The Effect of Transition Metals Incorporation on the Structural and Magnetic Properties of Magnesium Oxide Nanoparticles

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**Abstract**

Pure and doped magnesium oxide nanoparticles were successfully synthesized employing a sol-gel process. The synthesized nanoparticles were characterized by thermal differential analysis, X-ray powder diffraction, transmission electron microscopy, scanning electron microscopy, energy-dispersive X-ray spectroscopy, and vibrating sample magnetometer. X-ray diffraction patterns confirmed the crystallization of MgO structure and correspondingly ratified that the transition metal atoms were incorporated into the MgO host lattice. The crystallite size decreases as the concentration of dopants were augmented. TEM images showed that the particles of pristine magnesium oxide were embedded in the sheet matrix of the graphene-like layer with a size of 22.06 nm. The EDS spectra revealed the presence of carbon in pure MgO nanoparticles, while nickel and chromium were distributed in the host lattice. Based on VSM measurements, room temperature ferromagnetism in pristine MgO nanoparticles was confirmed and the presence of either Mg vacancy or carbon atoms. Furthermore, paramagnetic ordering had been observed upon doping. Overall, the prepared MgO-NPs may be found as a potential application in spintronics devices.

**Keywords**

- Diluted Magnetic Semiconductors
- Magnesium Oxide
- Nanomaterials
- Structural Properties
- Transition Metals

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1. **INTRODUCTION**

Recently, the concept of nanotechnology and its nanomaterials is a thriving specialty of research in a variety of fields. This field of science has the potential to breakthrough many applications such as electric and electronic devices, catalysts, optical, magnetic, and biomedical devices [1–3]. On the account of their excellent physicochemical features, high surface area to volume ratio, and distinctive nanosize structure, metal oxides nanomaterials such as TiO$_2$, ZnO, CuO, and MgO is being increasingly applied as an alternative agent in different applications [4, 5]. In the last decades, many studies on the preparation of metal oxides nanocrystals with large surface area and high reactivity have been offered [6]. At present, several types of metal oxides nanoparticles play very important role in numerous areas of physics, chemistry, and materials science. The numbers of metals can form a large miscellany of metal oxides. They involve the structural geometries with an electronic structure that can reveal semiconductor, metallic or insulator character [7]. In practical applications, metal oxides nanoparticles are utilized in the production of microelectronic circuits, optoelectronics, fuel cells, sensors, and piezoelectric devices [8]. MgO is considered as one of the most important among the metal oxides nanoparticles for several reasons such as nontoxicity, and relatively easy to obtain.

Magnesium oxide (MgO) has drawn wide research interest [9]. MgO nanoparticles have exhibited excessive potential as an adsorbent for toxic chemical agents [10]. It possesses unique magnetic, electronic, optical, thermal, chemical, and mechanical properties because of its excellent chemical and physical properties [11]. It will be interesting to know that bulk magnesium oxide is nonmagnetic [12]. However, the nanoparticle size of magnesium oxide and other nonmagnetic oxides such as ZnO and HfO$_2$ show the new route of magnetism [13, 14]. Magnetic ordering demonstrated via these metals

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oxides is acknowledged as $d^5$ ferromagnetism, as they either have filled or empty $d$-$f$ orbitals. This magnetism is supposed to take place as a result of polarization brought by $p$-orbitals, where magnetic polarization arises because of anion and/or cation vacancies [15].

Many methods have been adopted to synthesis MgO-NPs such as sol-gel [16], co-precipitation [3], and hydrothermal method [17]. The sol-gel method is an appropriate method to produce pristine and doped MgO [18]. This technique is known as a method for the formation of inorganic oxides with gel structures, which are converted to solid glass at low temperatures and can be well-defined from a thermodynamic perspective as a relatively stable solid phase formation at a certain temperature, liquid [19].

The sol-gel route has a number of benefits. Due to the handy decontamination of liquids, it is conceivable to harvest materials with high quality [20]. Materials with exceptionally good chemical homogeneity can also be produced, which are highly desirable, especially in the case of complex oxides, because the mixing of components takes place at the molecular level during chemical reactions [16]. Low condensation temperatures are another good feature. The existence of real disadvantages must not be forgotten. Initial starting materials (e.g., metal alkoxides) are somewhat expensive. Difficulties in the usual drying phase during which cracks and significant shrinkage are common problems mentioned earlier [21].

