

Research Article

A Study and Comparison of the Preparation of Gadolinium Aluminate Nanoparticles Using γ -Irradiated and Unirradiated Precursors

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The effects of γ -irradiation and the application of different precursors on the formation of gadolinium aluminate (GdAlO_3) nanoparticles (NPs) have been studied in detail. GdAlO_3 NPs were prepared by using different gadolinium-based precursors including gadolinium acetate ($\text{Gd}(\text{CH}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$) and gadolinium nitrate ($\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), while Al_2O_3 and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were used as the source of Al^{3+} . The preparation of GdAlO_3 was carried out by two different methods, solid-state reaction and sol-gel process. To study the effect of γ -irradiation, both irradiated and unirradiated $\text{Gd}(\text{CH}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$ have been tested for the preparation of gadolinium aluminate (GdAlO_3). Notably, $\text{Gd}(\text{CH}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$ did not produce GdAlO_3 in both solid-state and sol-gel processes even after optimizing various parameters, including the application of γ -irradiation. However, single-phase nanocrystalline GdAlO_3 NPs were successfully obtained from the reaction of gadolinium nitrate $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ by a sol-gel process. The formation of NPs has been confirmed by X-ray diffraction analysis (XRD) and Fourier-transform infrared (FT-IR) spectroscopy. The results indicate towards the formation of an orthorhombic perovskite structure of GdAlO_3 in the $Pbnm$ space group. Transmission electron microscopy (TEM) has been employed for the particle-size analysis, which revealed the formation of spherical-shaped nanoparticles with the size range of 50–70 nm. Surface morphology of the sintered pellet was obtained from high-resolution scanning electron microscopy (HR-SEM). Besides, the effect of irradiation with γ -rays on the quality of resultant NPs has also been studied.

1. Introduction

Among various rare-earth materials, perovskite-type rare-earth oxides and aluminates have gained immense interest as a promising class of materials [1]. Due to their high chemical stability, these types of materials have been commonly used in various device-based applications including magnetic, optical, and electronic devices [2, 3]. Particularly, the perovskite aluminates, such as gadolinium aluminum perovskites, are a kind of ceramics based on the Ln_2O_3 - Al_2O_3 system (Ln-lanthanide element) offering advantages of longer lifetimes and higher, polarized cross sections with respect to most other oxide matrices. These materials can be

potentially applied for various optical, electronic, and structural applications [4].

Among these aluminates, gadolinium aluminate (GdAlO_3) belongs to an important class of rare-earth aluminates from the perovskite family of compounds with the ABO_3 structure [5]. This material at the nanoscale level possesses superior powder properties including better phase homogeneity, good sinter ability, and enhanced physico-chemical properties [6]. The synthesis of GdAlO_3 is often challenging and usually carried out by solid-state reaction of Gd_2O_3 and Al_2O_3 at elevated temperatures [7]. However, this technique usually requires extensive mixing of materials or vigorous grinding and high temperatures, which may

negatively impact the microstructure of the resultant material [8]. Therefore, GdAlO_3 has also been prepared using different wet-chemical methods such as, polymerized complex route, combustion synthesis, and sol-gel process [9]. Using these methods, the powder characteristics of the resultant material such as its sinter ability and phase purity can be potentially improved [10].

Notably, during the preparation of aluminates, the method, conditions of preparation, and precursors of the reaction play an important role in determining the morphology and physicochemical properties of the resultant material. These properties can also be affected by doping with certain foreign cations and subjecting to ionizing radiation. Particularly, irradiation of precursors with γ -rays may potentially modify one or more properties of the material in a significant way by creating point defects or by increasing the number of nucleation sites for the formation of product on the surface of reacting materials [11]. Besides, choosing the right precursor during aluminate synthesis is a key factor in forming aluminate NPs with desirable properties.

