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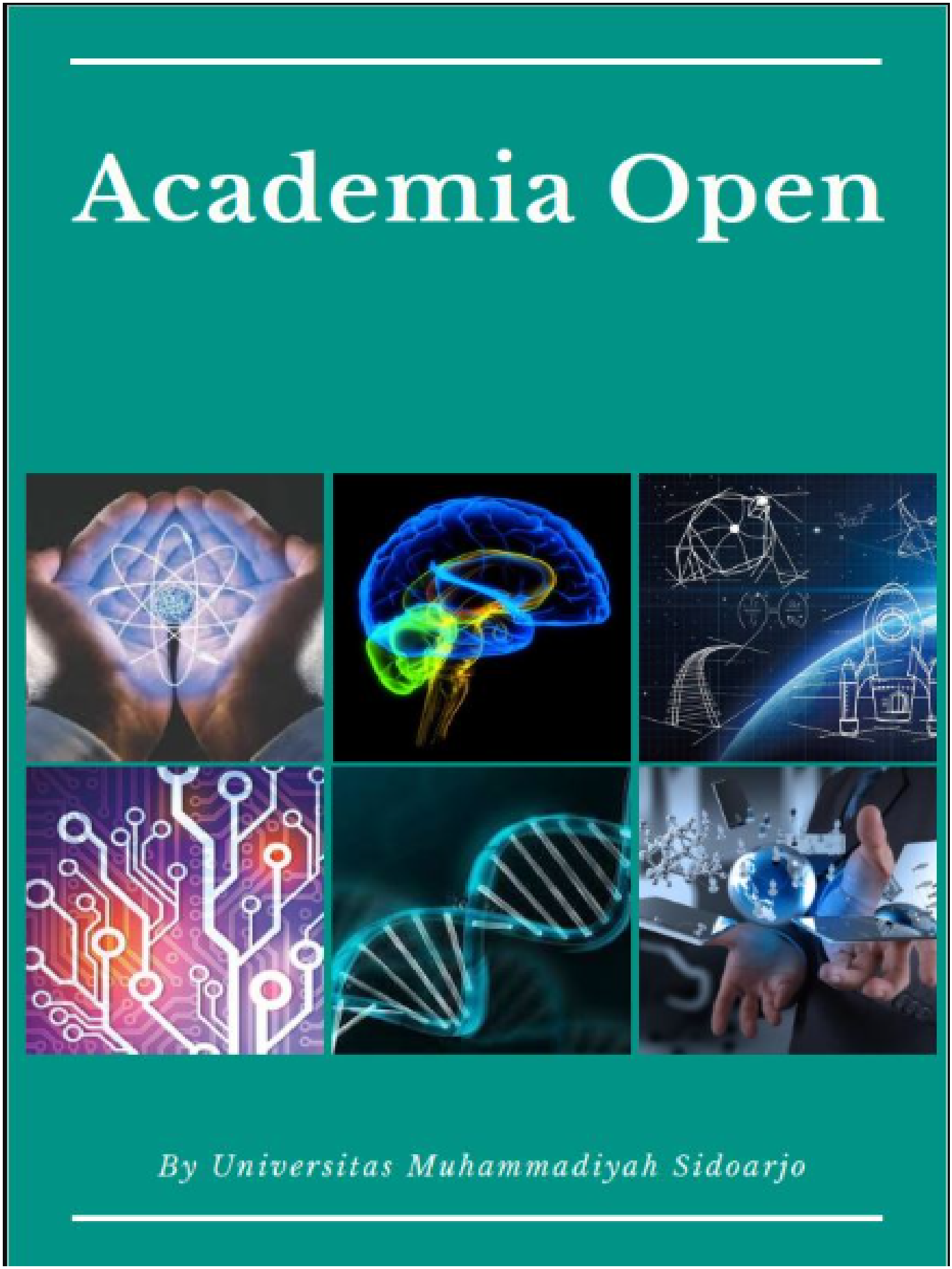
Vol. 11 No. 1 (2026): June
DOI: 10.21070/acopen.11.2026.13951



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Academia Open

Vol. 11 No. 1 (2026): June
DOI: 10.21070/acopen.11.2026.13951

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Academia Open

Vol. 11 No. 1 (2026): June
DOI: 10.21070/acopen.11.2026.13951

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Vol. 11 No. 1 (2026): June
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Academia Open

Vol. 11 No. 1 (2026): June
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Eco-Friendly Synthesis of Magnesium Oxide Nanoparticles Using Eggplant (Solanum melongena) Extract: Structural and Morphological Characterization: Sintesis Ramah Lingkungan Partikel Nano Oksida Magnesium Menggunakan Ekstrak Terong (Solanum melongena): Karakterisasi Struktur dan Morfologi

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Abstract

General Background: Nanotechnology enables the design of advanced materials with unique physicochemical properties at the nanoscale. **Specific Background:** Magnesium oxide nanoparticles are widely studied due to their high stability, biocompatibility, and applicability in catalysis, environmental remediation, and antimicrobial systems. **Knowledge Gap:** Conventional synthesis methods often involve toxic chemicals and complex processing, while systematic studies using eggplant extract remain limited. **Aims:** This study aims to synthesize MgO nanoparticles using aqueous eggplant extract as a bio-reducing and stabilizing agent and to evaluate their structural and morphological properties. **Results:** XRD confirmed phase-pure cubic MgO with a rock salt structure and an average crystallite size of 18.56 nm. FESEM analysis showed quasi-spherical morphology with an average particle size of 23.14 nm, while EDX verified stoichiometric Mg and O composition with high purity. FTIR identified characteristic Mg–O vibrations and residual phytochemical functional groups. **Novelty:** The study demonstrates the use of eggplant extract as a sustainable bio-resource for producing well-crystallized MgO nanoparticles. **Implications:** The synthesized nanoparticles exhibit nanoscale dimensions and structural purity suitable for applications in catalysis, environmental remediation, and antimicrobial systems.