Generally, any impurities of transition metals incorporated in the host of MgO nanocrystals either as a dopant or as a solid solution may cause defects in the form of vacancies, interstitials, and antisites, which are considered the enhanced source of magnetism, electronic, optical, and thermal properties of bare MgO-NPs. The main objective of this work is to unveil the influence of nickel and chromium transition metals incorporation on the crystal structure, magnetic, and morphological properties of the prepared MgO-NPs. To bring a better understanding of the work to the readers, we organize this report as follows: Experimental method, including chemicals used, methodology and essential parameters are given in Section 2. The results and appropriate discussion are offered in Section 3, which is divided according to subsections including Thermal analysis (Subsection 3.1), Structural analysis (Subsection 3.2), Morphological analysis Subsection 3.3, and Magnetic analysis (Subsection 3.4). Finally, Section 4 summarizes the results of this work.

2. EXPERIMENTAL METHOD

2.1. Chemicals Used

Magnesium nitrate hexahydrate [Mg(NO$_3$)$_2$·6H$_2$O] (BDH Chemicals Ltd, England, > 99% purity), nickel nitrate hexahydrate [Ni(NO$_3$)$_2$·6H$_2$O] (Fisher Certified, > 99% purity), Cr(NO$_3$)$_3$·9H$_2$O (CDH, India, >99%), citric acid [C$_6$H$_8$O$_7$·H$_2$O] (CDH, India, >99%) and high quality doubled distilled water were used to prepare pristine and doped MgO by sol-gel method. All reagents were used without any further purifications.

2.2. Samples Preparation

The pristine and doped MgO samples nanoparticles were prepared by the sol-gel method [22]. For the preparation of pristine MgO-NPs, 6.0897 g of Mg(NO$_3$)$_2$·6H$_2$O and 5.253 g of citric acid [C$_6$H$_8$O$_7$·H$_2$O] were dissolved in 50 ml distilled water in two beakers separately. They were stirring for 60 min using a magnetic stirrer (FALC instrument-F91). Then, the citric acid solution was added to Mg(NO$_3$)$_2$·6H$_2$O solution drop by drop. The mixture was kept stirring with a rotation speed of 800 rpm at 80°C until a wet gel formed after 3 hours. The temperature and pH values of the aqueous solution were controlled using a portable multi-parameter meter (BANTE instrument-900P). Then, the white precipitate was aged overnight, washed, and centrifuged (High speed refrigerated centrifuge-GL-12) with a rotation speed of 3000 rpm for 10 min several times to remove the by-products. The magnesium hydroxide gel was dried at 130°C for 3 hours. The dried powder has been calcined at 500°C for 2 hours with 5°C/min as a heating rate using a furnace device (PIF 160/15- Made in Turkey). For the synthesis of doped and co-doped samples, 0.5 M of Mg(NO$_3$)$_2$·6H$_2$O and various amount of transition metal nitrates, Ni(NO$_3$)$_2$·6H$_2$O and Cr(NO$_3$)$_3$·9H$_2$O, were dissolved in 50 ml distilled water. Citric acid [C$_6$H$_8$O$_7$·H$_2$O] has been added to the solution drop by drop. After 3 hours, a gel was formed at a temperature of 80°C. The gel was dried at 130°C for 4 hours and then heat-treated at 500°C for 2 hours. Figure 1 shows the flowchart of the experimental methodology.

Differential thermal analysis (DTA) curve was recorded by Shimadzu (DTA-50) at a heating rate of 10°C/min. The X-ray diffraction patterns were obtained in the range of 30°–90° using Shimadzu-6000 diffractometer with Cu-Kα radiation (λ = 1.5406 Å) and Ni filter. The emission current of 30 mA, accelerating voltage of 40 kV, step size of 0.02° and a scan speed of 2 s/step were used. The experimental diffraction patterns were investigated using x’pert highscore plus (v 3.0e) software. Crystalline phases were identified by the ICDD reference database. Crystal size and crystal defects were evaluated based on Scherrer and Williamson-Hall plot. Furthermore, the whole powder pattern fitting technique (WPPF) was also performed to evaluate the structural parameters of pure and doped samples. The morphology and the chemical compositions of the prepared samples have been revealed using scanning electron microscopy coupled with energy dispersive – X - ray spectroscopy (TESCAN MIRA3-SAMx EDX) and transmission electron microscopy (Philips CM 100). The magnetic properties have been
measured using a vibrating sample magnetometer (Cryogenic VSM).