In this study, approbation of solid-state reaction and sol-gel process for the synthesis of GdAlO_3 NPs was performed in order to compare the efficiency of both methods. The solid-state reaction was performed using gadolinium acetate as the precursor, while in the case of sol-gel technique, apart from gadolinium acetate, gadolinium nitrate has also been used. Moreover, in order to study the effect of γ -irradiation on the properties of resultant NPs, all the precursors used in this study have been irradiated using 100 and 300 kGy γ -irradiation. The properties of NPs prepared from irradiated precursors are also compared with GdAlO_3 NPs obtained from unirradiated precursors.

2. Materials and Methods

2.1. Materials. Gadolinium acetate ($\text{Gd}(\text{CH}_3\text{COO})_3 \cdot x\text{H}_2\text{O}$), gadolinium nitrate ($\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), aluminum nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) and aluminum oxide (Al_2O_3), oleic acid, sodium dodecyl sulfate, benzyl alcohol, and all other organic solvents were obtained from Aldrich chemicals (Steinheim, Germany) and were used without further purification.

2.2. Preparation of Gadolinium Aluminate (GdAlO_3) Nanoparticles

2.2.1. Solid-State Reaction. In these reactions, different ratios of unirradiated gadolinium acetate as Gd^{3+} raw material were mixed with aluminum oxide as Al^{3+} raw material ($0.1\text{Gd}^{3+}/0.9\text{Al}^{3+}$, $0.3\text{Gd}^{3+}/0.7\text{Al}^{3+}$, and $0.5\text{Gd}^{3+}/0.5\text{Al}^{3+}$). To this mixture, a small amount of (one drop) benzyl alcohol and oleic acid/SDS was added. Subsequently, the reaction mixtures were subjected to the calcination process at different temperatures including 600°C , 800°C , and 1000°C for different time periods between 6 and 36 hrs. For the solid-state reactions of irradiated $\text{Gd}(\text{CH}_3\text{COO})_3 \cdot x\text{H}_2\text{O}$, similar conditions were applied, except the addition of 4 drops of benzyl alcohol. All the irradiated samples were calcined at 1000°C for 10 hrs.

2.2.2. Sol-Gel Process

(1) Using Unirradiated Gadolinium Acetate. Prior to use, the unirradiated gadolinium acetate ($\text{Gd}(\text{CH}_3\text{COO})_3 \cdot x\text{H}_2\text{O}$) was dried at 100°C for 1.5 h. Stoichiometric amounts of the dried $\text{Gd}(\text{CH}_3\text{COO})_3$ (1.672 g) were mixed with Al_2O_3 (0.51 g) or $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ [1.896 g]. The mixture was dispersed in ~ 5 – 10 ml distilled water under stirring. Subsequently, 2 ml of oleic acid was added to reaction mixtures. Thereafter, a small amount of gelatin was gradually added under continuous stirring, until a hard transparent solid resin formed. This resultant resin was kept in a furnace at 200°C for 40 min. Finally, the dark gel powder was subjected to further calcination at 1000°C for 10 h.

(2) Using γ -Irradiated Gadolinium Acetate. Prior to use, the mixture of $\text{Gd}(\text{CH}_3\text{COO})_3$ with Al_2O_3 in appropriate ratio is irradiated with 100 or 300 kGy γ -rays. For irradiation, samples were encapsulated under vacuum in glass vials and were exposed to successively increase doses of radiation at a constant intensity using Co-60 γ -ray cell 220 (Nordion International Inc., Ontario, Canada) at a dose rate of 104 Gy h^{-1} . The aforementioned mixture (1.3–1.5 g) was dispersed in 10 ml of distilled water. To this mixture, 2 drops of benzyl alcohol and 2 drops of oleic acid were added under stirring. Thereafter, a small amount of gelatin was added gradually with continuous stirring till a hard transparent gel formed. The gel was left in the furnace at 100°C for 2 h. Finally, the gel powder was further calcined at various temperatures, including at 800°C for 24 h, 850°C for 24 h, and finally at 1000°C for 3 h in order to find the best conditions to synthesize GdAlO_3 nanocrystals by this method.

