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Microwave synthesis and characterization Mg(OH)₂/Gr nanocomposites in the presence of sodium dodecyl sulfate (SDS) as a stabilizer agent

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ABSTRACT

This work reports, microwave synthesis and characterization Mg(OH)₂/Gr nanocomposites in the presence of sodium dodecyl sulfate (SDS) as a stabilizer agent. Mg(OAc)₂·4H₂O and graphene powder have been used as starting reagents. Mg(OH)₂/Gr nanocomposites synthesized by microwave method, particle size distribution and characterization of products were done by X-ray diffraction (XRD), Atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Uv-Vis and IR spectroscopy. The results indicated that the synthesized Mg(OH)₂/Gr nanocomposites have a pure crystallinity structure with average size 75–110 nm.

1. Introduction

Since early 2008, graphene and nanomaterials based on graphene have extensively developed fundamentally and practically for use in optics and optoelectronics. Graphite oxides demonstrate considerable variations of properties depending on degree of oxidation and synthesis method and has been used for the synthesis of noble metal nanocomposites which have found various applications as antimicrobial material, sensors, electrochemical biosensors, catalysis, medicines, electronics, and so on [1–6]. Among the various methods developed for the preparation of metal oxide nanostructures researchers have exploited the Magnesium oxide (MgO) nanoparticles [7], the synthesis of various nanoparticles of metals and oxides, which employ the thermal decomposition of metal-surfactant complexes in hot surfactant solution in several applications including smart windows, photo catalyst, and active layer for gas sensors [8–10]. Various strategies have been used for the synthesis of MgO nanoparticles featuring sol-gel techniques, evaporation, electrodeposition, sputtering, and evaporation [11–15]. Generally, the tetragonal phase of metal oxides is preferentially formed relative to the monoclinic phase during the crystallization of amorphous hydrous metal.

Application of graphene-based nanomaterials in optic and optoelectronic instruments such as photodetectors, transparent conductive electrodes, photovoltaics, phototransistors has been studied. In addition, they have been used in ultrafast lasers as saturable absorbers and in biological and photocatalytic purposes [16–18]. In recent years, many studies have been conducted on the use of metal oxides such as Mg/CuO [19], Mg(OH)₂/CNT [20] in various applications. MgO is considered as semiconductor and crystallized in cubic structure similar to sodium chloride crystals [21–24]. Microwave approach is attractive in industrial applications due to its simplicity and cost effectiveness to obtain highly pure products in facile method. Accordingly, this method has been employed for the synthesis of metal oxide nanostructures and the effect of time and intensity of irradiation on morphology, purity and process

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conditions were thoroughly studied by chemists. In the present work, nanocomposites of $\text{Mg}(\text{OH})_2/\text{Gr}$ were successfully synthesized via microwave method at 600 w for 4 min. Then, the effect of time and irradiation power on the morphology of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites were investigated. Finally, the optical efficiency of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites was studied by Uv-Vis spectroscopy in the presence of sodium dodecyl sulfate (SDS) as a stabilizer agent.

2. Experimental section

2.1. General

All the chemical reagents used in our experiments were of analytical grade and were used as received without further purification. The annealed products were brown in color and taken for characterizations. The phase identification and purity of the annealed materials were tested by powder XRD patterns by the detail [Rigaku D-max C III, X-ray diffractometer using Ni-filtered $\text{Cu K}\alpha$]. Microscopic morphology of products was visualized by SEM (LEO 1455VP). Transmission electron microscopy (TEM) images were obtained on a Philips EM208 transmission electron microscope with an accelerating voltage of 200 kV. Infrared spectra were recorded on Perkin-Elmer Spectrum 100 FT-IR spectrometer. Room temperature Uv-vis properties were studied on a Perkin- Elmer (LS 55) fluorescence spectrophotometer.

2.1.1. Synthesis of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites

In this work, $\text{Mg}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$, graphene powder and sodium dodecyl sulfate (SDS) as a stabilizer agent powder were used as the starting materials. $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites were prepared in a simple and fast microwave irradiation. At first, 0.21 g $\text{Mg}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ along 0.44 g NaOH in 100 mL H_2O and 0.4 g graphene along 0.3 g sodium dodecyl sulfate (SDS) in 10 mL $\text{H}_2\text{O}/\text{EtOH}$ solution were dissolved and stirred for 45 min at 60°C separately. Then their container were mixed and was stirred at 60°C for 60 min. The obtained solution was stirred in ultrasonic therm (60 w) for 1 h and then the mixture was loaded into a microwave teflon container, and the reaction was performed in a microwave digestion system under various conditions. After thermal treatment at 600 w for 4 min, the system was allowed to cool to room temperature naturally, and the obtained precipitate was collected by filtration, and then washed with absolute ethanol and distilled water for several times. The final product was dried in vacuum at 60°C for 24 h.

