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RESEARCH ARTICLE

Green Synthesis of MgO Nanoparticles via Two Different Methods and Studying their Structural, Morphological, and Optical Properties

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ABSTRACT

Magnesium oxide nanoparticles (MgO NPs) have been prepared by a green synthesis using the leaves of Ficus carica extract, which can be easily used for various biomedical applications. Magnesium oxide nanoparticles (MgO NPs) prepared by a green synthesis are non-toxic, biocompatible, and environmentally friendly. Fig extract functions as a reducing and stabilizing agent. The precursors used for preparation are from Magnesium Nitrate MgO(n) and bulk magnesium oxide MgO(b) by two different methods. The MgO(n) prepared from Magnesium Nitrate and MgO(b) prepared from bulk magnesium oxide nanoparticles were characterized using UV-visible spectroscopy (UV-Vis) to analyze the absorption patterns, The energy band gap (Eg) decreased from 7.8 eV to 4.7 eV for MgO(n) and from 7.8 eV to 5.3 eV for MgO(b). X-ray diffraction Spectroscopy (XRD) analyses show that the cubic phase structure is well-represented in both MgO NPs. showing that crystallite size was at 6.3 nm for MgO(n) and 7 nm for MgO(b). Field-Emission Scanning Electron Microscopy (FE-SEM) analysis for both MgO NPs showed a decrease in average particle size, with particles taking on a spherical form. The Energy Dispersive Spectroscopy (EDX) revealed patterns originating from Mg and O for both MgO NPs. Atomic force microscopy (AFM) Digitized pictures taken using this method have surface characteristics like root mean square roughness (Rq) and average roughness (Ra) quantified., it was found that the root mean square roughness (Rq) and the average roughness (Ra) for MgO(n) are much higher than MgO(b), which makes the MgO(n) prepared from magnesium nitrate more suitable for use in antibacterial applications.

Keywords: AFM, MgO nanoparticles, Method of precipitation, SEM, UV-VIS spectroscopic

Introduction

The field of study known as nanotechnology focuses on developing and studying structures and devices with length ranges ranging from one hundred nanometers to a few hundred nanometers, via controlled manipulation and investigation at these sizes.[1] The field of nanotechnology deals with the creation and manipulation of materials on a scale that ranges from the size of a single molecule or atom to that of a submicron, as well as the incorporation of these smaller entities into larger systems.[2] Depending on their size and form, nanomaterials exhibit physicochemical characteristics that vary from those of the bulk material. By manipulating its size and structure on a microscopic scale, the nanomaterial unexpectedly takes on a new personality with enhanced powers.[3] There are some physical and chemical techniques for synthesizing nanoparticles; nevertheless, it is not always possible to avoid using harmful substances in the synthesis process. As a result, finding a more sustainable way to create nanoparticles is a top priority. Biological approaches, which make use of either fungus or plant extracts, are a dependable and environmentally conscious substitute for traditional physical and chemical processes.[4]

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Concerns about energy consumption, chemical emissions, and the need for complicated machinery and synthesis conditions have led to the progressive replacement of physical and chemical processes with green synthesis techniques.[5] Biological synthesis or green synthesis has been described as a cost-effective, biocompatible, and eco-friendly nature.[6] Extensive attention from researchers in materials chemistry, medicine, agriculture, IT, biomedicine, optics, electronics, catalysis, the environment, energy, and senses is drawn to metal oxide nanoparticles due to their crucial significance in these domains.[7,8] As an example, nanoparticles of CuO,[9] SnO_2, $Al2O_3$, ZrO_2, TiO_2, and CeO_2, etc., have shown changes in cell characteristics as a result of size-related structural changes.[10] Catalysis, hazardous waste cleanup, the refractory material sector, paints, and superconductors are just a few of the many applications for magnesium oxide, one of the most significant functional metal oxides.[11] Recent reports have detailed the production of MgO-NPs from a variety of plant species, including Arachis hypogea L., Rosmarinus officinalis L., Trigonella foenum-graceae, and Manihot esculent. Consequently.[10] Consequently, the biodiversity of plants is considered in biological nanoparticle production because plant extracts may contain substances that can reduce mineral salts and cause nanoparticle formation. Ongoing research into the diagnostic and therapeutic applications of these nanostructures further supports this view.[12] The size and morphology of MgO particles can be modified using parameters such as pH, ionic strength, and different calcination temperatures.[13] Previous research has employed a variety of precursors to synthesize MgO, leading to a wide range of morphologies. To create MgO nanoparticles, scientists use a variety of preparation processes, each with its own set of pros and cons, such as sol-gel, hydrothermal, spray pyrolysis, combustion, microwave, and co-precipitation.[14,15] The current progress of magnesium oxide nanoparticles is through green synthesis. The aim of this research is a synthesis of Magnesium oxide nanoparticles (MgO NPs) by an eco-friendly method via two precursors to produce two different types of (MgO NPs) and make a comparison between them to use each one in a specific application.