Keywords: Magnesium Oxide Nanoparticles, Green Synthesis, Eggplant Extract, Structural Characterization, Nanomaterials

Key Findings Highlights

1. Phase-pure cubic structure verified through diffraction analysis
2. Biogenic synthesis produced uniform nanoscale morphology
3. Elemental composition confirms high chemical purity

Published date: 2026-03-26

Academia Open
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DOI: 10.21070/acopen.11.2026.13951

1. Introduction

Nanotechnology has also become one of the most revolutionary areas in modern materials science and engineering, which provide new opportunities to manipulate matter at an atomic and molecular level to design materials with specific physicochemical characteristics. Metallox nanoparticles are one of the most extensive groups of nanomaterials that have received unprecedented scientific and technological attention due to their optical, electronic, magnetic, catalytic, and antibacterial unique attributes, unlike their bulk counterparts [1]. These characteristic features are mainly imposed by quantum confinement effects, highly enhanced surface-area-volume ratios, and the development of new surface dominated phenomena that are prevalent at the nanoscale [2].

Magnesium oxide (MgO) is a broadbandgap alkaline earth metal oxide (E g =7.8 eV) that crystallizes with the face-centered cubic (FCC) rock salt structure, space group Fm3m. It has a variety of impressive characteristics, such as high thermal and chemical stability, low dielectric constant, non-toxicity, and biocompatibility, all of which make it an extremely desirable material in terms of application in a range of applications, such as heterogeneous catalysis, advanced ceramics, environmental remediation, antimicrobial coatings, drug delivery platforms, and as a functional filler in polymer nanocomposites [3], [4]. On the nanoscale, the surface basicity and reactivity of MgO is further increased and its application to photocatalytic degradation of organic pollutants and its use as efficient antibacterial agent against multidrug-resistant pathogens is further extended [5].

Traditionally, MgO nanoparticles have been produced in a variety of physicochemical processes such as sol-gel synthesis, co-precipitation, hydrothermal and solvothermal, chemical vapor deposition, and flame assisted spray pyrolysis [6]. Even though these methods have the potential to produce nanoparticles with highly controlled morphologies and phase compositions, they are often associated with serious consequences, such as the use of toxic starting chemicals and organic solvents, high energy usage, and the formation of by-products that can be a hazard to the environment, and complicated multi-step processing regimens that present a barrier to scalable and cost-effective manufacturing [7]. These restrictions have created a lot of concern in the creation of sustainable, environmentally friendly synthetic substitutes.

Green synthesis, which exploits the use of biological materials, in the form of plant extracts, microorganisms and farm by-products, has presented an attractive paradigm of the manufacture of metal oxide nanoparticles eco-friendlily. Of interest is the ability of plant-mediated synthesis to use a rich repository of phytochemical constituents, such as polyphenols, flavonoids, alkaloids, terpenoids, and reducing sugars, which can serve as reducing agents and stabilizing/capping agents during nanoparticle nucleation and growth, eliminating the necessity of using exogenous toxic reducing chemicals [8]. The use of a wide variety of plant sources has been explored in the green synthesis of MgO NPs and has thus far produced nanoparticles with various morphologies and functionalities of leaf extracts of Aloe vera, Calotropis gigantea, and Psidium guajava, among others [9], [10].

The eggplant (Solanum melongena L.), which is a common and extensively grown plant of Solanaceae family, has shown a great promise as a bio-resource in the synthesis of nanoparticles. Its peel and fruit contain vast amounts of bioactive phytochemicals, such as nasunin (a potent anthocyanin antioxidant), chlorogenic acid, delphinidin, other phenolic acids, flavonoid glycosides and have high electron-donating and chelating capabilities that can stabilize and reduce metal ion precursors [11]. The application of eggplant extract is also explained by the fact that it is cheap, easy to obtain and is non-toxic with the valorization power that it holds on agricultural wastes. Although there are such benefits, the systematic description of MgO NPs produced through eggplant extract has not been thoroughly covered in the literature, which is why the current study is proposed.

The main aim of the current paper is to explain the preparation protocol of MgO nanoparticles by employing aqueous extract of eggplant as a bio-mediator and to offer a detailed structural and morphological characterization of the product of the synthesis through XRD, FESEM/EDX, and FTIR methods, and a goal to define the physicochemical identity and the phase purity of the yield..

2. Materials and Methods

2.1 Chemicals and Reagents

Magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$, purity ≥ 99%), purity, was bought at Sigma-Aldrich (Germany) and was not purified any further. Mature and fresh eggplant (Solanum melongena) fruits were purchased in a local market in Wasit, Iraq. The entire procedure of the experiment was conducted with the help of the use of double-distilled water to all solutions. Sodium hydroxide (NaOH, 97% and above) was purchased at Fluka Chemika and used to correct the pH where needed.

