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Magnetic, magnetocaloric and optical properties of nano $Gd_3Ga_5O_{12}$ garnet synthesized by citrate sol-gel method

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Original Research

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Abstract:

$Gd_3Ga_5O_{12}$ (GGG) nanoparticles with an average size around 50 nm were synthesized via citrate sol-gel method. Rietveld refinement confirmed the formation of a single-phase garnet structure. The optical properties characterized by UV-Vis absorption and photoluminescence spectra, exhibited bands centered at 300 nm and 436 nm, respectively. Magnetic measurements revealed paramagnetic behaviour above 15 K, as evidenced by the modified Curie-Weiss fitting with an effective magnetic moment of 13.86 μB per formula unit (F. U.). Arrott plots confirmed a second-order nature of the magnetic phase transition. Notably, the GGG nanoparticles displayed a significant magnetocaloric effect (MCE), with a maximum magnetic entropy change (-ΔSM) of 14.2 $Jkg^{-1}K^{-1}$ at 3.5 K under a 5 T field. These findings highlight the potential of GGG nanoparticles as refrigerants for low temperature magnetic refrigeration applications.

Keywords: Arrott plot; Effective magnetic moment; Magnetocaloric effect (MCE); Nano GGG; Optical properties; Rietveld refinement

1. Introduction

Rare earth (RE) based garnets, with their general formula $A_3B_2C_3O_{12}$(A = RE^{3+}, B and C = Fe^{3+}/Al^{3+}/Ga^{3+}), have emerged as a class of materials with exceptional magnetic, electrical, and optical properties [1, 2]. Their cubic crystal structure ($Ia\bar{3}d$), characterized by dodecahedral, octahedral, and tetrahedral sites, enables the accommodation of diverse cations [3–5]. This structural flexibility has led to their extensive application in optical components like modulators and magnetic field sensors [6]. Beyond their optical properties, the magneto-optical characteristics of ferrimagnetic garnets have garnered significant attention. The interplay between light and magnetization offers a powerful tool for visualizing magnetic domain structures and magnetization distributions, crucial for advancing technologies such as magnetic bubble memory [7, 8].

Rare earth garnets experienced a resurgence with the discovery of the magnetocaloric effect (MCE) in $RE_3(Ga/Fe)_5O_{12}$ by Belov *et al.*, which describes a material's ability to heat or cool in response to magnetic field changes [9, 10]. MCE, quantified by isothermal entropy change (ΔS_M) or adiabatic temperature change (ΔT_{ad}), holds immense potential for magnetic refrigeration applications operating near liquid helium temperatures, where traditional gas compression based refrigeration methods become inefficient [11]. The current challenge is to identify safer alternatives that minimize environmental impact while maintaining energy efficiency in active heating and cooling systems [12]. Consequently, a diverse array of magnetic solids, such as complex oxides [13–15], rare earth-transition metal alloys [16] and rare earth transition metal carbides [17] have been developed in recent years. Gadolinium Gallium Garnet (GGG) has become a benchmark refrigerant for temperatures below 20 K due to its favourable properties [11, 18]. The large magnetic moment of Gd^{3+} ions and the crystallographic structure of GGG contribute to its promising magnetocaloric properties [19]. Recently, Xiang Jin et al. investigated the effects of high pressure heat treatment on GGG single crystals, revealing enhanced magnetic refrigeration power and a broadened phase transition temperature range [20]. Researchers have extensively studied gadolinium gallium

garnets, substituting Gd with Dy or Ho and Ga with Fe to enhance magnetocaloric properties. The substitution of Gd with Dy or Ho in GGG creates superparamagnetic materials with fine clusters. This substitution notably enhances the magnetocaloric effect [21]. Phan *et al*. studied the MCE of bulk and nano $Gd_3Fe_5O_{12}$ garnet. The nanostructured gadolinium iron garnets exhibit a complex MCE behaviour compared to their bulk counterparts, influenced by factors such as grain size, surface spin disorder, and magnetic frustration [22].

GGG possesses a complex crystal structure with Gd^{3+} ions located on two interpenetrating corner-sharing triangular sublattices. This arrangement leads to geometric frustration in GGG, which prevents the establishment of long-range antiferromagnetic order. The frustration results in unique magnetic behaviours, making GGG a fascinating material for the study of frustrated magnetism [23–25]. GGG also exhibits noteworthy magneto-optical properties due to the presence of gadolinium ions with a substantial magnetic moment. This makes GGG a valuable material for faraday rotators and isolators in laser technology, where the rotation of the polarization plane of light in a magnetic field is utilized. GGG's transparency across the visible and near-infrared spectrum further enhances its suitability for optical devices [26–28].

