The band at 1624.7 cm^{-1} can be attributed to the H-O-H bending vibration of adsorbed water or to C=C stretching in aromatic rings of polyphenols.

The absorption bands in the range 1161.7–1060.3 cm^{-1} are due to C-O stretching vibrations of alcohols, ethers, and polyphenols, confirming the presence of biomolecules from the plant extract on the surface of NiO NPs.

Most importantly, the characteristic peaks observed at 450.3 and 418.5 cm^{-1} in the low-frequency region are assigned to Ni-O stretching vibrations.[11,12] confirming the successful formation of nickel oxide nanoparticles. The slight shift and splitting of the Ni-O band is likely due to surface effects and interactions with the capping biomolecules.

In conclusion, the FTIR results confirm the formation of NiO nanoparticles and indicate that phytochemicals from *Salvia officinalis* extract played a dual role as reducing and stabilizing agents during the green synthesis process.



Result of Peak Picking

No.	Position	Intensity	No.	Position	Intensity
1	3496.31	37.5359	3	1634.73	121.273
2	1542.77	193.376	4	1384.64	117.71
5	1033.49	199.057	8	861.68	291.164
7	473.439	108.866	9	461.868	100.403
9	450.297	91.9982	10	437.762	83.3338
11	410.477	79.7243	12	410.763	85.3338

[Comment]
Sample Name
Comment
User
Division
Company sharaf

[Measurement Information]
Model Name FTIR-4100typeA
Serial Number R025561016

Light Source Standard
Detector TGS

Figure 3. FTIR spectrum of the green-synthesized nickel oxide nanoparticles (NiO NPs).

4. Hydrogen peroxide decomposition via NiO NPs nanocatalyst

The enhanced catalytic performance of the green-synthesized NiO nanoparticles toward H_2O_2 decomposition is directly correlated with their surface properties identified by FTIR. The distinct vibrational band of Ni-O stretching (around 450 cm^{-1}) confirms the formation of active crystalline nano-surfaces with abundant catalytic sites. Additionally, the presence of plant-derived functional groups (such as -OH and C=O) on the NiO surface acts as a promoter. These groups facilitate the adsorption of H_2O_2 molecules via hydrogen bonding, thereby weakening the O-O bond of hydrogen peroxide and accelerating its kinetic decomposition, which was systematically monitored using the potassium permanganate titration method.

The catalytic decomposition of hydrogen peroxide (H_2O_2) is a standard model reaction to assess the catalytic activity of green-synthesized nickel oxide nanoparticles (NiO NPs). While H_2O_2 decomposes spontaneously at a negligible rate under ambient conditions, the addition of NiO NPs significantly enhances the reaction rate by lowering the activation energy. The overall decomposition reaction is given by:

$2H_2O_2 \text{—(NiO NPs)} \rightarrow 2H_2O + O_2\uparrow$

The reaction kinetics were monitored using a permanganometric titration method. At specific time intervals (e.g., every 5 min), an aliquot was withdrawn from the reaction mixture and the catalytic reaction was immediately quenched by adding it to a known volume of dilute sulfuric acid (H_2SO_4). The acidic medium serves to both terminate the catalytic reaction and provide the required H^+ ions for titration.

The residual concentration of H_2O_2 in each aliquot was then determined by titration against a standardized potassium permanganate ($KMnO_4$) solution. Potassium permanganate acts as a self-indicator, where the purple MnO_4^- ions are reduced to colorless Mn^{2+} ions by H_2O_2 in acidic medium, according to the following reaction:

$5H_2O_2 + 2MnO_4^- + 6H^+ \rightarrow 2Mn^{2+} + 8H_2O + 5O_2\uparrow$

The titration endpoint was marked by the appearance of a persistent faint pink color. The concentration of H_2O_2 at each time point was calculated from the titration data and used to evaluate the catalytic performance of the NiO NPs.

4-1- Catalytic activity evaluation for hydrogen peroxide decomposition

The catalytic activity of the green-synthesized nickel oxide nanoparticles (NiO NPs) was evaluated using the decomposition reaction of hydrogen peroxide (H_2O_2)

Preparation of solutions

1. Hydrogen peroxide solution: 0.12 g of commercial H_2O_2 was diluted to 100 mL with distilled water. Subsequently, 10 mL of this solution was further diluted to 100 mL with distilled water to obtain the working solution at a lower concentration.

2. Potassium permanganate solution: A 0.005 N $KMnO_4$ solution was prepared by dissolving 0.015 g of potassium permanganate in 100 mL of distilled water.