2.2 Preparation of Eggplant Aqueous Extract

Fresh eggplant fruits were washed by using double distilled water and tap water to get rid of surface contaminants and peelled along with the flesh cut into small pieces. The 50 grams of the cut eggplant matter were placed in 500 mL of double-distilled water in a 1000 mL Erlenmeyer flask and subjected to thermal extraction in 80 o C with continuous magnetic stirring at 400 rpm over 30 minutes. The extract was then left to cool at room temperature and then filtered using Whatman No. 1 filter paper to get rid of any leftover plant material. The clear light yellowish-brown filterate was kept in amber glass bottles at 4 o C until it was actually needed to make nanoparticles. The extract was utilised in the next 48 hours following

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the preparation to maintain phytochemical integrity.

2.3 Green Synthesis of MgO Nanoparticles

To prepare magnesium oxide nanoparticles, a fresh 0.1 M aqueous solution of magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$) was prepared in double-distilled water. A 50 mL eggplant extract was added dropwise to 150 mL of the magnesium precursor solution with continuous magnetic stirring at 60 °C. The mixture was maintained at this temperature for 2 hours, during which time the solution gradually changed color, indicating the formation of magnesium hydroxide precursor ($Mg(OH)_2$). The pH of the reaction mixture was adjusted to approximately 10–11 using a 1 M sodium hydroxide (NaOH) solution to ensure complete precipitation. The resulting precipitate was centrifuged at 6000 rpm for 15 minutes and washed three times, twice with distilled water and once with absolute ethanol, to remove residual organic impurities and nitrate ions. The washed precipitate was dried overnight in an oven at 80°C. The dried powder was then calcined in an electric furnace at 500°C for 3 hours under atmospheric conditions to analyze the hydroxide precursor and obtain the final crystalline magnesium oxide nanoparticles. The resulting white powder was left to cool in a dryer before analysis.

2.4 Characterization Techniques

X-ray diffraction (XRD) on a Shimadzu XRD-6100 diffractometer with Cu K alpha radiation (1.5406 A) at 1080o was used to determine the crystalline structure and phase purity of the MgO NPs synthesized and the average crystallite size estimated using the Scherrer equation. The field emission scanning electron microscopy (FESEM, TESCAN MIRA3) was used to examine the surface morphology and the size distribution of particles, whereas the elemental composition was measured simultaneously by energy-dispersive X-ray spectroscopy (EDX). To determine the functional groups and bonding properties of the synthetic nanoparticles on the surface, Fourier-transform infrared spectroscopy (FTIR) was conducted on a Bruker Vertex 70 spectrometer at the spectrum of 4004000 cm -1 with the use of KBr pellets.

3. Results and Discussion

3.1 X-ray Diffraction Analysis

X-ray diffraction analysis was employed to elucidate the crystallographic structure, phase composition, and crystallite size of the synthesized MgO nanoparticles. To clarify the crystallographic structure, phase composition and crystallite size of the produced MgO nanoparticles, X-ray diffraction analysis was used. The XRD pattern showed three sharp, intense, and unmistakably defined diffraction peaks measuring 2the values of 40.90deg, 62.27deg, and 78.57deg which had the distinctly defined indexed crystallographic planes (200), (220), and (222) of the rock salt face-centered cubic (FCC) MgO phase. The experimental d-spacing values of 2.106, 1.489 and 1.216 Angstrom were excellent compared with the standard reference values of 2.108, 1.4909 and 1.2173 Angstrom indicating that the synthesized product was pure and did not have any trace level impurity phases or secondary crystallographic contributions. The crystal structure is of the cubic Fm3m space group (No. 225), which is in agreement with the periclase type of structure of stoichiometric magnesium oxide. The steepness and uniformity of diffraction peaks all testify to the high quality of crystallinity attained after heat treatment at 500 o C which, to an adequate degree, supplied enough thermal energy to dissociate the hydroxide precursor to allow long-range crystallographic organization.