However, the synthesis of high quality GGG is crucial for optimizing its optical and magnetocaloric properties. Traditional solid state methods often result in inhomogeneous materials with limited control over particle size and morphology. Synthesis of GGG is challenging due to the need of high temperatures, around 1300 ^{O}C, and extended durations ranging from 12 to 48 hours [29]. A wide variety of wet chemical methods have been developed for low temperature synthesis of nanomaterials with tunable magnetic and optical properties [30, 31]. This study aims to optimize the synthesis of high quality GGG nanoparticles by citrate sol-gel method [32] and comprehensively characterize their structural, optical, magnetic, and magneto-thermal properties. By understanding the interplay between GGG's optical and magnetocaloric characteristics, we seek to contribute to the development of advanced materials for both magnetic refrigeration and optical applications.

2. Methods

2.1 *Synthesis of nano GGG by citrate sol-gel method*

High purity gadolinium (III) oxide (Gd_2O_3, Sigma-Aldrich, 99.9%) and gallium (III) oxide (Ga_2O_3, Sigma-Aldrich, 99.99%) were accurately weighed in the specified mole ratio and separately dissolved in concentrated nitric acid (HNO_3). Upon dissolution, the oxides were converted into their corresponding nitrates. To the resulting homogeneous solution of Gd^{3+} and Ga^{3+} nitrates, citric acid and ethylene glycol were added as complexing agents. The entire mixture was then heated at 90 ^{O}C for four hours with continuous stirring using a magnetic stirrer to form a gel. The gel was charred, resulting in the formation of a fine precursor powder, which was then ground and calcined at 750 ^{O}C for 2 hours to obtain pure nano GGG.

2.2 *Characterisation of nano GGG*

The X-ray diffraction (XRD) pattern of nano GGG was recorded using a PANalytical X'Pert Pro diffractometer configured in Bragg-Brentano geometry with copper Kα radiation. The scan was conducted in the 2θ range of 10^{O} to 90^{O} with a step size and scan step time of 0.017^{O} and 40.702 seconds, respectively. Particle morphology was studied using High Resolution Transmission Electron Microscopy (FEI Tecnai G2 30 EDAX). The magnetization was measured as a function of temperature and applied magnetic field using a Quantum Design's SQUID (Superconducting Quantum Interference Device) magnetometer (Model MPMS3). The magnetization data were employed to derive isothermal magnetic entropy change (-ΔSM) through the application of integrated Maxwell's relation [18].

$$\Delta S_M = \frac{\int_0^H M(T+\Delta T,H')dH' - \int_0^H M(T,H')dH'}{\Delta T} \quad (1)$$

The ultraviolet-visible absorption spectrum was measured using a Shimadzu UV-3600 double beam spectrophotometer. Emission spectrum of the nano GGG powder was recorded using a Jobin Yvon SPEX-Fluorolog 3 (Horiba) spectrofluorometer. A 450 W xenon arc lamp served as the excitation light source. Both excitation and emission monochromators were employed and emitted photons were detected using a photomultiplier tube detector.

3. Results and discussion

The recorded X-ray diffraction pattern of nano GGG was initially analyzed with the JCPDS file no-71-0701 to verify phase formation, with the indexed pattern presented in Fig. 1 (a) [26]. Subsequently, the XRD pattern was refined using Rietveld refinement with GSAS software [33]. The background was fitted using a shifted Chebyshev polynomial with 10 variables. The refined structural parameters confirm the presence of a single-phase garnet structure with the ($Ia\bar{3}d$) space group. Figure 1 (b) presents the observed, calculated, and difference XRD patterns of nano GGG after refinement, while the various structural parameters are listed in Table 1. As reported in the literature, the XRD pattern of nano GGG shows peak broadening, which is attributed to the larger surface-to-volume ratio of the nanoparticles [34]. To determine the crystallite size using XRD, a Gaussian fit was applied to the (420) peak, as shown in Fig. 1 (c). The crystallite size of nano GGG was calculated to be 16.10 nm using the Debye-Scherrer formula, $D = K\lambda/\beta\cos\theta$ [35, 36]. Figures 2 (a) and 2 (b) depict the TEM image and SAED pattern of nano GGG, respectively, revealing the agglomerated nature of the particles with an approximate size of 50 nm. Magnetic nanoparticles can experience dipole-dipole interactions, where the magnetic moments of different particles attract each other. This magnetic attraction can cause the nanoparticles to come together and agglomerate [37, 38]. Figure 3 (a) presents the absorption spectrum of synthesized nano GGG, exhibiting a maximum at 300 nm and the absorption band spans from 250 nm to 350 nm, resulting in a transparent window encompassing the visible and near-infrared regions of the electromagnetic spectrum. As depicted in Fig. 3 (b), the photoluminescence of nano GGG