3. Sulfuric acid solution: A 1.0 M H_2SO_4 solution was prepared to be used for quenching the reaction and providing the acidic medium required for titration.

Catalytic procedure

90 mL of the prepared H_2O_2 solution was placed in a conical flask, and 0.02 g of NiO NPs was added with continuous stirring at room temperature to initiate the catalytic reaction.

To monitor the reaction kinetics, 10 mL aliquots were withdrawn at specific time intervals: 5, 10, 15, 20, 25, 30, 45, and 60 minutes. Each aliquot was immediately transferred into a flask containing 1.0 M H_2SO_4 to quench the reaction instantly.

Each sample was then titrated against the standardized 0.005 N $KMnO_4$ solution using the self-indicator endpoint, indicated by the appearance of a persistent faint pink color.

For comparison, a blank sample consisting of 10 mL of H_2O_2 solution before catalyst addition was titrated, which consumed 3.6 mL of $KMnO_4$ solution.

Figure 4. Color change of the reaction mixture at the endpoint of KMnO4 titration.

4-3- Kinetic evaluation and catalytic efficiency of NiO nanoparticles

Based on the experimental data presented in Table 1, the catalytic efficiency of the nickel oxide nanoparticles was evaluated by monitoring the rate of decrease in hydrogen peroxide H_2O_2 concentration as a function of time. Since the balanced redox reaction between potassium permanganate and hydrogen peroxide in an acidic medium follows a 2:5 molar ratio, as shown in the equation below, the consumed titration volumes of $KMnO_4$ were utilized to calculate the residual concentration of H_2O_2 at each time interval, followed by the determination of the kinetic decomposition constants:

Main titration equation

$5H_2O_2 + 2MnO_4^- + 6H^+ \rightarrow 2Mn^{2+} + 8H_2O + 5O_2\uparrow$

Equation for calculating the residual hydrogen peroxide concentration (Ct)

$Ct = [H_2O_2]t = (5 \times M_{KMnO4} \times V_{KMnO4}) / (2 \times V_{H2O2})$

Where

Ct: the remaining concentration of H_2O_2 at time (t) in mol/L.

M_{KMnO4}: the molar concentration of the permanganate solution in the burette (0.001 M in this study).

V_{KMnO4}: the consumed volume of permanganate at time (t).

V_{H2O2}: the volume of the hydrogen peroxide aliquot taken for titration (10 mL).

Table 1: Kinetic, molar concentrations, and catalytic efficiency of H2O2 decomposition over green NiO nanoparticles.

T (min)	V_{KMnO4} (mL)	C_t (mol/L)	Catalytic efficiency (%)	Ln[C_t]
0	3.6	0.0009	0.00%	-7.01
5	2.8	0.0007	22.22%	-7.26
10	2.7	0.00068	25.00%	-7.30
15	2.6	0.00065	27.78%	-7.34
20	2.6	0.00065	27.78%	-7.34
25	2.5	0.00063	30.56%	-7.38
30	2.1	0.00053	41.67%	-7.55
45	1.8	0.00045	50.00%	-7.71
60	1.4	0.00035	61.11%	-7.96

The calculation results presented in Table 1 clearly demonstrate the outstanding catalytic performance of the green-synthesized NiO nanoparticles in accelerating the decomposition of hydrogen peroxide. At the initial stage of the reaction (t = 0), the molar concentration of H_2O_2 was at its peak (0.00090 M). Upon the addition of the nanocatalyst, a progressive decline in the residual concentration was observed, reaching 0.00035 M after 60 minutes of reaction time. Concurrently, the catalytic efficiency exhibited a sharp upward trend, evolving from 0.00% at the start of the process to 61.11% by the end of the hour. This significant consumption of H_2O_2 over a relatively short period confirms the presence of highly active catalytic sites on the surface of the green nanoparticles. To further explore the degradation behavior and establish the precise reaction mechanism, these experimental data were systematically fitted into kinetic models, as visually represented in the following figures.

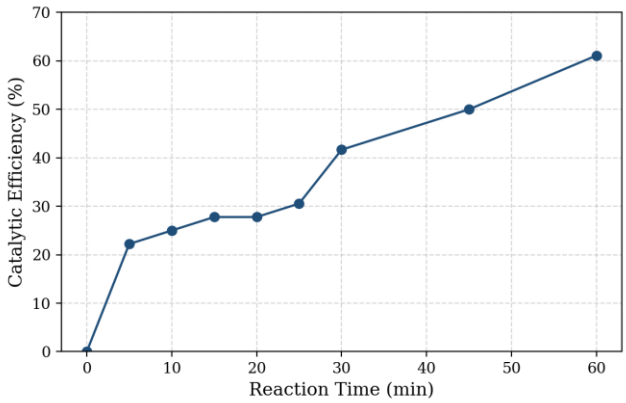


Figure 5. Catalytic efficiency (%) of H2O2 decomposition over green NiO nanoparticles as a function of reaction time.