Materials and methods

Preparation of aqueous leaf extract

Focus carica (Fig. 1) leaves were collected from the home garden washed well with distilled water to clean them from dust and dried in the shade for two days at room temperature and grind with an electric grinder. Using 400 milliliters of de-ionized water, 5 grams of leaf powder were combined in a 500 ml beaker heated to boiling for one hour then left to cool at ambient temperature(In boiling the extract, only a physical change occurs, without a change in

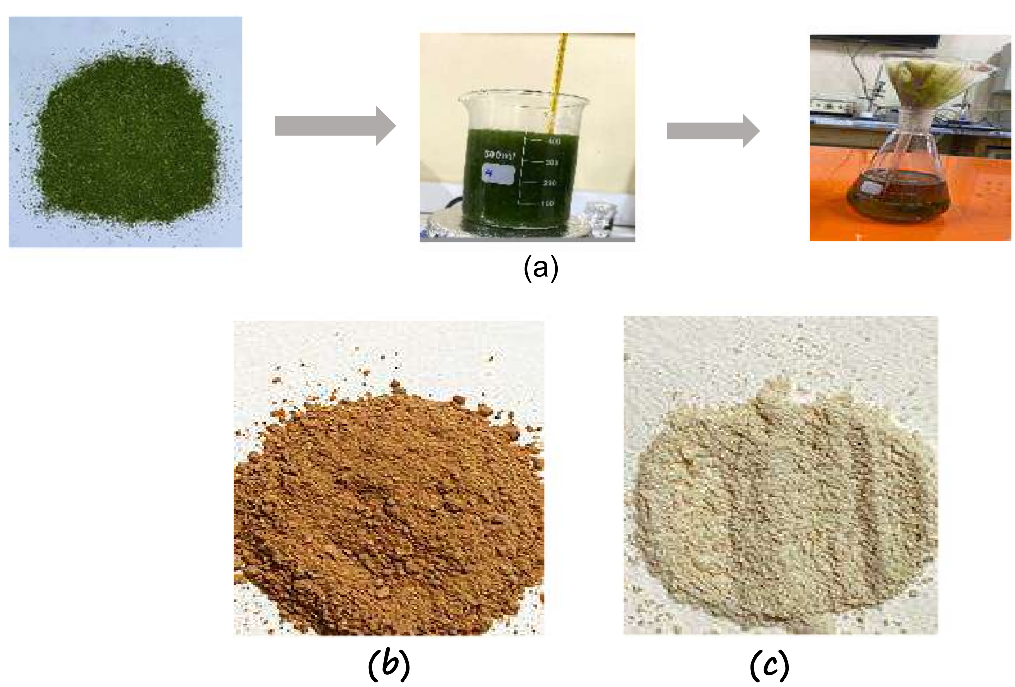


Fig. 1. Procedure steps (a) plant extract preparation (b) MgO NPs from bulk magnesium oxide (c) MgO NPs from Magnesium Nitrate hexahydrate Mg $(NO_3)2.6H_2$.