3. RESULTS AND DISCUSSION

3.1. Thermal Analysis The differential thermal analysis (DTA) curve of the prepared magnesium hydroxide [Mg(OH)$_2$] is illustrated in Figure 2. It is found that the thermal decomposition of magnesium hydroxide undergoes three thermal events. The first one associated with the removal of hygroscopic molecules of water, which are absorbed on the surface of Mg(OH)$_2$ particles. The adsorbing water molecules appear as a small endothermic started at 83°C. The second thermal event occurs at 314°C which represents a loss of hydroxyl ions from the structure of Mg(OH)$_2$ and converted into a distorted MgO structure. This event shows a major endothermic peak. Finally, an exothermic peak was found at 620°C and attributed to the beginning order of MgO particles in a cubic structure. This observation is in good agreement with previous experimental reports [3, 23].

3.2. Structural Analysis XRD analysis offers information about the structural characteristics of the materials as the width, and the intensity of the diffraction peaks depend on lattice strain, crystalline size, and other imperfections. The bare and doped MgO-NPs have been characterized by means of X-ray diffraction (XRD). The XRD patterns of the prepared samples are given in Figure 3(a). The diffraction peaks of samples are indexed as (111), (200), (220), (311), and (222), which is matched with ICDD PDF (no.075-0447). The samples exhibit the reflection corresponding to the cubic MgO phase having space group Fm-3m (#225). The results are in line with earlier reports [3, 23]. All patterns are in a single-phase, with no diffraction peaks from related oxides. Figure 3(b)
exhibits the variation of XRD intensity and peak position along (200) plane with 20 values from 41° to 45°. One can observe a heterogeneous slight shift in the peak position towards the high angles upon doping. For pristine structure, the peak is positioned at 42.69°, while for 5% Cr and 5% Ni doping, the peaks are shifted to higher 20 positions at 42.77° and 42.79°, respectively. For the Mg_{0.95}Ni_{0.05}Cr_{0.05}O and Mg_{0.95}Ni_{0.02}Cr_{0.02}O samples the peaks are at 42.71° and 42.74°, respectively. It is also evident from Figure 3(b) that the intensity of the XRD peak along (200) plane gradually increased for Ni doping while it is decreased for Cr substitution. This observation may indicate that the TM ions successfully occupy the lattice site rather than interstitial ones. This statement is in line with previous experimental report [24, 25]. The slightly shifted peak position towards higher 20 values can be ascribed to the ionic radius difference between Ni, Cr, and Mg ions. This trend is consistent with the earlier published report [26]. The broadening of XRD reflection indicates that pristine and doped MgO-NPs are of small size. The average crystallite size (D) of the pristine and doped MgO-NPs was estimated using Scherrer’s formula along (200) and (220) planes [27].

\[ D = \frac{\lambda K}{\beta \cos \theta} \]  

where D is the crystallite size, \( \lambda \) the X-ray wavelength of the copper anode (1.5406 Å), \( k \) the shape factor (\( k = 0.9 \)), [28], \( \beta \) the full width at half maximum (FWHM) of the diffraction peaks, and \( \theta \) the Bragg diffraction angle. The broadening of the Bragg peak is a combination of both samples and instruments. To isolate these contributions, it is crucial to achieve a diffraction pattern from the line broadening of standard reference material to correct the instrumental broadening. Silicon powder was utilized to correct this influence. The instrument peak broadening of the studied MgO-NPs can be rectified according to Gaussian (strain effect), and Lorentzian (size effect) distributions [29].