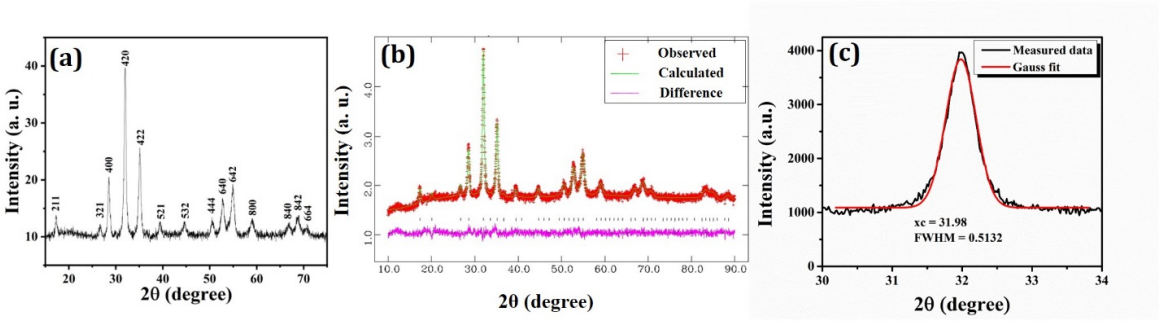

Figure 1. **(a)**. Indexed XRD pattern of nano GGG synthesised by citrate sol-gel method (JCPDS file no-71-0701), **(b)** Rietveld refined XRD pattern of nano GGG and, **(c)** Detailed view of the (420) XRD peak with corresponding Gaussian fit overlay

Table 1. Structural parameters of nano sized $Gd_3Ga_5O_{12}$.

Material	**Nano GGG**
Space group	$Ia\bar{3}d$
Lattice parameter, a (^{O}A)	12.4852 (3)
Volume (^{O3})	1946.2 (1)
Atomic positions	
Gd x	0.125
Gd y	0
Gd z	0.25
Ga1 x	0
Ga1 y	0
Ga1 z	0
Ga2 x	0.375
Ga2 y	0
Ga2 z	0.25
O x	-0.0348 (3)
O y	0.0579 (1)
O z	0.1641 (4)
Goodness of fit	
Rwp %	2.27
Rp %	1.80

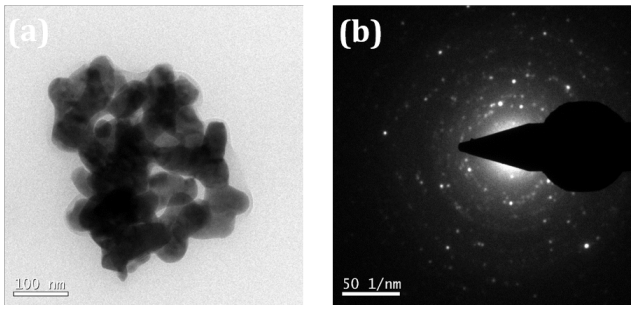

Figure 2. **(a)**. TEM image and, **(b)** SAED pattern of nano GGG.

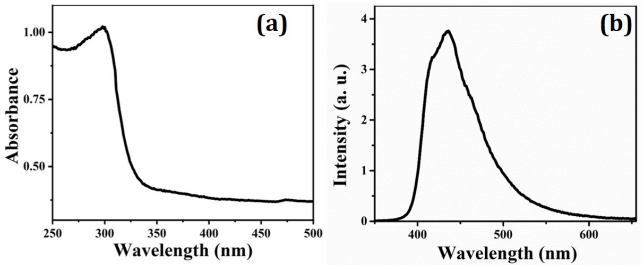

Figure 3. **(a)**. Absorption spectrum and **(b)** Emission spectrum (λ_{exc} 340 nm) of nano GGG.