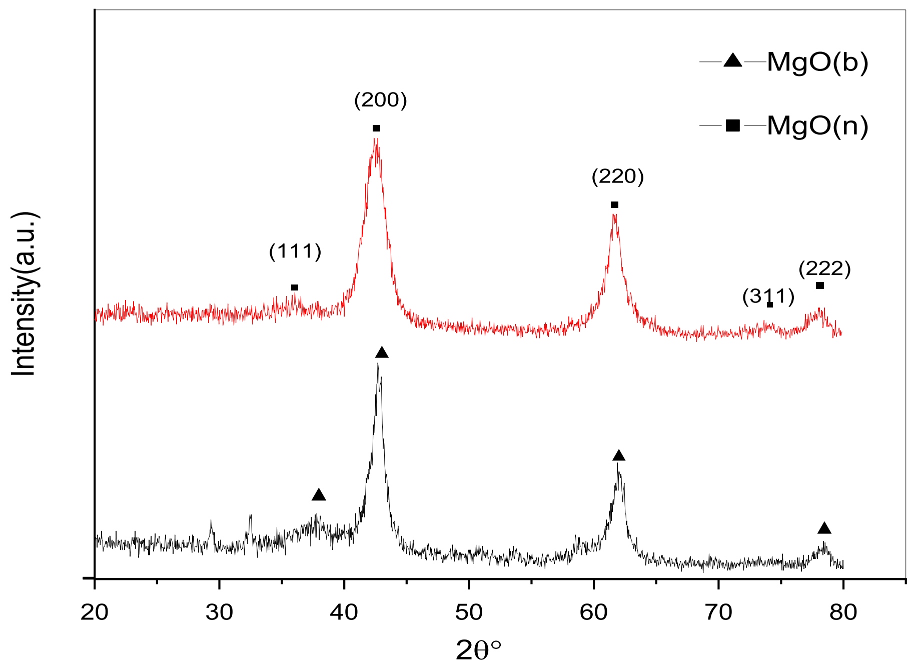


Fig. 2. Patterns of X-ray diffraction for both MgO NPs synthesized by green Method.

its chemical composition.) (as shown in Fig. 1). Afterward, aqueous extract was obtained by filtration with Whitman's filter paper and kept at 4°C for further use.

Green synthesis of MgO nanoparticle using fig leaves extract by

Using bulk magnesium oxide

The method described by Abdallah Y, et al.[16] (The study presented a biological method for manufacturing nanoflowers from magnesium oxide (MgO) without any catalyst. Aqueous rosemary extract was used to fabricate MgO nanoflowers (MgONFs) under stirring and temperature conditions at 70 °C for 4 h.)has been followed for the synthesis of MgO NPs with some modifications. For 30 minutes at 1500 rpm and 60°C, a magnetic stirrer was used to continuously mix 100 ml of Fig leaf extract with one gram of bulk magnesium oxide. In the next step the mixture was separated using a centrifuge the mixture at 4000 rpm for 15 minutes to separate the components. The final product was obtained by washing a precipitant with ethanol and de-ionized water to eliminate any contaminants. Then, it was calcined at 300 °C for 2 hours after naturally drying in the air and kept in dry condition for further characterization.

Using magnesium nitrate hexahydrate (Mg $(NO_3)_2.6H_2O$)

For 15 minutes, 2 grams of Magnesium Nitrate Hexahydrate (Mg $(NO_3)_2.6H_2O$) were mixed with 100 milliliters of deionized water while stirring, using a magnetic stirrer, for 15 min, 15 mL of Fig leaf extract was added to the solution. Once the reaction mixture reached the desired temperature, a 1M NaOH solution was added drop-wise to reach pH = 8. This caused a brownish-yellow solution to develop, which is proof that magnesium nanoparticles (MgO NPs) had been generated. For 20 minutes, the resulting suspension was diluted with water and ethanol to eliminate any remaining solution contaminants. Next, the solution was spun at 4000 rpm for 10 minutes and then calcined at 300 °C for 2 hours to get the pure MgO NPs. Fig. 2 illustrates the Magnesium oxide nanoparticles prepared by the green method from bulk magnesium oxide and from Magnesium Nitrate hexahydrate (Mg $(NO_3)_2.6H_2O$).

Table 1. Crystalline size and structural parameters of MgO(b).

2θ (Deg)	FWHM (Deg.)	Crystalline size D(nm)	hkl
37.9154	1.44000	5.830999932	(111)
42.6971	1.17000	7.287383961	(200)
61.9170	1.33330	6.945500293	(220)
78.2490	1.34000	7.639138034	(222)

Table 2. Crystalline size and structural parameters of MgO(n).

2θ (Deg)	FWHM (Deg.)	Crystalline size D(nm)	hkl
37.6756	1.12000	7.491631115	(111)
42.5855	2.09330	4.071561145	(200)
61.7737	1.70000	5.443235977	(220)
73.8602	1.28000	7.76119375	(311)
78.0191	1.52000	6.723544888	(222)

