

Synergetic effect of MgO-Chitosan-pluronic F-127 nanocomposites

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Abstract

In the present work, Magnesium oxide (MgO)-Chitosan-pluronic F-127 nanocomposites were prepared using a green process with *Amomum subulatum* Roxb extract. The synthesized nanocomposites were characterized by X-ray diffraction studies, dynamic light scattering, Fourier transform infra-red spectroscopy, UV-Vis spectroscopy, Photoluminescence spectroscopy and antibacterial studies.

1.Introduction

Nanotechnology is a new technology which has the potential to change the way we live, interact and communicate on a massive scale. Never before has there been a technology that has so much potential for the modern world. The main concept of nanotechnology is the ability to create functional materials, devices and systems through the control of atoms and molecules on an atomic-scale and make these materials more accessible, faster, lighter and more compact[1-2].

Magnesium (Mg), chemical element, one of the alkaline-earth metals of Group 2 (IIa) of the periodic table, and the lightest structural metal. Its compounds are widely used in construction and medicine, and magnesium is one of the elements essential to all cellular life. This element is produced in large, aging stars from the sequential addition of three helium nuclei to a carbon nucleus. When such stars explode as supernovas, much of the magnesium is expelled into the interstellar medium where it may recycle into new star systems. Magnesium is the eighth most abundant element in the Earth's crust^[11] and the fourth most common element in the Earth (after iron, oxygen and silicon), making up 13% of the planet's mass and a large fraction of the planet's mantle. It is the third most abundant element dissolved in seawater, after sodium and chlorine.

Magnesium occurs naturally only in combination with other elements, where it almost always has a +2 oxidation state. The free element (metal) can be produced artificially, and is highly reactive (though in the atmosphere it is soon coated in a thin layer of oxide that partly inhibits reactivity –

see passivation). The free metal burns with a characteristic brilliant-white light. The metal is now obtained mainly by electrolysis of magnesium salts obtained from brine, and is used primarily as a component in aluminium-magnesium alloys, sometimes called magnalium or magnelium. Magnesium is less dense than aluminium, and the alloy of the two is prized for its combination of lightness and strength.

This element is the eleventh most abundant element by mass in the human body and is essential to all cells and some 300 enzymes. Magnesium ions interact with polyphosphate compounds such as ATP, DNA, and RNA. Hundreds of enzymes require magnesium ions to function. Magnesium compounds are used medicinally as common laxatives, antacids (e.g., milk of magnesia), and to stabilize abnormal nerve excitation or blood vessel spasm in such conditions as eclampsia.

Chitosan is a sugar that comes from the outer skeleton of shellfish, including crab, lobster, and shrimp. It's used as medicine and in drug manufacturing. Chitosan is a fibrous substance that might reduce how much fat and cholesterol the body absorbs from foods. It also helps blood clot when applied to wounds. People use chitosan for high blood pressure, high cholesterol, obesity, wound healing, and many other purposes, but there is no good scientific evidence to support many of these uses[3].

Pluronic represent an important class of biomedical polymers. They are unique materials composed of triblock PEO–PPO–PEO copolymers of poly (ethylene oxide) (PEO) and poly (propylene oxide) (PPO). The pluronic PEO block is hydrophilic and water soluble while the PPO block is hydrophobic and water insoluble. In an aqueous environment, these block copolymers self-assemble into micelles with a hydrophobic PPO center core and a hydrophilic PEO outer shell that interfaces with water.

2. Experimental Method

2.1 Preparation of extract

5 g of dried *Amomum subulatum* Roxb was combined with 100 mL of ethanol and boiled for 20 minutes at 80 °C. The obtained extraction was filtered using Whatman No. 1 filter paper, and the filtrate was collected in a 250 mL Erlenmeyer flask and stored at room temperature for further usage.

2.2 MgO-Chitosan-pluronic F-127 nanocomposites preparation

90 mL of 0.1M magnesium (II) nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$) with 1% acetic acid of 0.5 gm chitosan and 0.5gm of pluronic F-127 solution combined with 10 mL of *Amomum subulatum* Roxb extract, which yields a light-black colored homogeneous mixture solution for the preparation of MgO NPs. This solution was stirred constantly at a temperature of 80 °C for 5 hours. The resultant ash-white precipitate was dried at 120 °C for 1 hour. The obtained MgO-Chitosan-pluronic F-127 nanocomposites sample in powder form were annealed at 800 °C in air for 5 hours and used for further studies.