For Lorentzian:

\[ \beta = \beta_{\text{obs}} - \beta_{\text{inst}} \]  

and for Gaussian:

\[ \beta^2 = \beta_{\text{obs}}^2 - \beta_{\text{inst}}^2 \]  

where \( \beta_{\text{obs}} \) is the observed peak width, and \( \beta_{\text{inst}} \) the instrumental broadening. When only taking into account the strain effect for the line broadening, the strain (\( \varepsilon \)) can be predicted as given below [30].

\[ \varepsilon = \frac{\beta}{4 \tan \theta} \]  

Scherrer formula has some drawbacks since the impact of strain broadening is not measured. Williamson–Hall (W-H) plot delivers information about crystallite size and lattice strain. Therefore, W-H plot was adopted to estimate the lattice strain and crystallite size through the following relation [5]:

\[ \beta \cos \theta = (k \lambda / D) + 4 \varepsilon \sin \theta \]  

Crystallite size and microstrain are measured from y-intercept and slope, respectively. It has been stated in the literature that a negative slope in the plot designates the presence of compressive strain, while positive slope shows the possibility of tensile strain (Figure 4). For pristine MgO-NPs, the obtained crystallite size is between 4 and 4.8 nm. This result is in good agreement with previous experimental calculations [31]. The crystallite size of the doped samples is decreased slightly as compared to the pure state. For all samples, one may perceive that the crystallite sizes measured by the W–H plot are smaller than those calculated by Scherrer expression since the latter does not uncover the influence of lattice defects on the XRD peaks broadening (Table 1). It is obvious from the scattered points in Figure 4 that the data do not fully obey the W–H formulation for different samples, where they demonstrate a non-monotonous increase of \( \beta \cos \theta \) versus 4\( \varepsilon \)\( \sin \theta \). This indicates that the broadening of Bragg peaks with respect to different Bragg reflections are anisotropic and triggered by anisotropic microstrain [5]. Thus, the microstrain anisotropy observed in these nanoparticles is attributed to the occurrence of point defects.
Williamson-Hall Plot for (a) pristine, (b) 5% Ni, (c) 4% Ni, 1% Cr, (d) 2% Ni, 3% Cr, and 5% Cr doped MgO-NPs. The last panel represents the crystal structure of the doped MgO-NPs where orange, red, green and blue represents the Mg, O, Ni, and Cr atoms.

Rietveld refinement was executed to study in detail the crystalline structure, counting both the structural (site occupancy, cell parameters, atomic position, etc) and microstructural parameters (crystallite size, microstrain, etc). The main objective of utilizing the Rietveld analysis is to reduce the difference between the calculated and observed powder diffraction pattern, see Figure 5. The minimization was performed by means of the agreement indices parameters; $R_w$ (weigh pattern factor), $R_B$ (Bragg’s angle), $R_{exp}$ (expected error factor), and the goodness of fit (GOF). These parameters along with the occupancy of the atoms are listed in Table 2. The values of these parameters indicate that derived samples are of high quality, and refinements are effective [5]. Observed and calculated values were good matching as can be recognized from figures. Refined atomic positions indicate the presence of Mg vacancy associated with the pristine structure. Cation vacancy may induce room-temperature ferromagnetism. Furthermore, the Mg$_{0.95}$Ni$_{0.05}$O and Mg$_{0.95}$Ni$_{0.04}$Cr$_{0.01}$O samples may show the existence of an oxygen vacancy.

Lattice parameters decrease as the doping concentration increases. As presented in Figure 6, a decrease in lattice parameters relies on the ionic radius, which can be happened if the ionic radius to some extent smaller than that of the host lattice. One can perceive a slight shift of the position in the diffraction peaks representing a slight variation in lattice parameters. The values of the refined lattice parameters are in good agreement with previous experimental results [32]. The slight variation of lattice parameters with doping may indicate the existence of point defects. Dislocation density augments with impurities where it is anticipated to disturb the physicochemical properties of the

| Compounds          | Density (g/cm³) | W-H plot | Scherrer |
|--------------------|----------------|----------|----------|
| Pure MgO           | 3.5124         | 0.6      | 1.3      | 0.0625   |
| Mg$_{0.95}$Ni$_{0.05}$O | 3.7440         | -1       | 3        | 0.111    |
| Mg$_{0.95}$Cr$_{0.05}$O | 3.6557         | -1       | 3.6      | 0.0771   |
| Mg$_{0.95}$Ni$_{0.04}$Cr$_{0.01}$O | 3.6728        | -1       | 3.2      | 0.0976   |
| Mg$_{0.95}$Ni$_{0.03}$Cr$_{0.02}$O | 3.7376        | -0.4     | 3.6      | 0.0771   |
Table 2: Rietveld refinement result of pure and doped MgO-NPs calcined at 500°C