3. Results and discussion

The produced nanocomposites were characterized by SEM, TEM, Uv-Vis and XRD. The crystalline structure, phase identification and purity of as-prepared nanocomposites were determined by XRD. Fig. 1a and b depicts XRD patterns of graphene structure and $\text{Mg}(\text{OH})_2/\text{Gr}$ obtained from microwave method, respectively. As seen in this figure, extremely sharp peaks have been appeared in the XRD of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites which indicate to a fine nature obtained through the microwave method in 600 w for 4 min.

All diffraction peaks can be assigned to the pure crystalline structure of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites. Scanning electron microscopy (SEM) was employed to study the morphology of samples and the distribution of particles. SEM image of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites prepared in 600 w for 4 min is shown in Fig. 2. It reveals that increasing the time of irradiation above 4 min lead to formation of agglomerated nano particles.

Fig. 3 shows dynamic light scattering (DLS) for $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites according to study the NC hydrodynamic sizes, all measurements were carried out at 25°C DLS data image of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites, the DLS experiments were performed using the Brookhaven Instruments 90 Plus particle size analyzer in Iran.

Morphology and particle's distribution was optimized in 600 w for 4 min. We anticipated that calcination of samples with different

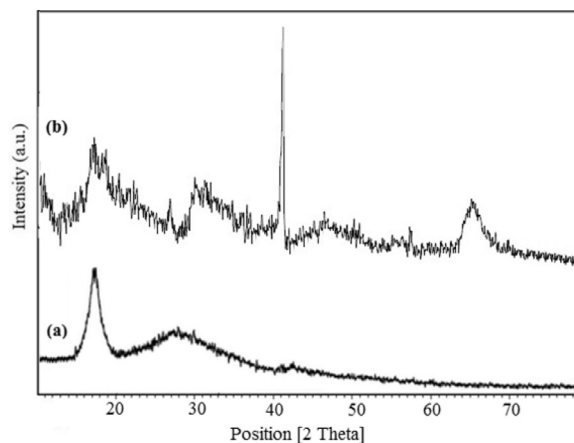


Fig. 1. XRD patterns of (a) graphene structure, (b) $\text{Mg}(\text{OH})_2/\text{GrO}$.

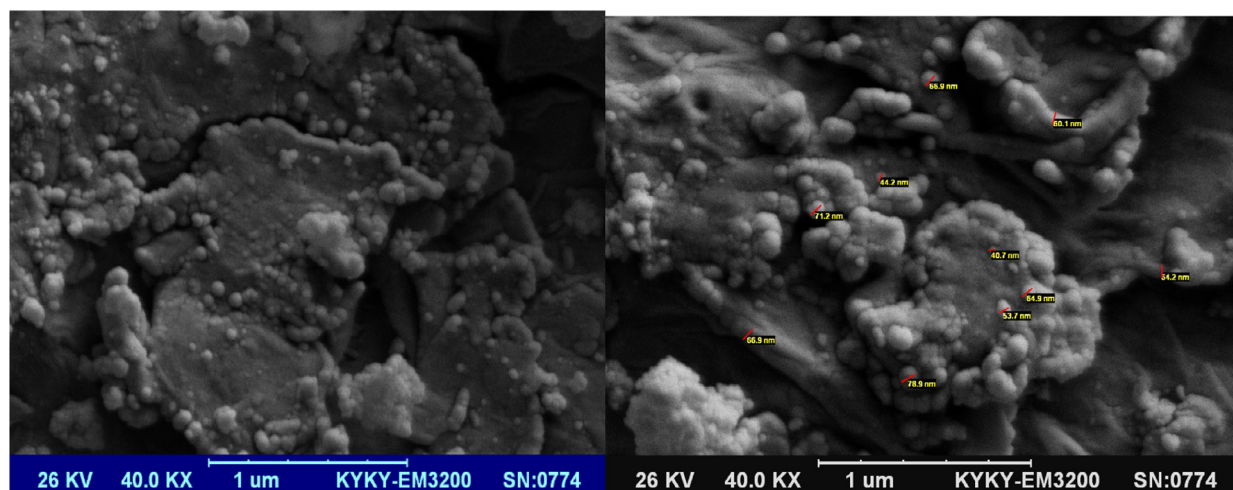


Fig. 2. SEM image of $\text{Mg}(\text{OH})_2/\text{GrO}$ nanocomposites prepared in 600 w for 4 min.