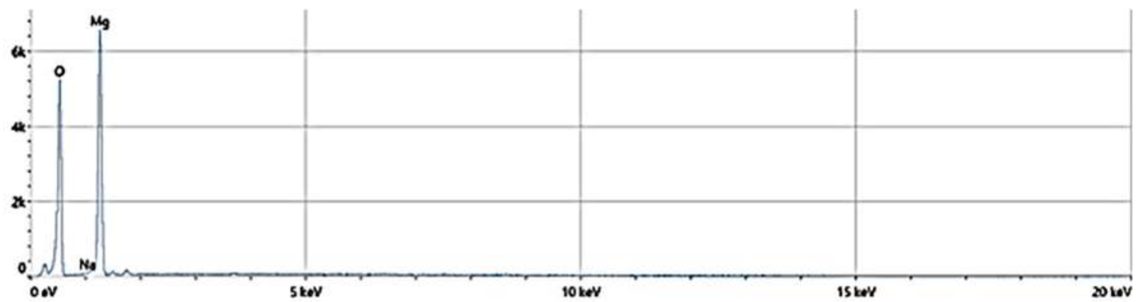


Fig. 3. EDX patterns for Nanoparticles of MgO(n).

Table 3. EDX value of MgO(n) NPs.

Element	Weight % Error	Weight %	Atomic % Error	Atomic %
Mg	0.4	57.4	0.4	67.2
O	0.2	42.6	0.2	32.8

Table 4. EDX value for Nanoparticles of MgO(b).

Element	Weight % Error	Weight %	Atomic % Error	Atomic %
Mg	0.3	58.0	0.3	67.7
O	0.2	41.5	0.1	31.9
Al	0.1	0.5	0.0	0.4

Results and discussion

X-ray diffraction spectroscopy

XRD investigations were performed in the 20°–80° angle range. The XRD analysis verified, that both of the produced MgO NPs had a cubic crystal structure, as shown in Fig. 2. XRD investigations were performed in the 20°–80° angle range. The 2θ peak locations for both MgO NPs that were synthesized were well-defined using the JCPDS NO. (00- 45 - 0946). Also, the crystalline size was evaluated by the following Debye Scherrer's formula:[17]

$$D = 0.94\lambda/\beta \cos\theta \quad (1)$$

In this context, D represents the crystal size, K is a constant with a value of 0.9, and λ and β are the full width half maximum (FWHM), signifying and validating that the MgO NPs possess face centered cubic (fcc) properties. it was found that the average crystallite size is 7 nm for MgO(b) (bulk magnesium oxide) and 6.3 nm for MgO(n) (Magnesium Nitrate). These results are in good agreement with Poonguzhali et al.[17] and with Abdallah et al.[16] The crystalline size values and structural parameters of MgO(b) NPs are shown in Table 1 while Table 2 illustrates the crystalline size values and structural parameters of MgO(n) NPs.

Energy dispersive X-ray spectroscopy (EDX)

EDS (energy diffusion spectroscopy) is a technique used to analyze the chemical composition of samples by analyzing the radiation that results from the interaction of the sample with an electron beam or an X-ray beam.

The EDX spectrum of MgO(n) nanoparticles, as shown in Fig. 3, reveals that the sample includes just Mg and O. The large peaks that correspond to Mg and O validate that the sample contains only those elements. There has been no detection of any other peak that corresponds to any other element (Ulwali et al.[14]). In Fig. 4 The EDX analysis of the area shows additional weak peaks of Al, for MgO(b), and peaks for Mg and O atoms, respectively as shown in Tables 3 and 4. Nickel, aluminum, silicon, or other materials may appear in an EDS examination for several reasons, including chemical reactions. Chemical

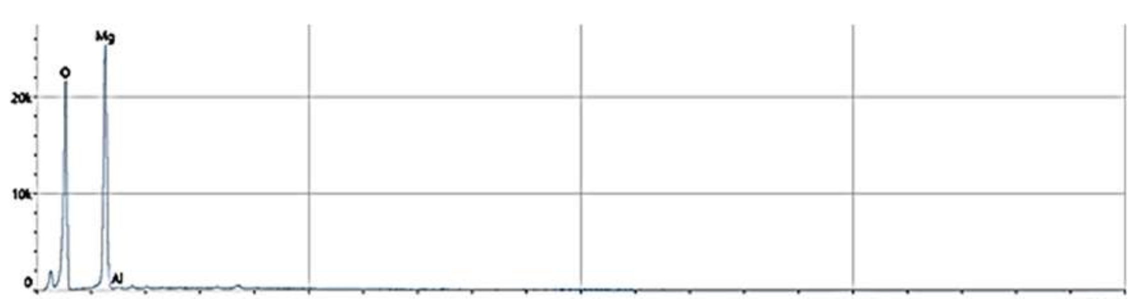


Fig. 4. EDX patterns for Nanoparticles of MgO(b).