(3) Using γ -Irradiated Gadolinium Nitrate as Precursor. For this purpose, the gel was prepared using stoichiometric amounts of 100 and 300 kGy γ -irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and 100 kGy $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. First, ~ 2.25 g (100 or 300) kGy $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ was dispersed in 50 ml of 0.2 M acetic acid at 65°C . Separately, ~ 1.87 g 100 kGy $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was dispersed in 50 ml of distilled water at 65°C . Subsequently, this solution is added to the Gd^{3+} solution. The resulting mixture was stirred for 1 hour at 65°C . Thereafter, 2 ml of 1,2-ethanediol was added to the reaction solution as a complexing agent. The solution was concentrated by slow evaporation at the same temperature under stirring for one day, and the Gd-Al-O acetate-nitrate-glycolate sol turned into a white transparent gel. The gel was left to dry for few days. It was then oven-dried at 200°C for 1 h. The resultant gel powder was preheated for 3 h at 800°C in air. Finally, the powder was additionally sintered in air for 10 h at 1000°C .

2.3. Characterization. XRD diffractograms were collected on a Altima IV (Rigaku, Tokyo, Japan) X-ray powder diffractometer using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). Transmission electron microscopy (TEM) was performed on a JEOL (Peabody, MA, USA) JEM 1101 microscope. The samples for TEM were prepared by placing a drop of the primary sample on a holey carbon copper grid and then dried

for 6 h at 80°C in an oven. The SEM images were recorded on a scanning electron microscope (NNL200; FEI). A PerkinElmer spectrum BX FT-IR system was used to measure FT-IR spectra.

3. Results and Discussion

3.1. Solid-State Reaction. The solid-state reaction is one of the most common methods applied for the preparation of rare-earth polycrystalline material including GdAlO₃. These materials are prepared by using solid gadolinium acetate or gadolinium nitrate as precursors. These solid starting materials do not react at mild temperatures and often require very high temperatures (~1500°C). During these reactions, repeated grinding and heating are also applied to accelerate the diffusion of atoms between raw materials. During this study, both irradiated and unirradiated gadolinium acetate and Al₂O₃ were calcined at different temperatures in the range of 600°C to 1000°C. To stabilize the resultant NPs, two types of surfactants were used including oleic acid and SDS.

Initially, to confirm the formation of GdAlO₃ NPs from various solid-state reactions of both irradiated and unirradiated gadolinium acetate precursor, FT-IR spectra of all the products were recorded (data are not provided here). Typically, perovskite-type rare-earth aluminates with ABO₃ structures consist of some characteristic bands in the low-frequency region of 400–1000 cm⁻¹ [12]. However, all the recorded spectra did not show these characteristic bands, which are expected to appear at 670 and 460 cm⁻¹ in the case of GdAlO₃ NPs [8]. Even after optimizing various reaction parameters, including varying the concentration of the reactants (0.1Gd/0.9Al, 0.3Gd/0.7Al, and 0.5Gd/0.5Al), calcination temperature, and type of surfactants, the reactions did not succeed; this was clearly indicated by the IR spectra. This was further confirmed by XRD analysis, as in all the products obtained from different reactions, the XRD pattern indicated the presence of physical mixture of Gd₂O₃ as the major phase and Al₂O₃ besides the presence of some unknown phases (Figure 1). Indeed, after irradiation of gadolinium acetate precursor with γ -rays, no significant effect of γ -irradiation was observed where all these patterns reveal that the major phase is Gd₂O₃ besides Al₂O₃ and unknown minor phases.

