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Eco-friendly Synthesis of Magnesium Oxide Nanoparticles using Arabic Gum

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Abstract

Magnesium oxide nanoparticles were synthesized using Arabic gum as a biotemplate and magnesium nitrate as the magnesium source by the sol-gel method. This method has many advantages such as nontoxic, economic viability, ease to scale up, less time consuming and environmental friendly approach for the synthesis of MgO nanoparticles without using any organic chemicals. Nanoparticles were characterized by Fourier transform infrared (FT-IR) spectroscopy, UV-visible spectroscopy and powder X-ray diffraction (XRD). The average crystallite size of MgO nanoparticles was calculated using Scherrer formula and Williamson Hall equation. The powder X-ray diffraction (XRD) analysis revealed the formation of cubic phase MgO with average particle size of 14 (Scherrer formula) and 5.3 (Williamson Hall equation) nm.

Keywords: *MgO, Arabic gum, Nanobiotechnology, Natural Hydrogel, Sol-gel.*

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Introduction

In recent years, metal oxide nanoparticles have a great attention because of the modification of properties perceived due to size effects, distribution and morphology due to present a higher surface-to-volume ratio with decreasing size of nanoparticles, thus modifying the catalytic, electronic, and optical properties of the metal NPs [1-5]. Among various metal oxides, magnesium oxide (MgO) is the most promising candidate due to its unique and excellent optical, electrical, thermal, mechanical and chemical properties and also its high ionic character [6]. At the nanoscale, MgO shows high reactivity because of the comportment of a great number of highly reactive edges, structural defects on the surface, unusual lattice planes and high surface to volume ratio [7].

MgO has a very high melting point and low heat capacity, which makes it a suitable candidate for insulation applications. MgO has numerous applications in various fields such as catalyst supports, agricultural products, paints, superconductor products, antimicrobial materials, photonic devices, refractory materials, electro-optical devices, sensors and as adsorbent [8-11]. Various kinds of fabrication techniques are employed to synthesize MgO nanoparticles such as chemical vapor deposition (CVD) [12], microwave [11], solvothermal reaction [13], electrochemical [14], laser ablation [15], sonochemical synthesis [16], hydrothermal method [17], sol-gel method [18, 19] and co-precipitation method [20]. The sol-gel method has gained much interest among researchers as it offers controlled consolidation, shape modulation, patterning of the nanostructures and low processing temperature [21].

Polysaccharide hydrocolloids such as carrageenan, alginate, agar-agar, starches, pectin, guar and gums (Arabic, tragacanth, and karaya) are high molecular weight macromolecules. Gums are naturally occurring polysaccharide components in plants, which are mostly green, economical, and easily available [22]. Green synthesis of MgO NPs using natural polymers provides advancement over chemical and physical method as it is cost effective, no need to use high energy, environmentally friendly, and no toxic chemicals. Arabic gum (AG) is a complex polysaccharide, which is obtained as sticky exudates from the stems and branches of Acacia trees [23]. As the chief components of AG, Polysaccharides (galactosyl, arabinosyl, rhamnosyl, glucuronosyl, and 4-*O*-methyl-glucuronosyl), are effective reducing agents [24, 25]. In this work, we have synthesized magnesium oxide nanoparticles using AG by the sol-gel method as a flashy and friendly approach to the nature. Magnesium nitrate was used as the magnesium source. The synthesized samples were characterized using FTIR, PXRD, and UV-Vis spectroscopy.

Experimental

Material and methods

The Arabic gum (AG) was obtained from a local health food store. Magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$ was purchased from daijung (Darmstadt, Korea) and used without further purification. The IR spectra were measured on a Jasco 6300 FT-IR spectrometer (KBr disks). UV-Vis absorption spectra were prepared on a Metrohm (Analytical Jena-Specord 205) double beam instrument. The measurement was carried out from 195 nm to 700 nm wavelength for sample. The structural properties of synthesized nanoparticles were investigated by powder X-ray diffraction (XRD) pattern on a X'Pert-PRO advanced diffractometer using Cu (Kα) radiation (wavelength: 1.5406 Å) at 40 kV and 40 mA at room temperature in the range of 2θ from 20 to 80º.