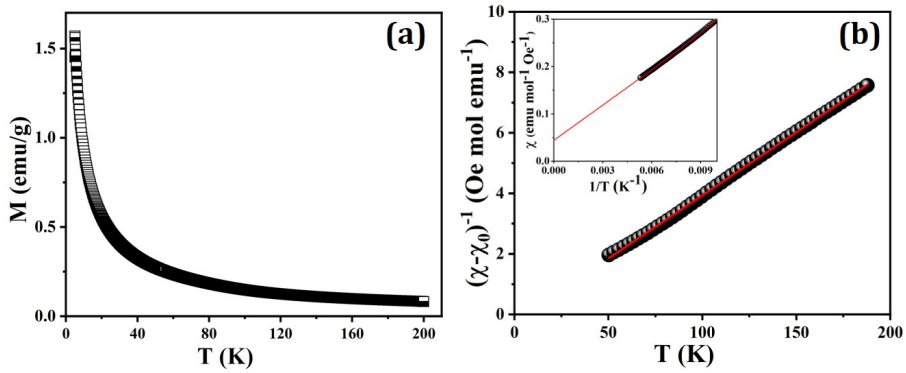

Figure 4. (**a**). M-T curve of nano GGG at 0.05 T, (**b**) Modified Curie–Weiss law fit of nano GGG and, ((**4 b**) **inset**) Determination of χ_0 from the plot of χ vs. 1/T.

displays a broad emission band centered at 436 nm, extending from 375 to 600 nm. Previous studies have reported similar broad band blue photoluminescence, which is attributed to Ga_2O_3 [39].

Figure 4 (a) represents the temperature dependence of magnetization of nano GGG in a magnetic field of 0.05 T. The data reveal a sharp increase in magnetization below 20 K, indicating magnetic ordering within the rare earth magnetic sublattice [40–42]. In the garnet structure, Gd^{3+}ions form a frustrated 3D network of corner-sharing triangles. This prevents antiferromagnetic interactions from being fully satisfied, leaving the system in a disordered state. This results in high magnetization at low temperatures [43–46]. In order to determine the effective magnetic moment, modified Curie-Weiss (C-W) fit was performed over the temperature range of 50 K to 200 K using the equation, $\chi = \chi_0 + C/(T - \theta_{cw})$ [42]. The temperature-independent susceptibility is denoted by χ_0, C represents the Curie-Weiss constant, while θ_{cw} is the Curie-Weiss temperature associated with the paramagnetic moment. The Fig. 4 (b) shows the plot of $(\chi - \chi_0)^{-1}$ vs. T, which suggests paramagnetic behaviour of nano GGG. The temperature independent susceptibility (χ_0) was determined from the plot of susceptibility (χ) vs. $1/T$ as shown in the inset of Fig. 4 (b). The effective magnetic moment, calculated using the formula $\mu_{eff}=\sqrt{8C}$, is found to be 13.86 μB / F. U. , which is in direct agreement with the theoretical value of 13.75 μB / F. U. of bulk GGG reported in the literature [47, 48].

Magnetization curves of nano GGG were recorded at different temperatures near the transition point (Fig. 5 (a)) to explore the variation in the magnetocaloric effect. At 2 K, the M-H curve of nano GGG exhibits significant magnetization even at low fields and remains unsaturated up to a field of 5 T, indicating the frustrated magnetic state of the sample, whereas, the linear field dependence of magnetization above 15 K, signifies the paramagnetic behaviour of nano GGG. To determine the nature of magnetic phase transition, the Arrott plots (M^2 vs. H/M) for nano GGG are presented in Fig. 5 (b). Arrott plots with a positive slope indicate a second-order phase transition, while those with a negative slope imply a first-order transition [49]. The Arrott plots for nano GGG reveal linear isotherms with positive slopes, confirming the second-order nature of the phase transition. From the M-H data, the magnetic entropy change was calculated using Equation 1 at various magnetic field strengths, as depicted in Fig. 6 (a). The results show that the magnetic entropy change (-ΔS_M) increases with the field strength, with the maximum observed value of -ΔS_M for nano GGG being 14.2 J/kgK at 3.5 K under a 5 T magnetic field. This suggests that nano GGG has potential as a refrigerant for low temperature magnetic refrigeration applications.