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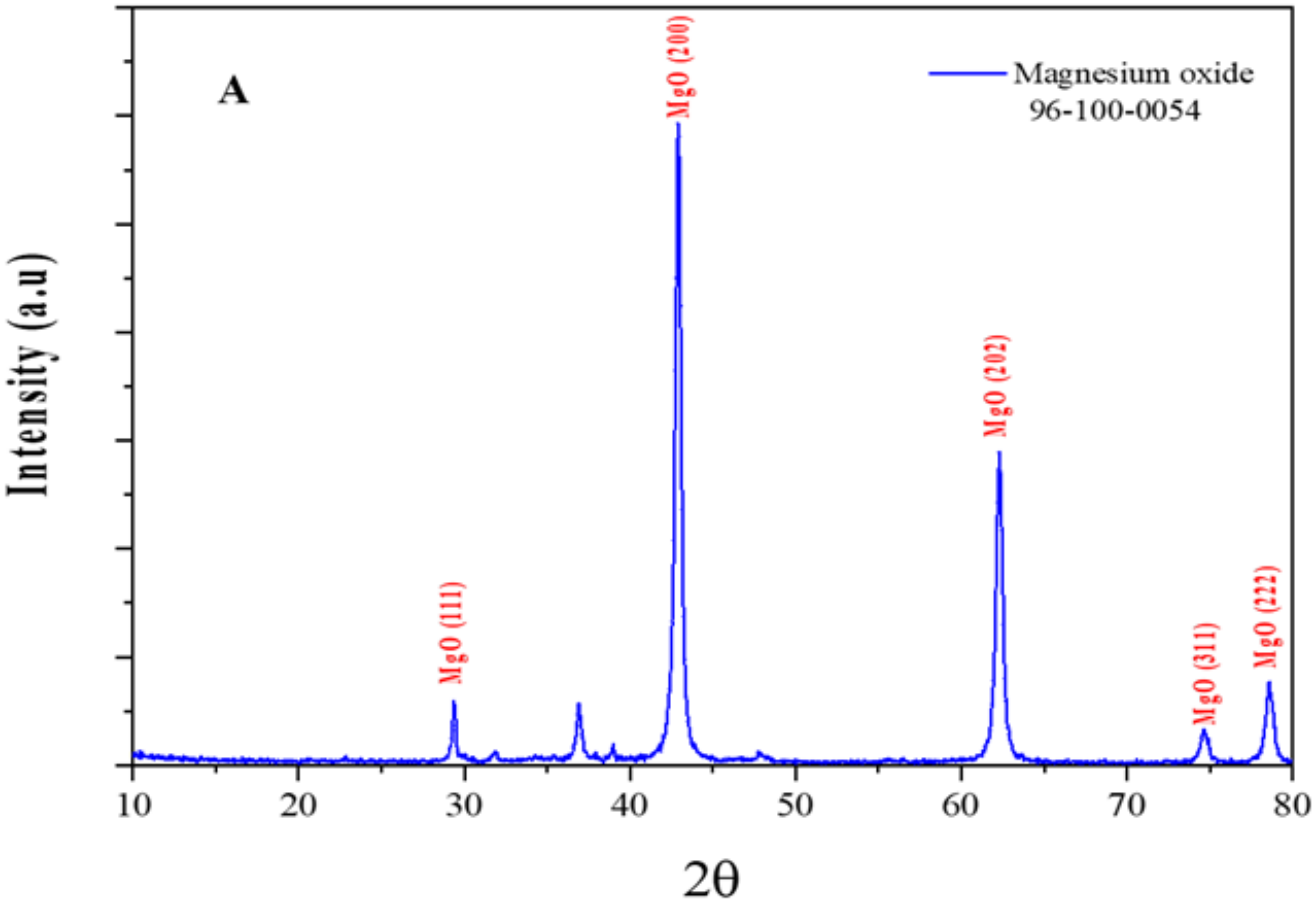

Figure 1. Figure 1 XRD Pattern of MgO NPs.

The average crystallite size (D) was calculated from the three most intense diffraction peaks using the Debye-Scherrer equation:

$D = K\lambda / (\beta \cos \theta)$

in which K is the Scherrer constant (K = 0.94 in the case of spherical crystallites), 0 is the X-ray wavelength of the Cu 1.5406 Ku -ray (here), 0 is the full width at half maximum (FWHM) of the diffraction peak which is in radians, and 0 is the Bragg diffraction angle. The values of individual crystallite sizes based on (200) (220) and (222) reflections were 18.53, 18.07, and 19.11nm respectively and resulted to an average crystallite size of 18.56nm as shown in Table 1. This nanoscale size is also consistent with the quantum size regime and with published values of the plant mediated MgO nanoparticles synthesized under similar conditions [12], [13]. The almost identical crystallite sizes of the various crystallographic planes indicate that crystallite growth was isotropic as a result of phytochemical capping to the eggplant extract, which was quite effective in controlling the kinetics of nucleation, and preventing favoritism with regard to crystal growth.

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Sample	MgO		
Two θ° (Deg.)	Strongest 3 Peaks		
	40.90	62.27	78.57
hkl	200	202	222
d_{hkl} Exp. (A°)	2.106	1.489	1.216
d_{hkl} Std. (A°)	2.108	1.4909	1.2173
Crystallite size (nm)	18.53	18.07	19.11
The average Crystallite size (nm)	18.56		
Card No.	96-100-0054 cubic		

Figure 2. Table 1. Structural parameters of green-synthesized MgO nanoparticles derived from XRD analysis.

3.2 FTIR Spectroscopy Analysis

The Fourier-transform infrared spectroscopy was used to determine the functional groups that were characteristic of the MgO nanoparticles synthesized as well as determine the contribution of the phytochemical constituents present in the eggplant extract to the bio-mediated synthesis process. The FTIR spectrum of the calcined MgO NPs had a strong absorption band in the low-wavenumber region at around 400 and 600 cm -1 , and this is due to the characteristic stretch vibration mode of Mg-O bond, which forms the major diagnostic signature of magnesium oxide and it is a sure confirmation that the inorganic MgO lattice structure has been formed [14].