3.2. Sol-Gel Process. Among various wet-chemical methods, including polymerized complex route, combustion synthesis, and homogeneous precipitation methods, the sol-gel process is widely applied as it facilitates proper chemical interactions among reacting species of the initial mixture which leads to the evolution of a homogeneous solid-state structure at the atomic level [13]. In this study, GdAlO₃ NPs are prepared using both irradiated and unirradiated gadolinium acetate and Al₂O₃ or Al(NO₃)₂·9H₂O precursors. Initially, IR analysis was performed to evaluate the formation of GdAlO₃ NPs. The results reveal that using Al₂O₃ as the source of Al³⁺ with both irradiated and unirradiated gadolinium acetate precursor did not yield GdAlO₃ NPs. This is clearly indicated by the absence of characteristic

peaks in the range of 400 to 800 cm⁻¹ for the perovskite-type GdAlO₃ NPs, whereas, the application of Al(NO₃)₂·9H₂O with both irradiated and unirradiated gadolinium acetate precursors yielded GdAlO₃ NPs. This is confirmed by the IR spectra (spectra are not provided here) of resultant NPs which displayed characteristic M-O vibration peaks in the range of 850–400 cm⁻¹. IR analysis also revealed that the irradiation does not have significant effect on the formation of resultant NPs as the IR spectra of GdAlO₃ NPs prepared using both irradiated and unirradiated gadolinium acetate precursors are qualitatively identical.

The FT-IR results were found to be consistent with crystallization process observed by the XRD measurements. The absence of the characteristic diffraction peaks of GdAlO₃ in the XRD pattern shown in Figure 2(a) clearly demonstrates that the reaction between unirradiated gadolinium acetate and Al₂O₃ did not result in the formation of GdAlO₃ NPs, whereas, the product obtained from the reaction of unirradiated gadolinium acetate and Al(NO₃)₃·9H₂O produced GdAlO₃ NPs which is confirmed by the X-ray diffraction patterns as shown in the Figure 2(b). The XRD was measured after the product was sintered for 10 h at 1000°C. The XRD result revealed that, after sintering, fully crystalline single-phase GdAlO₃ ceramic was formed. The diffraction pattern indicates the presence of perovskite crystal structure of GdAlO₃ (JCPD card # 01-073-9322) [12]. However, along with perovskite gadolinium aluminates, the formation of the impurity phases is evident. The X-ray line broadening method was used to calculate the crystallite size of the prepared GdAlO₃ nanopowder using the Scherrer formula. The synthesized GdAlO₃ powder was found to be nanocrystalline in nature consisting of a crystallite size of 22 nm.

To study the effect of irradiation on the precursors as well as on the quality of resultant GdAlO₃ NPs, the reactant Gd(CH₃COO)₃ was irradiated with 100 and 300 kGy γ -rays, while Al(NO₃)₃·9H₂O was irradiated with only 100 kGy γ -rays. Notably, the reaction of both irradiated (100 kGy) and unirradiated Gd(CH₃COO)₃ with Al₂O₃ has produced similar results, i.e., did not yield GdAlO₃ NPs. Even the increased intensity (300 kGy) of the irradiation did not have any effect on the formation of the product. This was further confirmed by measuring the XRD pattern of the products obtained from both 100 and 300 kGy irradiated precursors, which did not exhibit any difference in their diffraction pattern (data not shown here). Therefore, to further study the effect of irradiation, another precursor Gd(NO₃)₃·6H₂O was used, which was also irradiated with both 100 and 300 kGy γ -rays. In this case, 100 kGy irradiated Al(NO₃)₃·9H₂O was used as a source of Al ions. These results were found to be consistent with the crystallization process observed by the XRD measurements (Figure 3). According to this analysis, both gels produced fully crystalline phase GdAlO₃ ceramic, where all diffraction patterns in these XRD could be attributed to the perovskite crystal structure of GdAlO₃. All reflections in the diffraction pattern as shown in Figure 2 are in good agreement with ICDD 46-0395, which confirms the formation of GdAlO₃ phase with orthorhombic perovskite structure [9]. The average size of the crystallites calculated from the XRD using the Scherrer formula was