| Compound                  | R_{exp} % | R_{B} % | R_{wp} % | GOF   | Site occupancy (SOF) | Lattice parameter (Å) |
|---------------------------|-----------|---------|----------|-------|----------------------|-----------------------|
| Pure MgO                  | 13.5093   | 12.02703| 16.15499 | 1.43003| O 1.0                | 4.226                 |
| Mg_{0.95}Ni_{0.05}O      | 7.4562    | 7.23128 | 9.70809  | 1.69524| Mg 0.958266          | 4.221                 |
| Mg_{0.95}Cr_{0.05}O      | 11.4525   | 10.16903| 13.80209 | 1.45239| Cr 0.043787          | 4.226                 |
| Mg_{0.95}Ni_{0.05}Cr_{0.01}O | 8.2652 | 8.26998 | 11.38103 | 1.89604| Ni 0.019099 Cr 0.029068 O 1.0 | 4.225                 |
| Mg_{0.95}Ni_{0.02}Cr_{0.03}O | 9.01573 | 8.36426 | 11.52438 | 1.63393| Mg 0.948252 Ni 0.019099 Cr 0.029068 O 1.0 | 4.221                 |

Figure 6. (a) Comparison of average lattice parameters (Å) and crystallite size (nm), (b) Comparison of average dislocation density (10^{15} / m^2) and strain% as a function of doping concentration

The elemental compositions of pure and doped MgO-NPs are determined with energy-dispersive X-ray

![Diagram](image)
spectroscopy (EDS), as displayed in Figure 8. EDS is a surface-sensitive technique and it typically offers elemental composition existent up to few layers from the surface. In stoichiometric MgO, the Mg/O atomic percentage composition is 50/50 [32]. The EDS spectrum from the MgO synthesized by the aqueous method shows carbon impurities in addition to the expected magnesium and excess oxygen (Figure 8(a)). Thus, an excess amount of carbon and oxygen envisages the presence of organic citric acid in pure MgO-NPs. Furthermore, the existence of dopants in the MgO host lattice has also been confirmed, see Figure 8(b). Both nickel and chromium are detected with a minor amount of carbon compared to the bare state. This can be perceived in the TEM images.

Figure 9 shows the transmission electron microscope (TEM) images of pure MgO and TM doped MgO. TEM images have been adopted to visualize the size and shape of the nanoparticles. Since the nanoparticles are prepared without any surfactant, agglomerated ions persist in the prepared sample. This is observed clearly in the TEM image, where the particles of pristine magnesium oxide are embedded in the sheet matrix of the graphene-like layer with a size of 22.06 nm, in Figure 9(a, c). The origin of the graphene-like layer is due to the use of citric acid as a chelating agent. A similar observation has been reported by Singh et al. [34]. The TEM image of the prepared doped nanoparticles is shown in Figure 9(b). The later shows a clear decrease in particle size (12.65 nm) with better dispersion (less amount of carbon layer) compared to pristine crystal structure. This is confirmed by the EDS quantitative analysis. One may also observe that crystallite size from XRD line profiles is smaller than the particle size achieved from TEM analysis. This is related to the agglomeration of lots of small domains to form a particle [5].