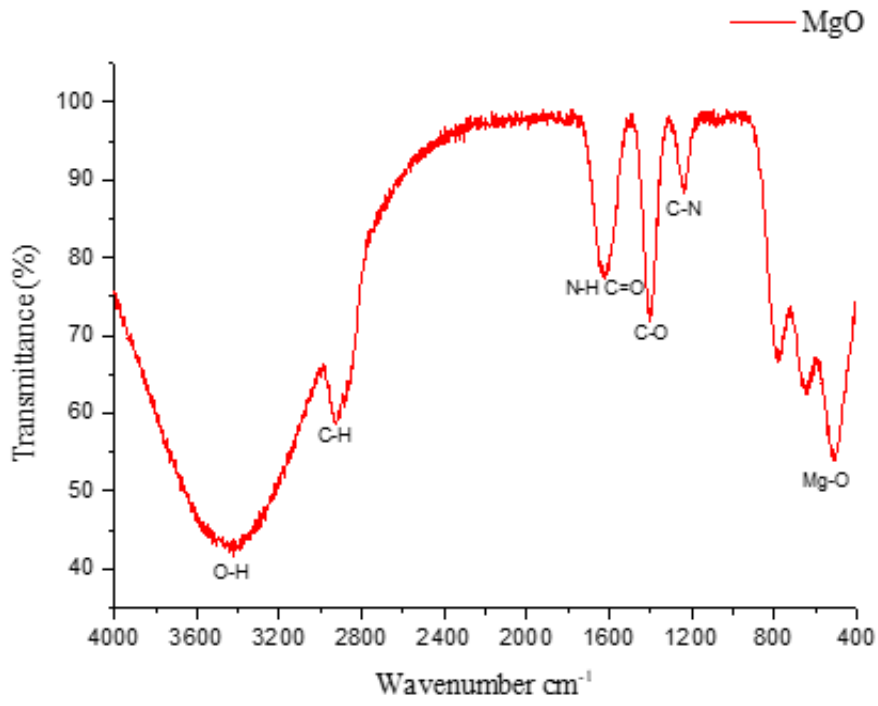

Figure 3. Figure 2 FTIR of MgO NPs.

This Mg -O stretch is the result of transverse optical phonon modes at the Brillouin zone centre, and is expected by the cubic

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rock-salt coordination environment of Mg 2 + in an octahedron of six oxygen anions. Mid-infrared absorption bands between 1300 and 1600 cm -1 can be tentatively attributed to residual surface carbonate species (CO3 -2) due to adsorption of atmospheric CO 2 on the highly basic MgO surface, which has been extensively reported in the MgO literature because of the high basicity of the surface of this material [15]. The broad absorption in the range of 3200-3500 -OH stretching vibrations of surface hydroxyl groups and adsorbed water molecules explain the appearance of the broad absorption in the range of 3200-3500 -OH, which means that there was a small layer of surface Mg(OH) 2 due to the interaction of atmospheric moisture, and this process is thermodynamically favourable as MgO is a hydroscopic substance. Bands of C-O and C=O vibrations, at 1000-1150 -1 and 1720 -1 respectively, indicating residual organic compounds of the eggplant extract upon the nanoparticle surface after the calcination process, are indicative that these phytochemical molecules were also used as effective surface-capping and stabilising agents in the formation of the nanoparticles. These organic signatures also indicate the existence of the active involvement of the biomolecules in the bioreduction and nucleation processes in the extract [16].

3.3 Field Emission Scanning Electron Microscopy (FESEM) Analysis

The surface morphology, size distribution, and micromorphology of the green-synthesized MgO nanoparticles were studied using the field emission scanning electron microscopy (FESEM). In the FESEM micrographs, it was found that the synthesized MgO NPs are mainly of quasi-spherical to near-equiaxed morphology and have a preference to slight agglomeration by van der Waals forces between particles, and magnetic dipole to dipole interaction, inherent to high-surface-area nanoscale materials. The experimentally determined agglomeration effect can be explained by the fact that nanomaterials have a relatively high surface energy and the coalescence of particles occurs at thermodynamic favorability. Individual nanoparticles had rather smooth surfaces, and there was no sign of a second phase or a second precipitate, as required by the phase-pure cubic MgO structure of XRD results.