2.3 Antibacterial assay

The antibacterial activity of the MgO-Chitosan-pluronic F-127 nanocomposites was investigated by the well diffusion method and tested against G+ and G-bacteria (*Bacillus subtilis*) and (*Proteus vulgaris*) after molten nutrient agar, according to the Clinical and Laboratory Standards Institute (CLSI). After inoculation, well loaded with 1, 1.5, and 2 mg/ml of the test samples were placed on the bacteria-seeded well plates using micropipettes. The plates were then incubated at 37 °C for 24 hrs. The inhibition zone was measured. Amoxicillin (Hi-Media) was used as the positive control against G+ and G-bacteria.

2.4 Characterization Methods

The MgO-Chitosan-pluronic F-127 nanocomposites were characterized by an X-ray diffractometer (model: X'PERT PRO PANalytical). The diffraction patterns were recorded in the range of 20°-80° for the MgO-Chitosan-pluronic F-127 nanocomposites where the monochromatic wavelength of 1.54 was used. The Nano Plus Dynamic Light Scattering (DLS) Nano Particle Sizer was used for the particle size analysis of the MgO-Chitosan-pluronic F-127 nanocomposites. The spectrum of the FTIR was recorded in the wavenumber range of 400-4000 cm⁻¹ by using the Perkin-Elmer spectrometer. The UV-Vis-NIR spectrum was recorded in the wavelength range of 200-1100 nm using Lambda 35. Photoluminescence spectra were measured using the Cary Eclipse spectrometer.

3. Results and Discussion

3.1 X-ray diffraction (XRD)

The XRD patterns of the synthesized MgO-Chitosan-pluronic F-127 nanocomposites are, given in Fig. 1a. The diffraction peaks occurred at 2θ values of: 36.76, 42.67, 62.06, 74.14, and 78.22° for the MgO-Chitosan-pluronic F-127 nanocomposites corresponding to the (111), (200), (220), (311) and (222) (hkl) crystal planes of the Face Centered Cubic (FCC) structure. The maximum intensity peak was found at the (200) (hkl) plane, which indicates the fact that the MgO-Chitosan-pluronic F-127 nanocomposites have an FCC structure (JCPDS no: 89-7746), consistent with the crystal shape of MgO NPs, described previously. The average crystallite size of the MgO-Chitosan-pluronic F-127 nanocomposites was estimated by using the Scherrer equation, as follows:

The average crystallite size of the sample is calculated after appropriate background correction from X-ray line broadening of the diffraction peaks of using Debye Scherrer's formula,

$$\text{Average crystallite size } D = \frac{k\lambda}{\beta_{D\cos\theta}}$$

Where D is the size in nanometers, λ is the wavelength of the radiation (1.5406Å for CuKα), k is a constant (0.94), β_D is the peak width at half-maximum in radian along (101) plane and is Bragg's diffraction angle. The MgO-Chitosan-pluronic F-127 nanocomposites average particle size is 45 nm.

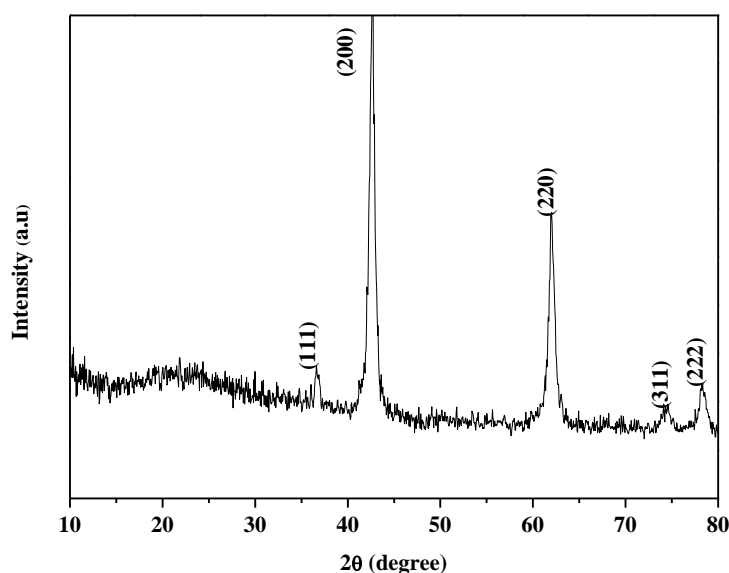


Figure 1 X-ray diffraction patterns MgO-Chitosan-pluronic F-127 nanocomposites

3.2 Dynamic Light Scattering (DLS)

The hydrodynamic diameter of MgO-Chitosan-pluronic F-127 nanocomposites was determined using dynamic light scattering to obtain particle size information. The MgO-Chitosan-pluronic F-127 nanocomposites size was observed at 55 nm; because a water medium surrounded the NPs, the DLS particle size was similarly as compared to the XRD results[4-5]. This is known as hydrodynamic size(Fig 2).