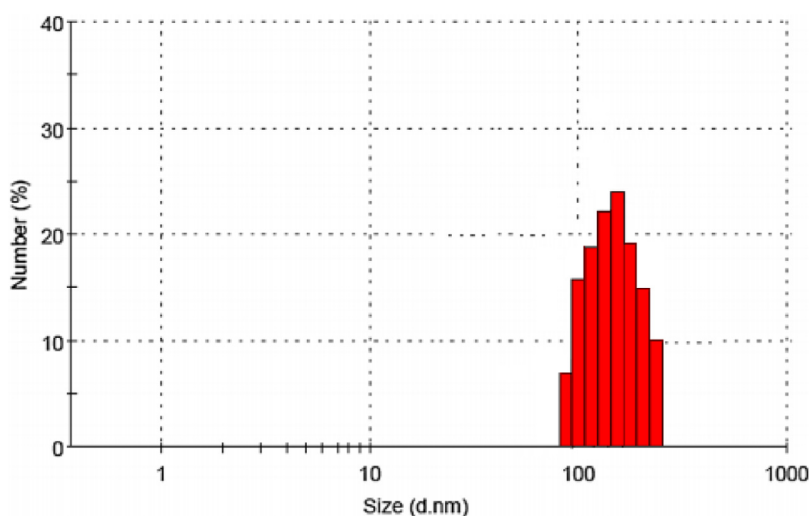


Fig. 3. DLS data image of $\text{Mg}(\text{OH})_2/\text{GrO}$ nanocomposites.

morphologies led to formation of products with homologous sizes. TEM images were taken in order to elucidate the details of morphology and particle's distribution of samples.

To prepare the TEM sample, the powder was dispersed in highly pure ethanol via ultrasonic equipment for 15 min. TEM image of $\text{Mg}(\text{OH})_2/\text{Gr}$ nanocomposites synthesized by microwave method in 600 w for 4 min, is shown in Fig. 4. It reveals that particle size of

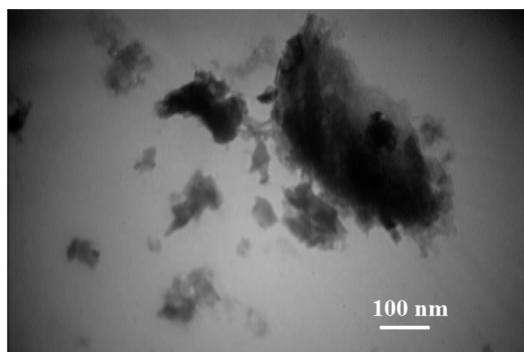


Fig. 4. TEM image of $\text{Mg}(\text{OH})_2/\text{GrO}$ nanocomposites synthesized by microwave method in 600 w for 4 min.