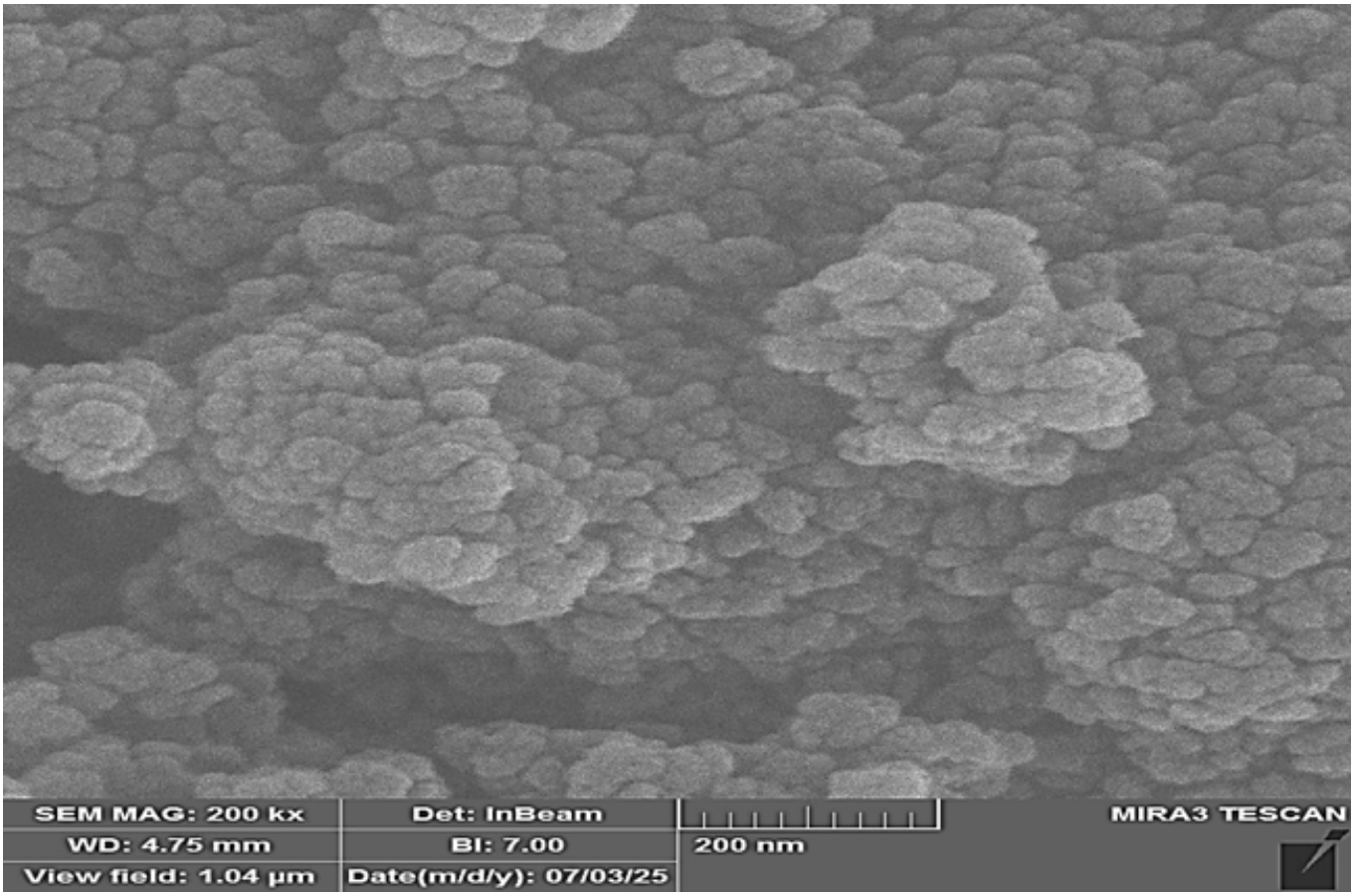

Figure 4. Figure 3 The FESEM images of MgO NPs.

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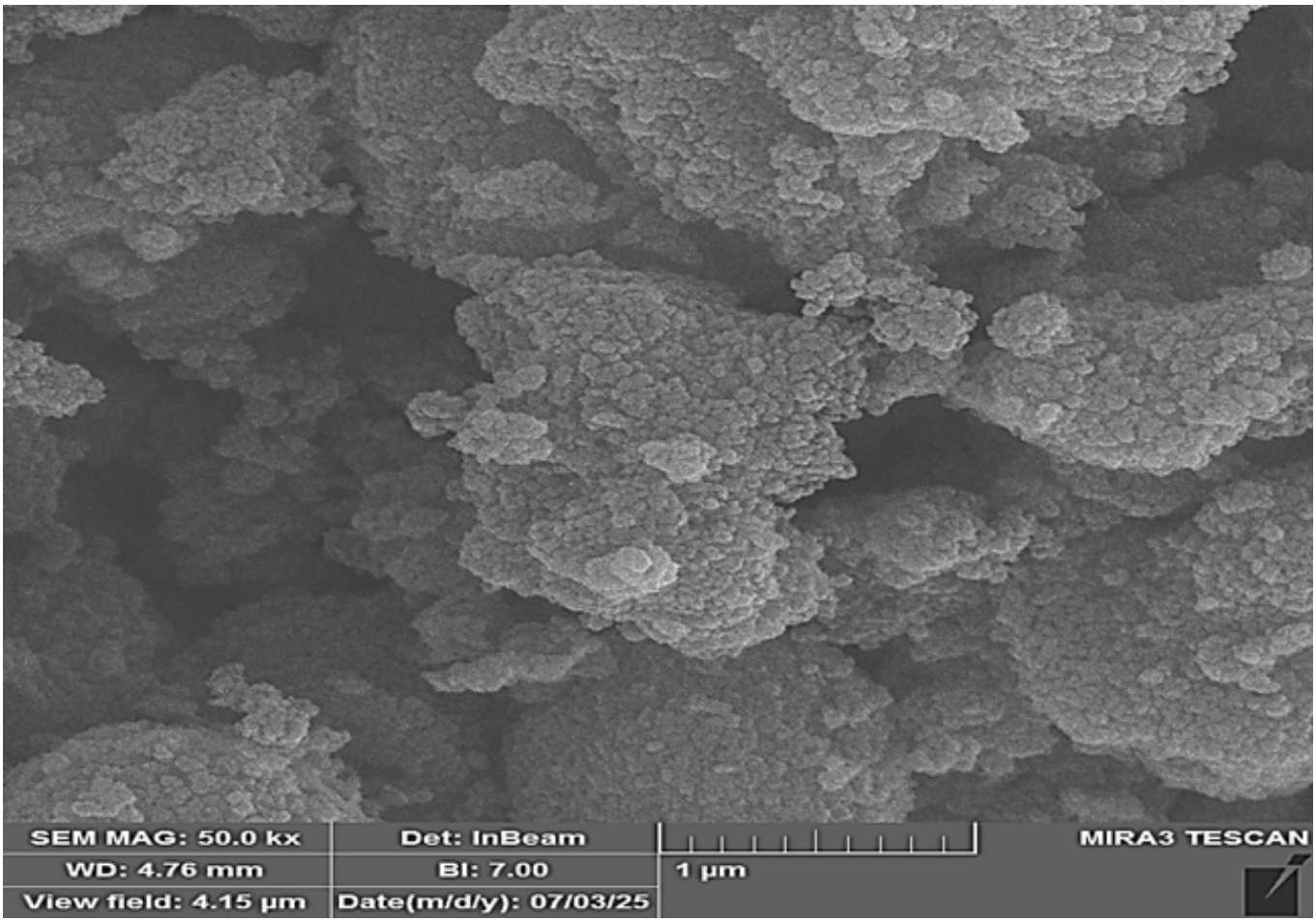

Figure 5. Figure 3 The FESEM images of MgO NPs.