Synthesis of MgO nanoparticles using Arabic gum

In a typical synthesis, 0.3 g of the Arabic gum (AG) was dissolved in 40 ml of distilled water and stirred for 120 min at 75 ºC to achieve a clear Arabic gel (AG) solution. After that, 2g of $Mg(NO_3)_2.6H_2O$ was added to the AG solution and the container was placed in a sand bath. The temperature of the sand bath was fixed at 75 ºC and stirring was continued for 14 h to obtain a brown color resin. The final product was calcined at 500 ºC temperatures in the air for 4h to obtain a white powder of MgO.

Results and discussion

Characterization of MgO nanoparticles.

FTIR spectra were recorded in solid phase using the KBr pellet technique in the range of 400-4000 cm^{-1}. Figure 1 shows the IR spectrum of the sample that calcined at 500°C for 4 hours. The spectrum shows the stretching vibration mode for the Mg–O–Mg moiety at the range of 587– 681 cm^{-1} as a broad band. Two distinct bands are seen at the wave number ranges of 1014–1074 cm^{-1} and 1590-1641 cm^{-1} are attributed to the bending vibration of absorbed water molecule and surface hydroxyl group (–OH), respectively. A broad vibration band is seen in the wavenumber range 3325–3553 cm^{-1} due to the O–H stretching vibration of absorbed water molecule and surface hydroxyl group [26]. This is due to the aerial adsorptions of water molecule onto the MgO surface when it is exposed to the atmosphere. The FT-IR absorption peak that seen at the wavenumber 1420 cm^{-1} is assigned to the asymmetric stretching of the carbonate ion, CO_3^{2-} species [9].

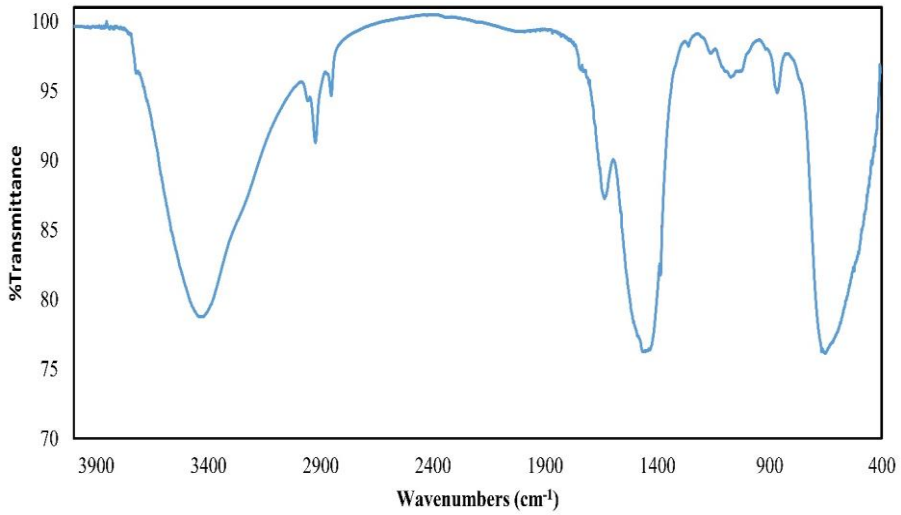

Figure 1. FT-IR spectrum of MgO NPs.

The room temperature UV-Vis absorption spectrum of the MgO is shown in Figure 2.