3.4. Magnetic Analysis

The magnetic properties of pure and doped MgO-NPs have studied at room temperature from magnetic-filed (M-H) hysteresis loops, as shown in Figure 10. For pure MgO-NPs, the hysteresis loop shows a clear ferromagnetic ordering at room temperature. Since bulk MgO is a diamagnetic insulator, the origin of this demeanor at the nanoscale can be ascribed to the existence of defects. Extensive theoretical and experimental studies show that Mg vacancy is possibly the origin of the magnetism in pristine MgO host lattice [13, 35, 36]. Nanograins of MgO are ferromagnetic, which is ascribed to the formation of cation vacancies at the surface [37]. In its bulk form, Mg and O atoms are periodically arranged conserving octahedral symmetry [15]. The periodicity may be broken in MgO nanocrystals due to the high surface area of the nanoparticles. Owing to the disorder of symmetry on the surface, Mg and O atoms are roughly bonded to the surface, and therefore easily disconnected from the surface. This may affect Mg and/or O vacancies on the surface of MgO nanoparticles. Even if we assume that MgO-NPs contain both Mg and oxygen vacancies, it is the Mg vacancy which is introducing ferromagnetic ordering. Theoretically, it is showed that Mg vacancy encourages spin polarization of 2p electrons of oxygen atoms around Mg vacancy, causing ferromagnetism in MgO host lattice [36]. Accordingly, it is the Mg vacancy that has a significant influence on the detected room temperature ferromagnetism [13]. In our study, the presence of Mg vacancy in MgO-NPs has been confirmed by Rietveld refinement, see Table 2. Other studies show that carbon atoms doped MgO crystal may induce room temperature ferromagnetism [38, 39]. Based on our quantitative EDS calculations, pristine MgO-NPs contain high inclusions of carbon atoms. Thus, carbon atoms may enhance the ferromagnetic ordering of the defected MgO-NPs. This observation may lead to more theoretical and experimental studies to unravel the cause of ferromagnetic ordering in diamagnetic MgO nanoparticles. We believe that the presence of complex defects (V_{Mg}–Carbon) is responsible for such intriguing behavior. The calculated magnetic moment of pristine MgO-NPs is 0.39 emu/g, while the coercive field (H_{c}) and remnant magnetization (M_r) are 55 Oe and 0.03
Emu/g, respectively. These values are in acceptable agreement with the previous experimental results [13]. Upon doping, the hysteresis loops appear to be linear indicating the paramagnetic ordering at room temperature. The paramagnetic ordering of 5% Ni2+ doped MgO is in good agreement with the previous experimental report. This is consistent with the non-interacting Ni2+ or Cr3+ ions that are occupying the substitutional Mg2+ sites. Narayan et al. [40] suggested that the presence of the cation/anion vacancy may mediate ferromagnetic ordering in Ni-doped MgO. In our study, the presence of Mg vacancy and carbon inclusions in doped samples are less compared to that of pristine MgO structure. This observation has been long-established by Rietveld analysis and EDS measurements (Figure 8 and Table 2). The prepared MgO-NPs can be found a potential application in spintronics devices due to the presence of room temperature ferromagnetic ordering.

4. CONCLUSIONS

In the present work, pure and doped MgO-NPs have been synthesized via a facile sol-gel method. The incorporation of transition metal ions was confirmed by the X-ray diffraction technique. These nanoparticles exhibit the rocksalt phase of MgO without the presence of secondary phases. The morphologies and the elemental composition of the prepared samples were studied by TEM and FSEM coupled with the EDX technique. The TEM findings supported the XRD results indicating the nanoparticles decrease in size with increasing doping content. The hysteresis loop of the pure MgO nanoparticles shows a ferromagnetic ordering at room temperature, which can be ascribed to the presence of either Mg vacancy or carbon atoms. Furthermore, complex defects may also play a role in such an intriguing phenomenon. On the other hand, paramagnetic ordering has been observed upon doping. Thus, a very cheap and facile synthesis route is adopted to prepare ferromagnetic MgO nanoparticles that are suitable for magnetic applications.

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چکیده
نانوذرات اکسید منیزیم خالص و دوپ شده با استفاده از یک فرآیند سلولولی با مواد تولید شدند. نانوذرات سنتز شده با تحلیل میکروسکوپ الکترونی و ابزارهای دیگر ارائه شده‌اند. این نانوذرات به میکروسکوپ الکترونی روبشی، میکروسکوپ انتزاعی مغناطیسی، میکروسکوپ الکترونی سطحی، و میکروسکوپ الکترونی سطحی ارائه شده‌اند. نانوذرات اکسید منیزیم با استفاده از طیف‌سنجی ایکس، اتاق و فرم спرماغناطیسی منیزیم و مغناطیسی اتاق و فرم sp و VSM به‌طور کلی، MgO-NP به عنوان یک کاربرد پلاستیکی مطرح می‌شود.