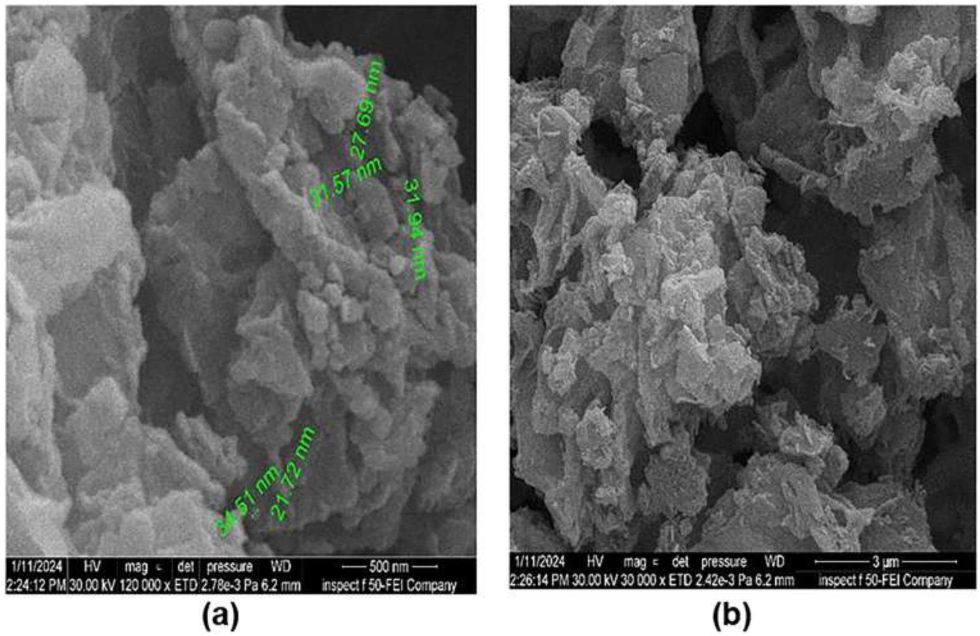


Fig. 5. The surface morphology of MgO(b) NPs by FE-SEM (a) 500 nm (b) 3 μm.

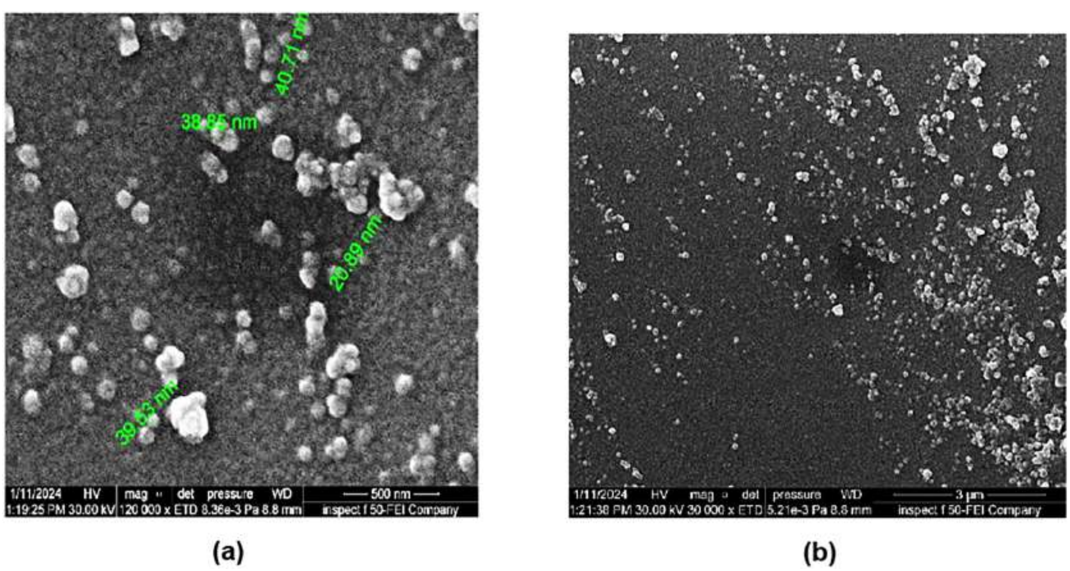
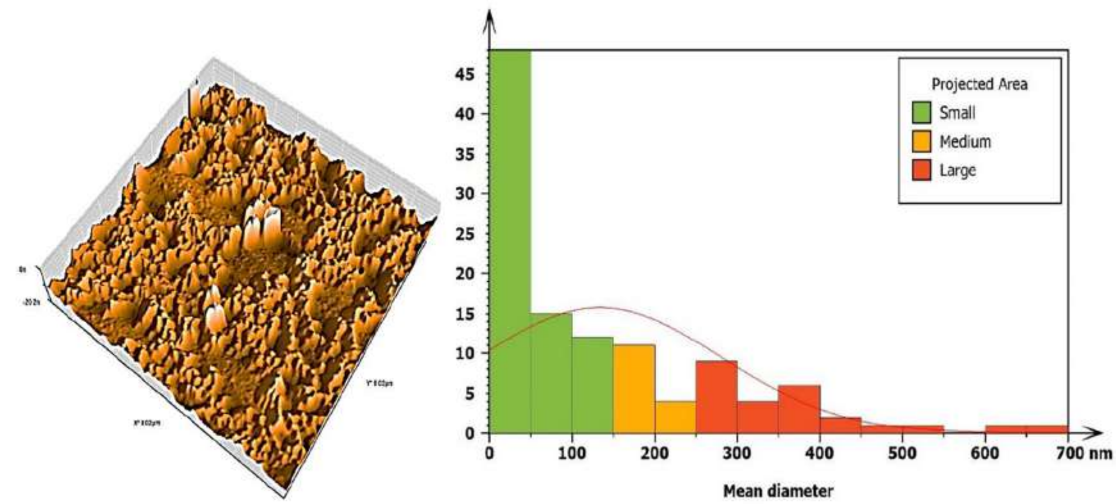