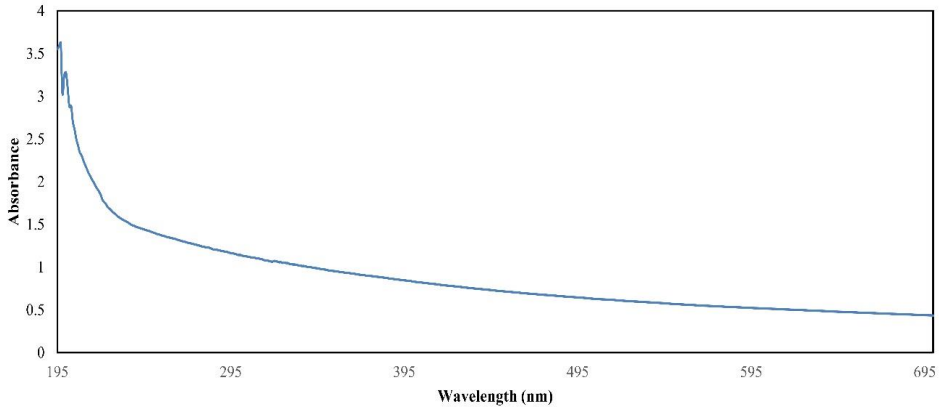

Figure 2. UV-Vis spectrum of MgO NPs.

The green synthesized MgO-NPs showed absorbance spectra at 200 nm in the UV-visible spectroscopy, which are attributed to the formation of magnesium oxide (MgO) nanoparticles [27]. The crystal structure confirmation analysis was carried out by the X-ray diffraction patterns. XRD patterns of the product obtained by calcination of precursor at 500 °C are shown in Figure 3.

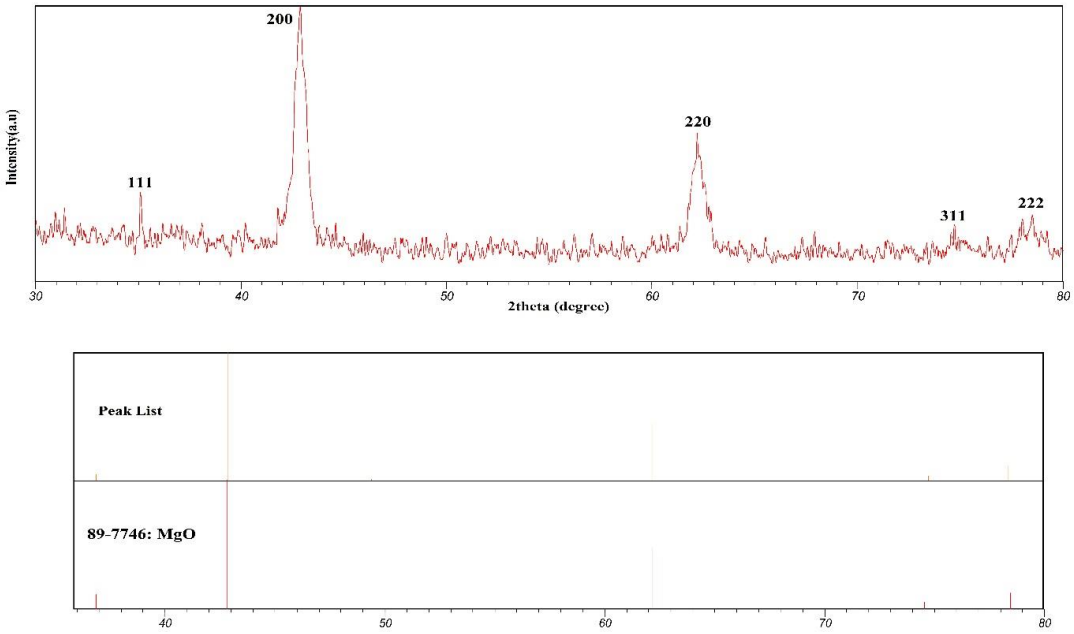

Figure 3. XRD pattern of synthesized MgO NPs.