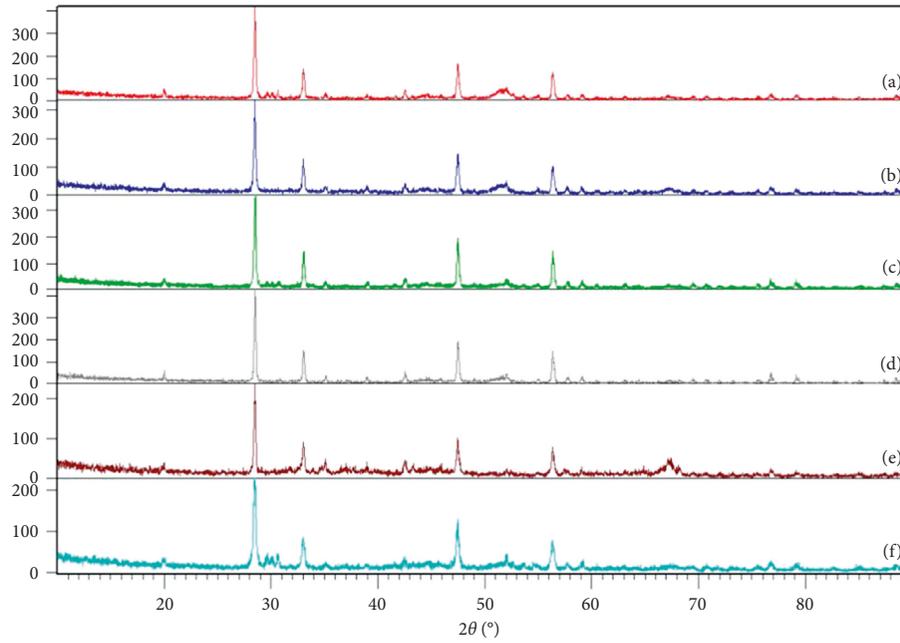


FIGURE 1: X-ray diffraction patterns of selected products gained from solid-state reactions using various ratios of precursors ($n\text{Gd}^{3+}/m\text{Al}^{3+}$) after irradiation at different conditions. (a) $0.3 \text{ Gd}^{3+}/0.7 \text{ Al}^{3+}$ at 1000°C for 10 h, benzyl alcohol + SDS (sodium dodecyl sulfate); (b) $0.3 \text{ Gd}^{3+}/0.7 \text{ Al}^{3+}$ at 1000°C for 10 h, benzyl alcohol + oleic acid; (c) $0.5 \text{ Gd}^{3+}/0.5 \text{ Al}^{3+}$ at 1000°C for 10 h, benzyl alcohol + SDS; (d) $0.5 \text{ Gd}^{3+}/0.5 \text{ Al}^{3+}$ at 1000°C for 10 h, benzyl alcohol + oleic acid; (e) $0.1 \text{ Gd}^{3+}/0.9 \text{ Al}^{3+}$ at 1000°C for 10 h, benzyl alcohol + oleic acid; (f) $0.3 \text{ Gd}^{3+}/0.7 \text{ Al}^{3+}$ at 800°C for 10 h, benzyl alcohol + SDS.