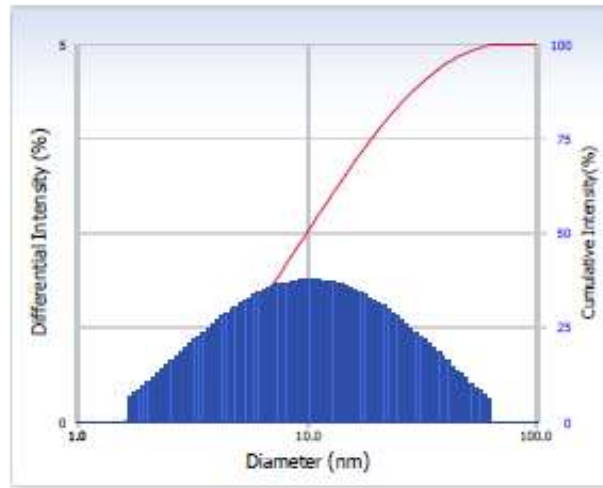


Figure 2 DLS spectrum of MgO-Chitosan-pluronic F-127 nanocomposites

3.3 Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum of MgO-Chitosan-pluronic F-127 nanocomposites is shown in Fig.3. The chitosan main characteristics peaks are, the broad -OH and -NH with hydrogen bond presence at: 3450 cm^{-1} and 1634 cm^{-1} , revealing the amide I group (C-O stretching along with the N-H deformation mode), 1383 cm^{-1} is due to the COO⁻ group of carboxylic acid salt, 10476 cm^{-1} is attributed to the C-O-C bonds in glucose circle (Tian et al., 2003). The peaks Pluronic F-127 molecules have a C-H bending vibration of 844 cm^{-1} . The metal-oxygen peaks found in the wavenumber range of between 400 and 800 cm^{-1} , strongly confirm the stretching vibrations of Mg-O (Choudary et al., 2003, Ai et al., 2012), while the Mg-O stretching bands are observed at: 630 and 519 cm^{-1} . From the results, it was decided that chitosan and pluronic F-127 can form a strong and high intermolecular hydrogen bond with MgO.

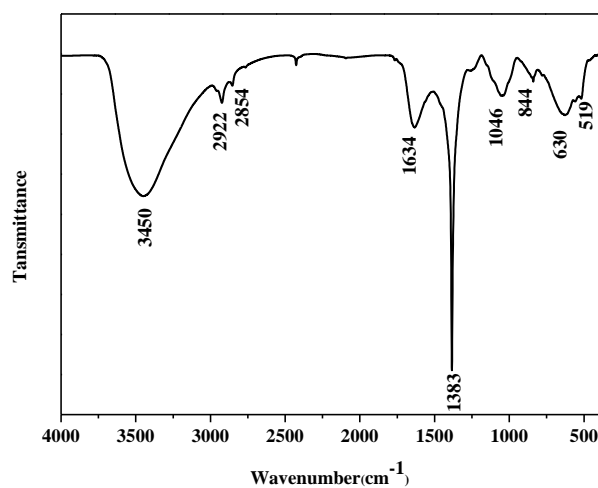


Figure 3 FTIR spectrum of MgO-Chitosan-pluronic F-127 nanocomposites

3.4 UV-vis spectroscopy

The absorbance of the sample depends on several factors, such as band gap, oxygen deficiency, surface roughness, and impurity centers. The excitonic peaks are observed at around 299 nm for MgO-Chitosan-pluronic F-127 nanocomposites (Fig. 4).