The field dependent magnetic entropy change is a key characteristic that provides valuable critical information into the magnetic behaviour of materials, particularly concerning magnetic refrigeration applications and magnetocaloric effects. This dependence typically follows a power-law relationship, with the exponent (n), varying depending on whether the material is in the low temperature frustrated

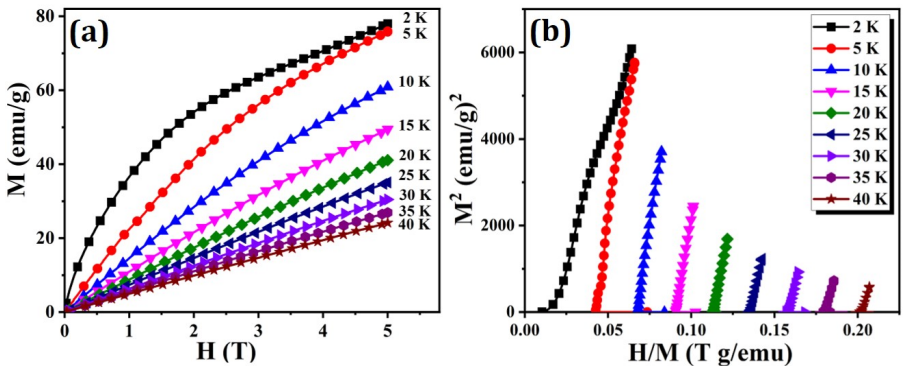

Figure 5. (**a**). Field dependant magnetization and, (**b**) Arrott plots of nano GGG.

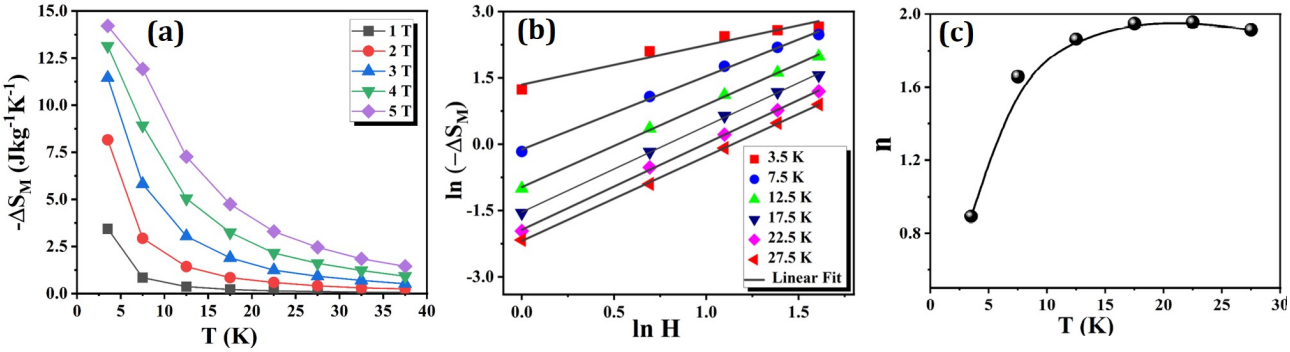

Figure 6. (**a**). Temperature dependence of magnetic entropy change, **(b)** Linear plots of ln(-ΔS_M) vs. ln(H) at various temperatures and **(c)** Temperature dependence of exponent, n of nano GGG.

state, near the critical temperature, or in the high temperature paramagnetic region. Direct integration of the Curie-Weiss law for ferromagnetic materials above their Curie temperature yields an exponent of $n = 2$. In contrast, a mean-field approach predicts that the magnetic entropy change at the Curie temperature will have a field dependence characterized by n = 2/3 [50]. A linear relationship between $\ln(-\Delta S_M)$ and ln(H) was investigated for temperatures ranging from 3.5 K to 27.5 K (Fig. 6 (b)). The slope of these plots determined the value of n, which was subsequently plotted against temperature for nano GGG (Fig. 6 (c)). The results indicate that the exponent "n" ranges from 0.89 to 1.96, suggesting significant magnetic ordering in nano GGG at low temperatures.

4. Conclusion

This study demonstrates the successful synthesis of $Gd_3Ga_5O_{12}$ (GGG) nanoparticles using a low temperature, short duration process, optimized for magnetocaloric applications. The particles exhibited a uniform size around 50 nm and a pure garnet phase, as confirmed by TEM and Rietveld refinement, respectively. Optical studies revealed characteristic absorption and emission bands. Magnetic measurements indicated paramagnetic behaviour at higher temperatures. The magnetic phase transition was confirmed to be second-order by Arrott plots. Most importantly, the GGG nanoparticles displayed a considerable magnetocaloric effect, with a maximum -ΔS_M of 14.2 J/kgK at 3.5 K under a 5 T field. These results highlight the potential of GGG nanoparticles as a promising material for magnetic refrigeration at low temperatures. Further investigations aimed at enhancing the magnetocaloric properties through size and compositional engineering are warranted.

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Authors contributions
Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials
The authors declare that the data supporting the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interests
The authors declare no conflict of interest.

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