This plot (Figure 5) illustrates the variation in the catalytic efficiency of the green-synthesized NiO nanoparticles as a function of reaction time. The curve exhibits a progressive and proportional increase in decomposition efficiency over time, initiating from 0% at the baseline (t = 0) and escalating to reach 61.11% after 60 minutes. This active catalytic behavior is attributed to the abundance of highly active surface sites on the green nanoparticles, which facilitate the rapid adsorption of hydrogen peroxide molecules and weaken their chemical bonds to accelerate their decomposition.

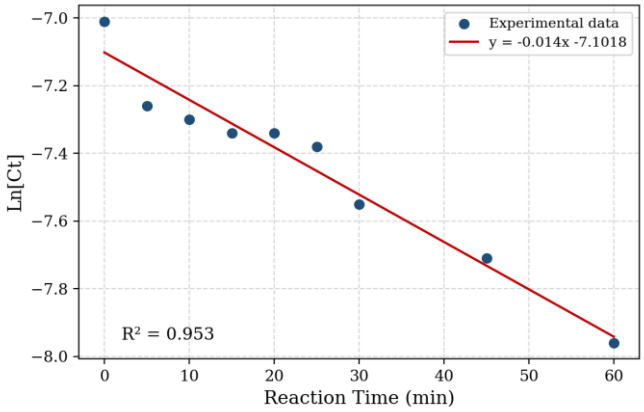


Figure 6. Pseudo-first-order kinetic linear plot for the decomposition of H2O2 catalyzed by green-synthesized NiO nanoparticles.

This plot (Figure 6) represents the kinetic study of hydrogen peroxide decomposition, where the natural logarithm of the residual molar concentration (Ln[Ct]) is plotted as a function of reaction time. The graph displays a well-defined linear relationship with a negative slope and a strong coefficient of determination (R^2 = 0.953), precisely proving that the decomposition reaction catalyzed by the green NiO nanoparticles follows pseudo-first-order kinetics.[13] From the slope of the linear regression equation (y = -0.014x - 7.1018), the reaction rate constant (k) was calculated to be 0.014 min^{-1}. This value clearly reflects the superior efficiency of the nanocatalyst in accelerating electron transfer and weakening the chemical bonds of the reactant.

Table 2: Comparative catalytic performance of green-synthesized NiO nanoparticles against reported literature.

Plant Extract Source	Catalyst Dosage (g)	Target / Reaction	Decomposition / Efficiency (%)	(k, min^{-1})	Ref
Calpurnia aurea	0.05	Malachite Green / Photocatalysis	~90% (in 120 min)	0.009	[9]
Aegle marmelos	0.03	Organic Pollutant Degradation	~58% (in 60 min)	0.011	[11]
Salvia officinalis (Sage)	**0.02**	**H2O2 Catalytic Decomposition**	**61.11% (in 60 min)**	**0.014**	**This Work**

As presented in Table 2, the green NiO NPs synthesized via *Salvia officinalis* exhibit a superior kinetic rate constant (k = 0.014 min-1) at a relatively lower catalyst dosage (0.02 g) compared to other recent bio-synthesized analogs. This confirms the enhanced performance of the active crystalline nano-surfaces, which are effectively promoted by the sage plant's phytochemical capping groups.

5. CONCLUSION

In this study, nickel oxide nanoparticles (NiO NPs) were successfully synthesized via an eco-friendly and cost-effective green method using the aqueous leaf extract of *Salvia officinalis* (sage) as a sustainable reducing and capping agent. The successful formation of NiO nanoparticles and the capping role of plant phytochemicals were firmly confirmed by FTIR analysis**.** Crucially, the green-synthesized NiO NPs demonstrated outstanding catalytic performance in the decomposition of hydrogen peroxide (H2O2). The catalytic process was systematically monitored using a precise potassium permanganate titration method, revealing that the nanocatalyst achieved a maximum decomposition efficiency of 61.11% within 60 minutes. Kinetic modeling showed that the catalytic degradation strictly followed pseudo-first-order kinetics with a calculated rate constant (k) of 0.014 min-1 and a strong correlation coefficient (R2 = 0.953). These findings highlight that sage extract offers an efficient, green, and viable pathway to produce highly active metal oxide nanocatalysts suitable for environmental and catalytic applications.

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