Using the methods of analysis of quantitative images, the analysis of the FESEM micrographs was performed, and measurements of 85 separate nanoparticles were conducted using a Gaussian distribution. The results of the analysis were a mean particle diameter of 23.14 to 12.93 nm . Minimum and maximum measured sizes were 8.192 and 68.92nm, respectively and median position was 19.07nm, which show a right-skewed size distribution typical of bottom-up synthesised nanomaterials when Ostwald ripening might cause a fraction of the total material to be larger. The size of the particle as calculated using FESEM analysis (23.14 nm) is relatively larger than the size of crystallite as calculated using XRD/Scherrer (18.56 nm), a phenomenon that is frequently seen in the characterization of nanomaterials, and is due to an individual FESEM-measured particle potentially being a polycrystalline aggregate of multiple coherently diffracting crystalline domains. Also, the Scherrer equation examines the volume weighted mean crystallite dimension, and FESEM examines projected two-dimensional particle diameters intrinsically giving slightly larger values. Table 2 summarizes the data.

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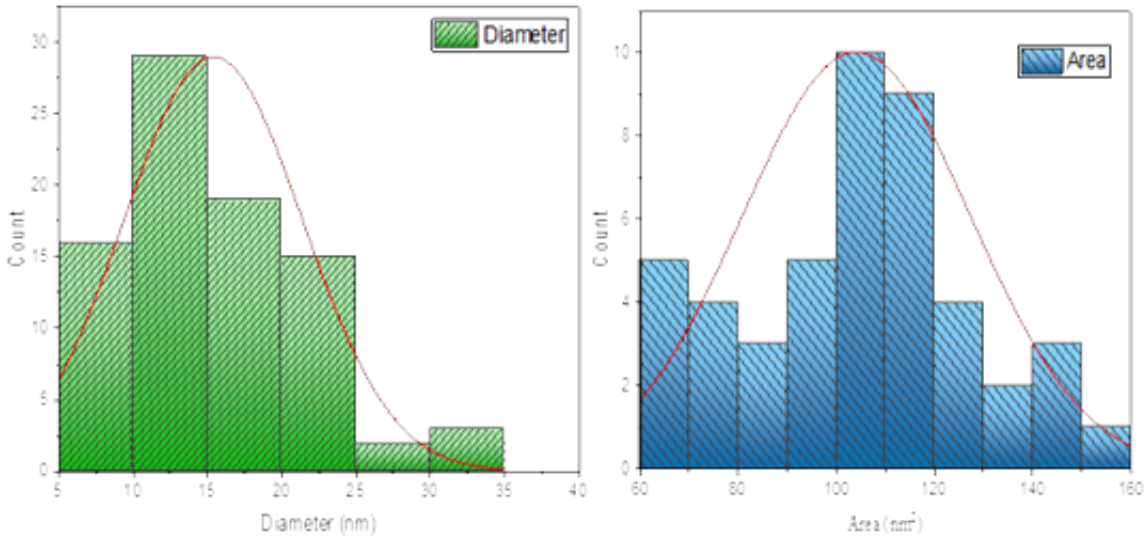

Figure 6. Figure 4 Histogram charts with Gauss distribution for MgO NPs.

	(N)Total	Mean	St.D.	Sum	Min.	Med.	Max.
Diameter (nm)	85	23.14	12.93	1967.70	8.192	19.07	68.92
Aera (nm^2)	80	104.107	23.310	4788.92	63.799	103.674	151.523

Figure 7. Table 2. Particle size statistics of MgO NPs derived from FESEM image analysis.

3.4 Energy-Dispersive X-ray Spectroscopy (EDX) Analysis

Energy dispersive X -ray spectroscopy (EDX) was used to establish elemental composition and confirm stoichiometric purity of the formed MgO nanoparticles. The EDX spectrum showed two prevailing characteristic peaks and these were magnesium (Mg), and oxygen (O), the only elemental constituents of stoichiometric MgO. The Mg and O weight and atomic percentages were determined to be in agreement with the expected values of pure MgO and this showed that the synthesis protocol using eggplant extract formed a chemically pure product free of any metallic or organic elemental impurities. The presence of neither nitrogen (N) nor carbon (C) at high intensities in the EDX spectrum indicates that the calcination process at 500 o C was successful in degrading and volatilizing any remaining nitrate anions of the Mg(NO3)2 source and organic biomolecules of the plant extract to produce a high chemical purity product. The fact that the experimental elemental ratios were close to the stoichiometry of Mg:O 1:1 (atomic ratio) of the rock-salt MgO phase is also an extra confirmation of the successful formation of the target compound. The combination of these results and XRD and FTIR data clearly showed the chemical identity of the biosynthesized MgO nanoparticles and their phase purity.