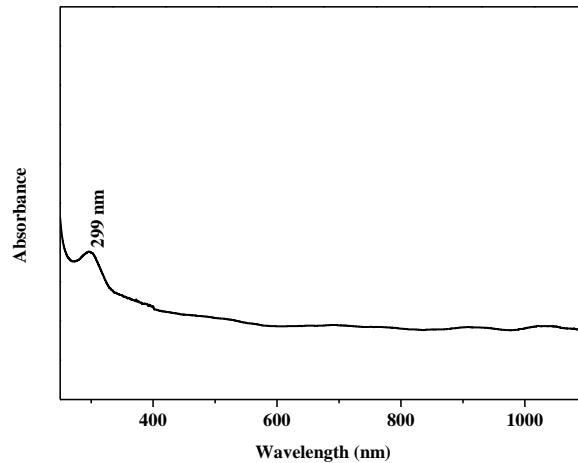


Figure 4 UV-Vis spectrum of MgO-Chitosan-pluronic F-127 nanocomposites

3.5 Photoluminescence spectroscopy (PL)

The PL spectra of the MgO-Chitosan-pluronic F-127 nanocomposites are shown in Fig.5. The MgO-Chitosan-pluronic F-127 nanocomposites are excited at a wavelength of 325 nm. The PL emission values for MgO were observed at 366 nm, 398 nm, 417 nm, 440 nm, 479 nm and 525 nm, which correspond to the peaks observed in the UV and visible regions due to the surface. The near-band edge emission peaks are located at 366 nm and 398 nm for MgO-Chitosan-pluronic F-127 nanocomposites. This is attributed to the recombination of free exciton-exciton collision processes. The violet emission peak is observed at 417 nm, which reveals the band to band transition. The 440 and 479 nm emission peaks are due to the recombination of electrons with oxygen vacancies. The green emission at 525 nm is due to the oxygen vacancy.

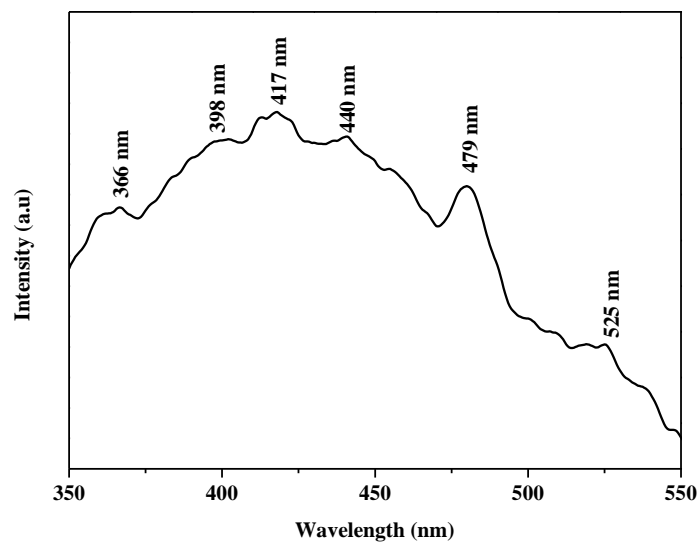


Figure 5 PL spectrum of MgO-Chitosan-pluronic F-127 nanocomposites

3.6 Antimicrobial activity

The green synthesis of MgO-Chitosan-pluronic F-127 nanocomposites was tested for antibacterial activity against gram positive bacteria (*Bacillus subtilis*) and gram negative G- (*Proteus vulgaris*) strains using the agar well diffusion method, as shown in Fig. 6. The MgO-Chitosan-pluronic F-127 nanocomposites exhibit greater antibacterial activity than the standard antibiotics, Amoxicillin pharmaceutical formulation. The Zone inhibition of bacterial cells may be due to disturbances of the cell membrane but is mainly due to the combination of various factors such as reactive oxygen species (ROS) and the release of Mg^{2+} ions. Bacteria lose viability. Finally, bacterial cells die.



Figure 6 Antibacterial activity of MgO-Chitosan-pluronic F-127 nanocomposites

Conclusion

In summary, MgO-Chitosan-pluronic F-127 nanocomposites were prepared by the green method using the *Amomum subulatum* Roxb extract method. The X-ray diffraction study confirmed that the prepared particles were of the cubic structure. The MgO-Chitosan-pluronic F-127 nanocomposites sample absorption spectra have sharp edge peaks at 299 nm from the UV-Vis spectra. Photoluminescence studies showed that the MgO-Chitosan-pluronic F-127 nanocomposites obtain various defects, such as zinc vacancies and oxygen vacancies. The antibacterial activity studies used *Bacillus subtilis* and *Proteus vulgaris* bacterial strains for MgO-Chitosan-pluronic F-127 nanocomposites NPs.

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