Fig. 6. The surface morphology of MgO(N) NPs by FE-SEM (a) 500 nm (b) 3 μm.

reactions may occur between the various components in the sample and the electron beam or X-ray used in the examination, leading to the appearance of signals of nickel, aluminum, or silicon. This means that everything is due to the way the sample is prepared for examination.

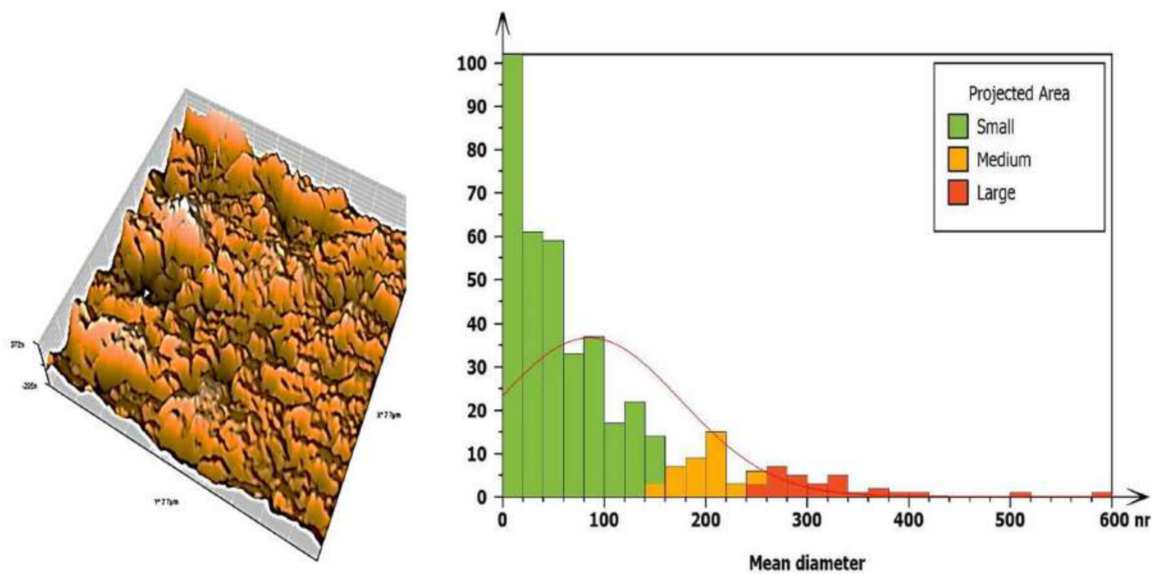
Field emission scanning electron microscopy (FE-SEM)

A field-scanning electron microscope was used to detect the surface morphology of magnesium oxide nanoparticles, as seen in Fig. 5. Through the use of the FE-SEM picture, the surface morphology of MgO nanoparticles was studied. Image analysis reveals that the synthetic MgO (b) NPs (tested as a powder) were in an agglomerated sphere shape with an average diameter of 29.49 nm. The FE-SEM analysis for MgO(n) NPs was obtained as shown in Fig. 6. (tested as glued particles on a glass substrate), which were approximately spherical with an average diameter of 34.9 nm. Believes that the reason for the difference in diameter is that the MgO(b) NPs were measured as powdered while the MgO(n) NPs were measured as



(a) (b)

Fig. 7. AFM images of MgO(b) NPs (a) three dimensions, and (b) histogram of the distribution of grain size.