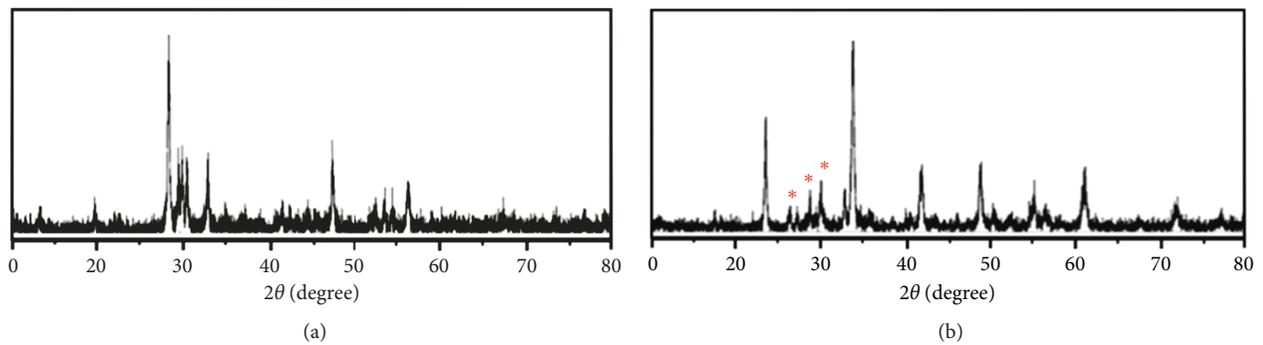


FIGURE 2: X-ray diffraction patterns of products obtained from sol-gel processing using (a) $\text{Gd}(\text{CH}_3\text{COO})_3$ with Al_2O_3 and (b) $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. The impurity phases are marked.

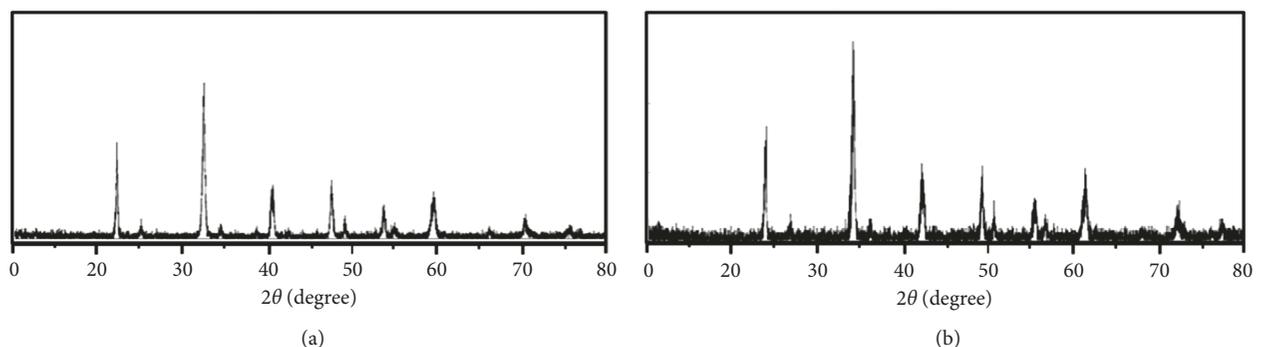


FIGURE 3: X-ray diffraction patterns of GdAlO_3 NPs prepared using (a) 100 kGy irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and (b) 300 kGy irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ in a sol-gel process at 1000°C .