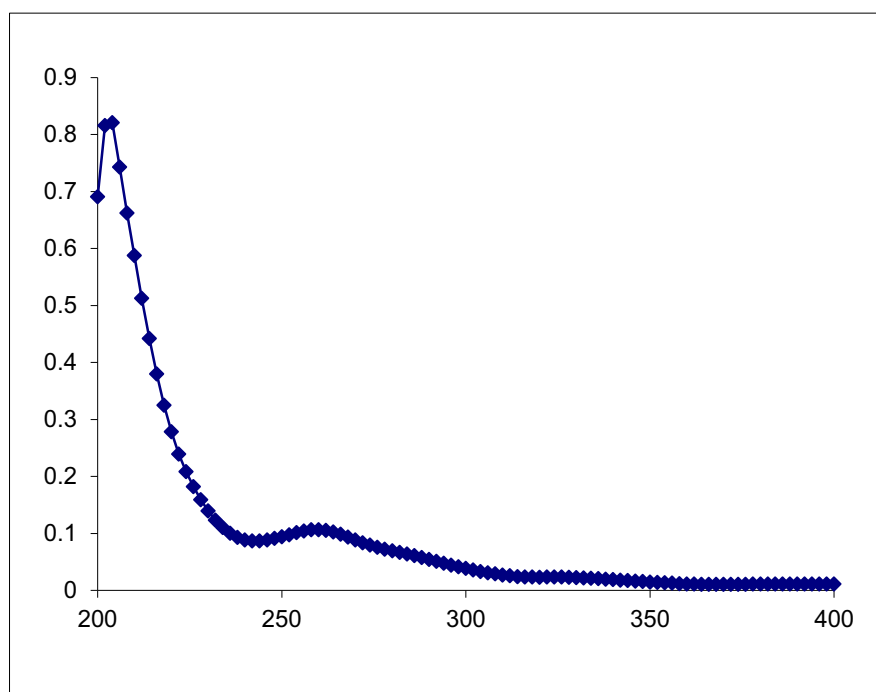


Fig. 5. UV-VIS of Mg(OH)₂/GrO nanocomposites.

nanocomposites are within 75–110 nm which is quite consistent with the results obtained from SEM images.

UV-VIS spectroscopy were used to study the optical properties of Mg(OH)₂/Gr nanocomposites as shown in Fig. 5. The broad peak around 268 nm implies to the quantum confinement of particles [25].

The FT-IR spectrum of Mg(OH)₂/Gr nanocomposites synthesized at 600 W for 4 min after calcination is shown in Fig. 6. In this figure the sharp peak at 3697 cm⁻¹ is attributed to free Mg(OH)₂/Gr (without H-bonding) and strong broad peak at 3447 cm⁻¹ can be

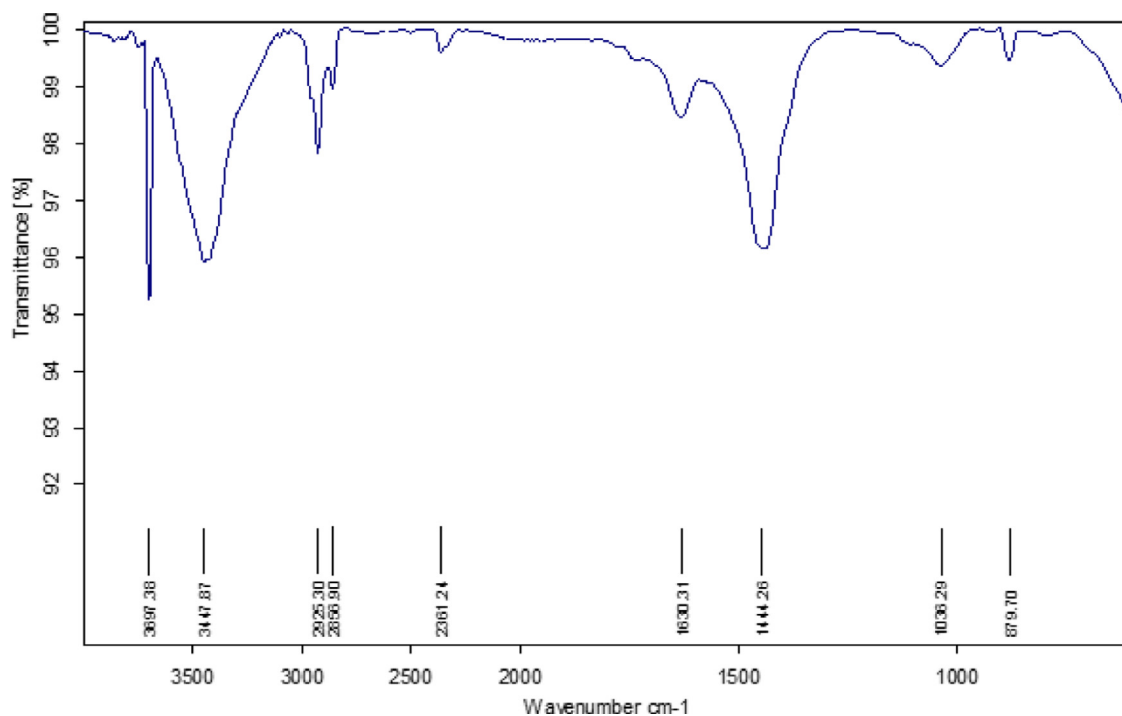


Fig. 6. FT-IR spectrum of Mg(OH)₂/GrO in 600 w for 4 min.