(a) (b)

Fig. 8. AFM images of MgO(n) NPs (a) three dimensions, and (b) histogram of the distribution of grain size.

glued particles on a glass substrate, these results agree with Kumar et al.,[18] and Kurhade et al.[19]

Atomic force microscopy (AFM)

The surface's structure and elevation may be determined by analyzing the plot topographies under the Atomic Force microscope. The digital pictures used in this method may be analyzed from many angles, including 3D modeling, and quantitative surface properties like root mean square roughness (Rq) and average roughness (Ra) can be measured. Two examples of MgO NPs produced using environmentally friendly processes are shown in Figs. 7 and 8, which show the granularity distribution and three-dimensional AFM images, respectively. The root mean square roughness (Rq) for MgO(b) is 5.181 nm, and the average roughness (Ra) is 4.346 nm.

The root mean square roughness (Rq) for MgO(n) is 22.4 nm and the average roughness (Ra) is 15.06 nm. As we can see, the root mean square roughness (Rq) and the average roughness (Ra) for MgO (n) are much higher than MgO (b), so it has the possibility of being used in biomedical applications. The surface roughness increases the surface topography and has the potential to improve cell attachment. then increases

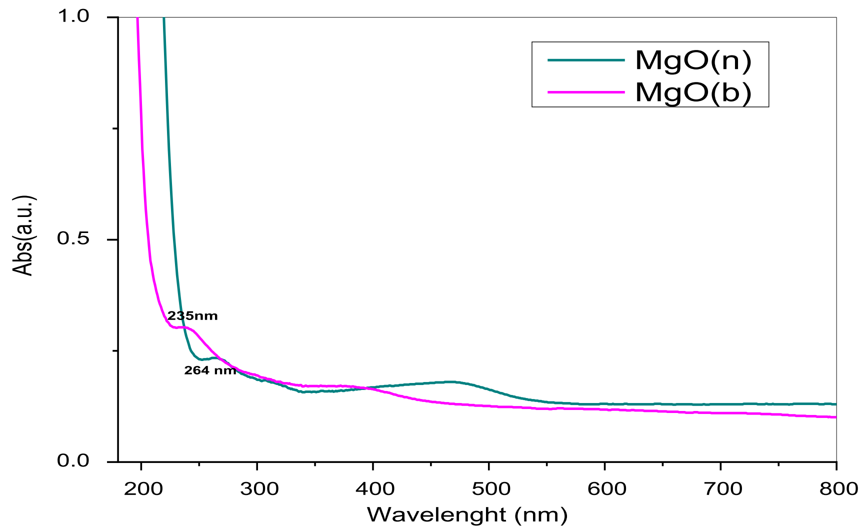


Fig. 9. UV–Visible spectrum of MgO Nanoparticles.

Table 5. Eg value of MgO NPs.

MgO NPs	Eg of MgO NPs	Eg of bulk MgO
MgO(b)	5.3 ev	7.8 ev
MgO(n)	4.7 ev	7.8 ev

the cell adhesion. These results agree with Ulwali et al.[14]

The optical analysis

The formation of MgO NPs with smaller sizes has a significant impact on the MgO NPs' optical absorption spectra, it has a strong property in the UV range (The wavelength of UV-vis spectroscopy ranges from 200 nm to 800 nm), (the number of outer electrons becomes more), and when the peak position moves to the UV zone, the energy landing on the molecules' outer electrons stimulates them and causes them to transfer to other levels. This depends on the type of radiation falling on it, either ultraviolet or visible light.[20,21] The energy band gap of MgO NPs (Fig. 9) was calculated using the Plank equation[22]

$$E = hc/\lambda \quad (2)$$

It was found that Eg = 4.7 eV for MgO(n) and 5.3 eV. for MgO(b) The Eg values are listed in Table 5. As. These results agree with Ulwali et al.,[14] Palanisamy et al.,[23] Kumar et al.,[18] Kurhade et al.[19]