XRD analysis showed a series of diffraction peaks at 2θ of 36.86, 42.83, 62.18, 74.71 and 78.31 that can be assigned to (111), (200), (220), (311) and (222) planes, respectively. All the diffraction peaks were readily indexed to a pure cubic phase of MgO (JCPDS Card no. 89-7746) with a=b =c= 4.2198 Å. The diffraction patterns are well matched with the literature [28] and no impurity peaks were observed. Furthermore, the strong and sharp diffraction peaks confirm the high crystallinity of the products.

Scherrer formula

The average crystallite size of MgO nanoparticles was determined from the full width at half maximum (FWHM) of the XRD patterns using the well- known Scherrer formula:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

where D is the crystallite size (nm), β is the full width at half maximum of the peak, λ is the X-ray wavelength of Cu Kα= 0.154 nm and θ is the Bragg angle [29]. Using the above method we obtained an average size of 14 nm for MgO nanoparticles.

Williamson Hall Equation

The Williamson Hall equation was used to calculate the crystallite size as well as the Micro strain of the sample, which equation was shown in below:

$$\beta cos\theta = \frac{K\lambda}{D} + 4\varepsilon sin\theta$$

Where D is the average crystallite size of the particles, K is Debye scherrer's constant (=0.9), λ is the wavelength of the CuKα-radiation (=0.154 nm), β is the full width half maximum (FWHM) of the peak, θ is the Bragg's angle and ε is the micro strain of the sample.

For this analysis a graph is drawn between βcosθ against 4sinθ along y and x axis respectively (Figure 4). Linear extrapolation is employed to this plot, the crystallite size is given by the intercept $\frac{K\lambda}{D}$ and the strain (ε) is given by the slope [30].

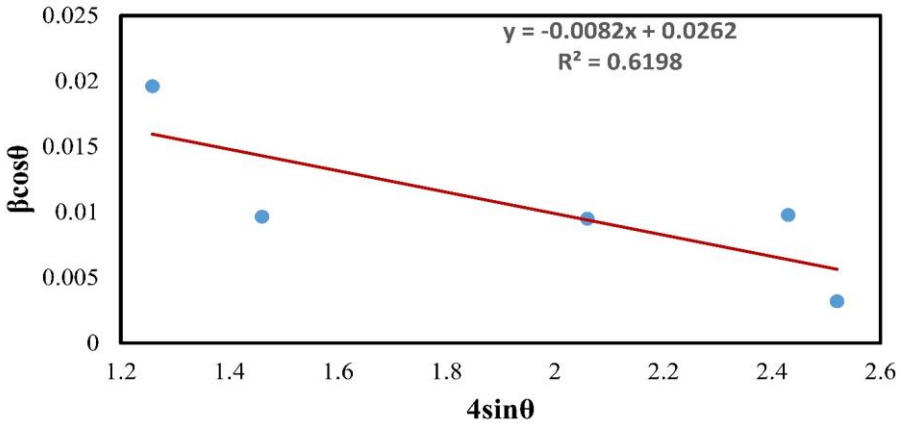

Figure 4. Williamson hall Plot for MgO NPs.

The micro strain was calculated from the slope of the straight line fit of W-H plots. The nature of strain can also be predicted from this technique. A negative slope indicates a compressive strain, whereas a positive slope is the signature of tensile strain. In the present case, a compressive strain was found to appear in the annealed sample. Using the above method micro strain was calculated as 0.0082. The average crystallite size was measured by taking $\frac{K\lambda}{D}$ to y intercept in the Figure 4. Then the results carried out as 5.3 nm as average crystallite size of the sample using Williamson Hall Equation.

Conclusion

We developed a simple, efficient, cheap, environment-friendly and green protocol for synthesis of MgO nanoparticles from $Mg(NO_3)_2.6H_2O$ in the presence of Arabic gum as a natural and non-toxic template and stabilizing agent. Applications of this process for the

preparation of other metal oxide nanoparticles are under investigation in our laboratory and will be reported in due course.

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