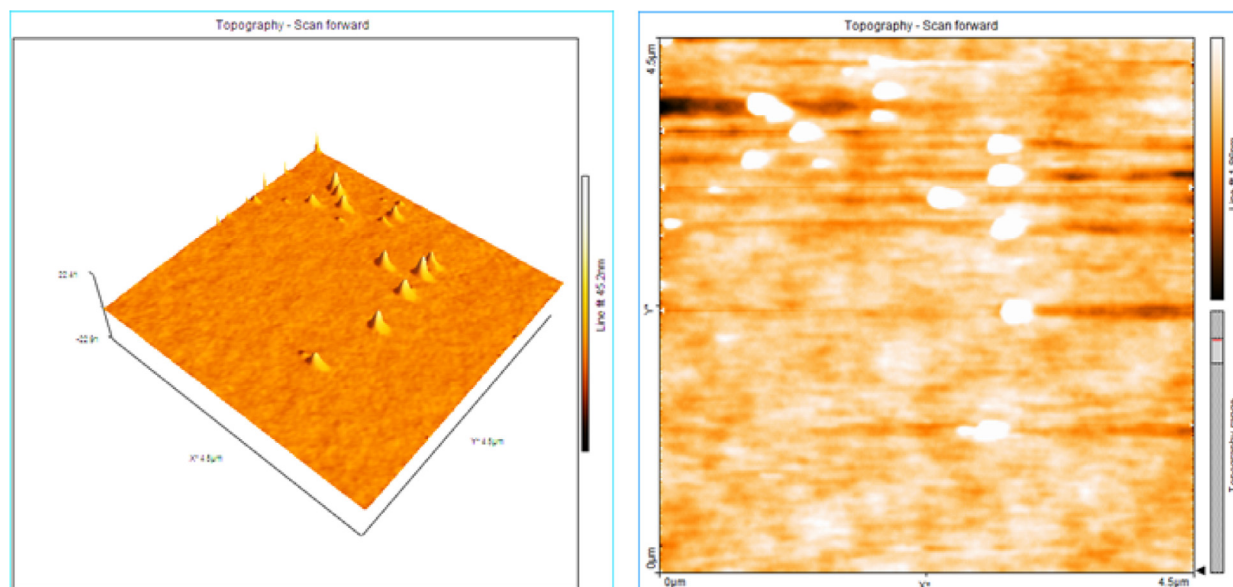


Fig. 7. AFM image of the $\text{Mg(OH)}_2/\text{GrO}$ nanostructures synthesized.

related to stretching vibration of the O–H band in $\text{Mg(OH)}_2/\text{Gr}$ molecules (with H-bonding). The stretching bands within the range of $600\text{--}1200\text{ cm}^{-1}$ is associated with the vibrations of metal–oxygen and metal–carbon, according to the results of Pramanik [28]. A weak peak centered at 1630 cm^{-1} is assigned to stretching vibration of the C–H band of Graphene-H in $\text{Mg(OH)}_2/\text{Gr}$ molecules [26–31].

Atomic force microscopy (AFM) or scanning force Microscopy (SFM) is a very-high-resolution type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The AFM image of $\text{Mg(OH)}_2/\text{Gr}$ nanostructure is shown in Fig. 7. The images show that the surface of the sample is smooth and flat.

4. Conclusion

In this manuscript, $\text{Mg(OH)}_2/\text{Gr}$ nanocomposites have been successfully synthesized through microwave method, after irradiation in 600 w for 4 min. We investigated the influence of the power and time on the morphology of $\text{Mg(OH)}_2/\text{Gr}$ nanocomposites. The characteristics of products were determined by using XRD, SEM, Uv-Vis, AFM and TEM analysis. The XRD results indicated that pure cubic $\text{Mg(OH)}_2/\text{Gr}$ nanocomposites without any impurities could be obtained after irradiation in 600 w for 4 min in the presence of sodium dodecyl sulfate (SDS) as a stabilizer agent. The optical property of $\text{Mg(OH)}_2/\text{Gr}$ nanocomposites shows the emission peaks at 258 nm.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:[10.1016/j.onano.2018.100028](https://doi.org/10.1016/j.onano.2018.100028).

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