Conclusion

This study is based on the green synthesis of MgO NPs by Fig leaves extract, which was synthesized by two different methods. The two types of MgO NPs have been characterized. XRD confirms that the particles formed are polycrystalline of both MgO nanoparticles with a crystal size from 6.2 to 7 nm for both MgO NPs. Both kinds of compounds were found to contain pure MgO, according to EDX analysis. The absorption spectra show an absorption edge shifted to the UV region with a decreased energy band gap for both types as compared with bulk MgO. From the results obtained, the average grain size was almost similar, and there were slight differences in the energy gap value, but there was a significant difference in the average roughness value. The average roughness (Ra) for MgO (n) is 15.06 nm, so it can be used in medical applications such as antibacterial and antifungal agents, while the average roughness (Ra) for MgO (b) is 4.346 nm, so there is a possibility of using it in the manufacture of sensors and solar cells, and also in biological applications.

Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Authors' contribution statement

N.KA. proposed the idea of research and guidance on how to prepare, as well as preparing the workplace, and supervised the findings of this work. A.H.O. prepared samples and conducted tests. All authors were verifying the analytical methods, discussing the results, and contributing to the final manuscript.

Journal declaration

N.K.A. author is an editor for the journal but did not participate in the peer review process other than as an author. The authors declare no other conflict of interest.

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التصنيع الأخضر لجزيئات أكسيد الماغنسيوم النانوية بطريقتين مختلفتين ودراسة خصائصها التركيبية والمورفولوجية والبصرية

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[2] *القسم الفيزياء ، الكلية العلوم للبنات ، جامعة بغداد ، بغداد، العراق.*

المستخلص

تم تحضير جزيئات أكسيد المغنسيوم النانوية (MgO NPs) عن طريق التخليق الأخضر باستخدام أوراق مستخلص نبات اللبخ كاريكا، والذي يمكن استخدامه بسهولة في العديد من التطبيقات الطبية الحيوية. تعد الجسيمات النانوية لأكسيد المغنيسيوم (MgO NPs) المحضرة بواسطة تخليق أخضر غير سامة ومتوافقة حيويًا وصديقة للبيئة. يعمل مستخلص التين كعامل اختزال واستقرار. السلائف المستخدمة للتحضير هي ا- نترات المغنيسيوم MgO(n) و2- ثأكسيد المغنيسيوم السائب MgO(b) . تم تشخيص MgO(n) المحضر من نترات المغنيسيوم وMgO(b) المحضر من الجسيمات النانوية لأكسيد المغنسيوم السائبة باستخدام التحليل الطيفي للأشعة فوق البنفسجية المرئية (UV-Vis) لتحليل أنماط الامتصاص.انخفضت فجوة نطاق الطاقة (Eg) من 7.8 فولت إلى 4.7 فولت لـ MgO(n) ومن 7.8 فولت إلى 5.3 فولت لـ MgO(b) . تُظهر تحليلات التحليل الطيفي حيود الأشعة السينية (XRD) أن بنية الطور المكعب واضحة بصورة جيدة في كلا MgO NPs. يُظهر أن الحجم البلوري كان عند 6.3 نانومتر لـ MgO (n) و 7 نانومتر لـ MgO (b). أظهر تحليل المجهر الإلكتروني لمسح الانبعاثات الميدانية (FE-SEM) لكل من MgO NPs انخفاضًا في متوسط حجم الجسيمات، مع اتخاذ الجزيئات شكلًا كرويًا. أظهر التحليل الطيفي المشتت للطاقة (EDX) أنماطًا ناشئة من Mg وO لكلا MgO NPs. مجهر القوة الذرية (AFM) الصور الرقمية الملتقطة باستخدام هذه الطريقة لها خصائص سطحية مثل جذر متوسط خشونة التربيع (Rq) ومتوسط الخشونة (Ra) كميا، حيث وجد أن جذر متوسط خشونة التربيع (Rq) ومتوسط الخشونة Ra ((Rq) بالنسبة لـ MgO(n) أعلى بكثير من MgO(b)، مما يجعل MgO(n) المحضر من نترات المغنيسيوم أكثر ملاءمة للاستخدام في التطبيقات المضادة للبكتيريا.

الكلمات المفتاحية: مجهر القوة الذرية ،جسيمات أكسيد المغنيسيوم النانوية، طريقة الترسيب المشترك، المجهر الإلكتروني الماسح، مطيافية UV-VIS.