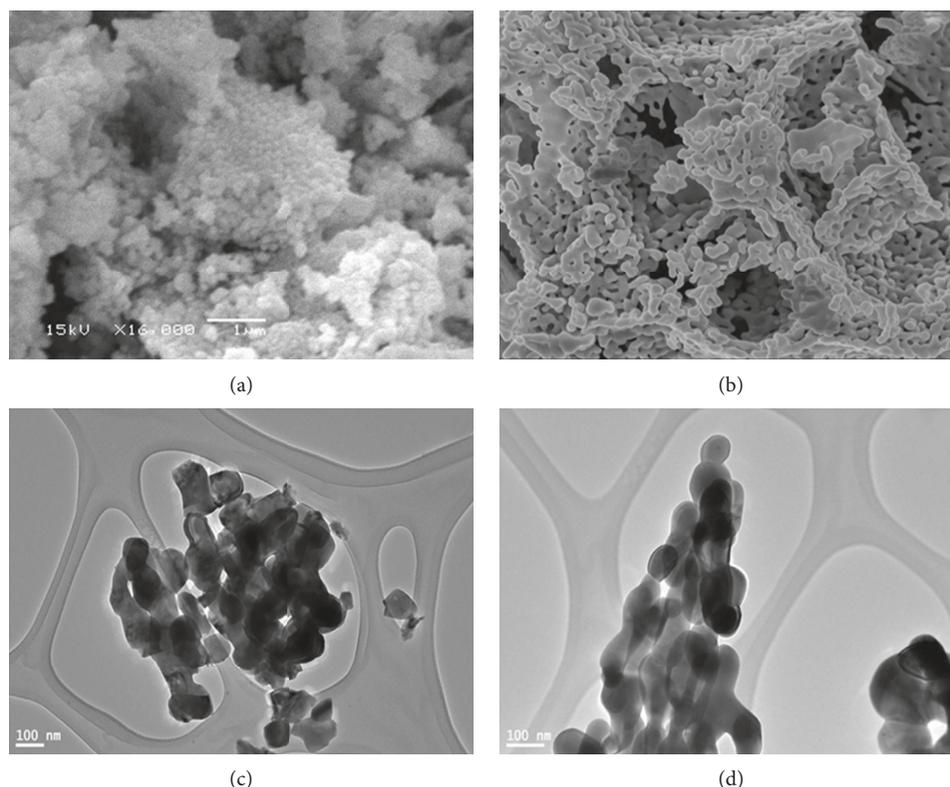


FIGURE 4: (a, b) SEM images of GdAlO_3 NPs prepared by using 100 and 300 kGy irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ with 100 kGy $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ precursors. (c, d) Respective TEM images.

~ 26 nm in case of 100 kGy irradiated, whereas the 300 kGy γ -irradiated precursors produced ~ 23 nm, respectively. This indicates that increasing the intensity of the irradiation the size of the resultant NPs decreases. This was further confirmed by SEM and TEM analysis.

For this purpose, SEM and TEM measurements were performed on the samples obtained from the reactions of irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ at 1000°C for 10 h. SEM micrographs of GdAlO_3 are shown in Figures 4(a) and 4(b). SEM images depict that individual particles seemed to exhibit the shape of micro-sized volumetric plate-like crystals which were partially fused to form soft agglomerates. Besides, the plate-like grains coexist with spherically shaped particles. These images also reveal that increasing the γ -ray doses from 100 to 300 kGy leads to the production of smaller size particles. This effect is clearly visible in the TEM images of the product obtained from both 100 and 300 kGy precursors.

The TEM images reveal that the spherical nanoparticles with the size between 50 and 70 nm were obtained in case of 300 kGy $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ with 100 kGy $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ as shown in Figures 4(c) and 4(d), while the 100 kGy irradiated sample produced slightly large nanoparticles with size between 70 and 100 nm. The difference between the particle sizes obtained from TEM results and XRD results is due to the serious agglomeration of crystallites. The particle size seemed to be higher than that corresponding to crystallite average sizes, which means that either the particles of the powder are

polycrystalline, or actually, the so-called “particles” are not the ultimate structural units, but rather they are agglomerates consisting of 2–3 single crystal particles very hard to be dispersed. Therefore, the irradiation by γ -ray can be considered as a new factor to prepare nanocrystalline GdAlO_3 NPs where the particles size of product can be optimized.

4. Conclusions

In this work, single-phase, perovskite-type GdAlO_3 NPs were prepared using γ -rays-irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ precursors in a sol-gel process. The surface morphology of the GdAlO_3 phase reveals spherical particles of smaller size coexisting with agglomerates. The TEM image shows the particles sizes of about 30–50 nm. During this study, it was revealed that both the precursor and irradiation with γ -rays have significant effect on the formation and quality of the resultant NPs. Since, the reaction with irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ with Al_2O_3 did not produce GdAlO_3 , whereas irradiated $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ precursors have successfully produced GdAlO_3 NPs. Moreover, with increasing the intensity of the irradiation, the size of the NPs also decreased.

Data Availability

The spectroscopic and microscopic data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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