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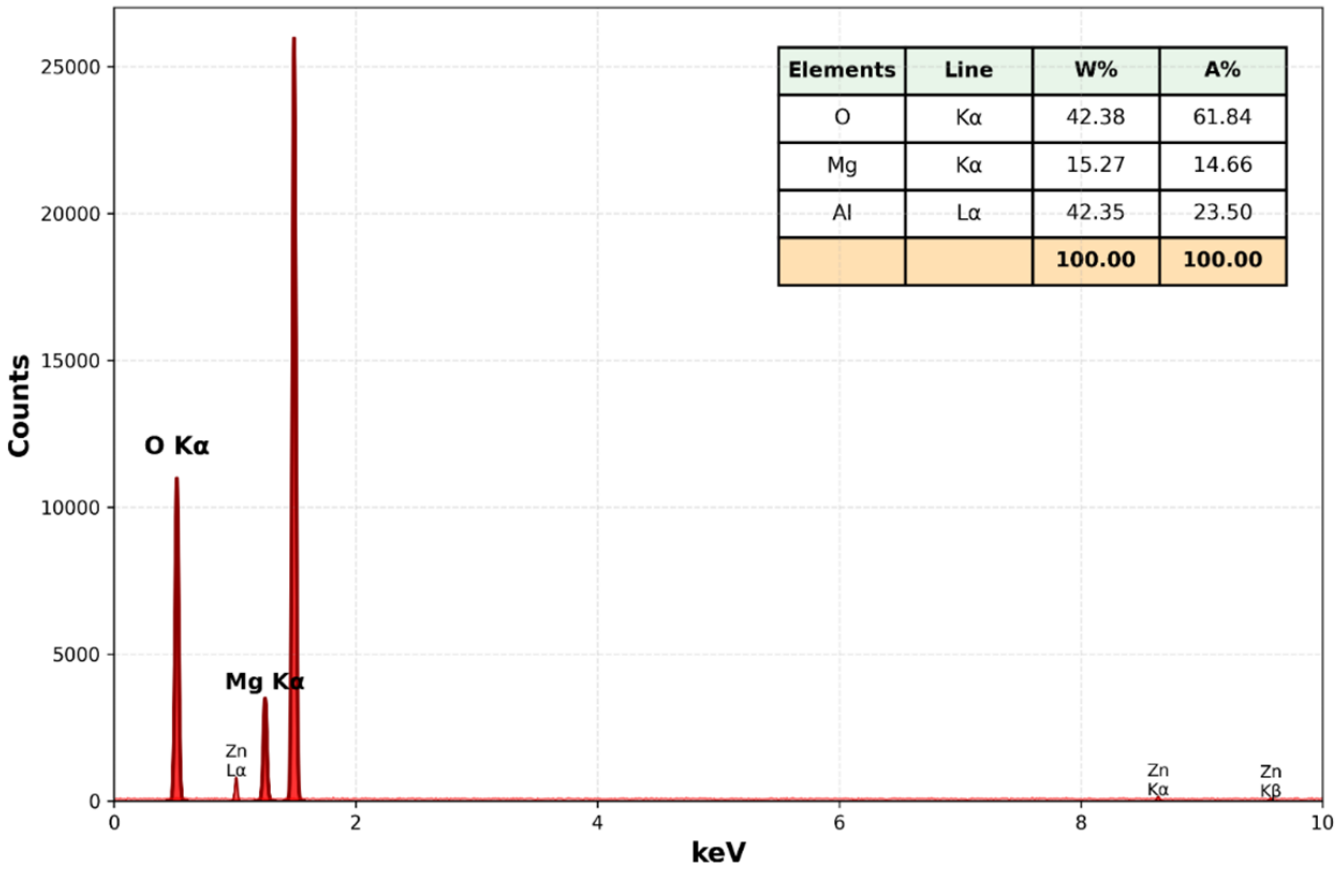

Figure 8. Figure 5 EDX of MgO NPs.

4. Conclusions

The current study has effectively indicated the viability and effectiveness of using aqueous eggplant (Solanum melongena) extract in the green regulation of magnesium oxide nanoparticles through the use of aqueous eggplant extract as a promoter and bio-mediator of non-toxic and cost-effective bio-adsorption of the desired highly crystalline magnesium oxide nanoparticles. The presence of phase-pure cubic MgO in the synthetic protocol with the rock salt crystal structure (space group Fm3m, COD Card No. 96-100-0054) and absence of impurity phases was unambiguously determined by XRD analysis at the high selectivity and efficiency of the synthetic protocol to the high degree of the plant. Using the DebyeScherrer equation on the three major diffraction reflections (200), (220), and (222)) gave an average crystal size of 18.56 nm which in itself was a solid indicator of the nanoscale nature of the synthesized material. Aqua fecita FESEM image analyses performed on a statistically representative sample of 85 particles indicated a quasi-spherical morphology, the mean size of the particles of 23.14 + 12.93 nm and a Gaussian size distribution, whereas the slightly larger particle size in comparison with the XRD-derived crystallite dimension is in line with the polycrystalline character of individual nanoparticles. The near-stoichiometric elemental ratio of Mg:O was again supported by EDX spectroscopy and the lack of significant elemental contamination made sure that the end product was pure in terms of its chemical composition. The phenomena of FTIR spectroscopy have given conclusive results to prove the formation of the Mg–O inorganic lattice due to its characteristic stretching vibration, and also depict the remaining signatures of the eggplant phytochemicals attached to the surface of the nanoparticle which point to its active participation in the biosynthesis process as a reducing and capping agent. Together, the collective evidence provided by all the applied characterization methods supports the claim that it is possible to extract eggplant using most of the techniques and proceed to the creation of well-defined MgO nanoparticles with high crystallinity, controllable nanoscale size, and surface purity, which are promising candidates to excel in the field of advanced applications in heterogeneous catalysis, photocatalytic degradation, antimicrobial coatings, and environmental remediation, and which should be the focus of systematic study in future studies.

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