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# Solgel Catalytic Interactions and Chemical Reactions during the Synthesis of $Y_{3-x}Ho_xFe_5O_{12}$ Garnet Nanoparticles

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**ABSTRACT:** This research explores the sol-gel synthesis of holmium-doped yttrium garnet ( $Y_{3-x}Ho_xFe_5O_{12}$ ,  $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$ ), which was chosen for its simplicity, cost-effectiveness, and ability to generate uniform fine powders. The synthesis process included dissolving precise quantities of yttrium, holmium, and iron nitrates in deionized water, then adding citric acid as a chelating agent and ammonia to adjust the pH to 7. The solution was heated to 80°C to create a gel, which was subsequently dried at 120°C to produce a xerogel. The final product was then heat-treated at 900°C for 4 hours to promote crystallization and form the garnet phase. Analysis confirmed the development of high-purity, fine garnet particles, showcasing the effectiveness of the sol-gel technique in producing holmium-doped yttrium garnet.

**KEYWORDS:** Solgel; Holmium; Yttrium Iron Garnet; Nanoparticle.

## I. INTRODUCTION

Yttrium iron garnet ( $Y_3Fe_5O_{12}$ , YIG) is a well-known material with remarkable magnetic, optical, and electrical properties [1], making it highly valuable for various technological applications, including microwave devices, magneto-optical sensors, and magnetic bubble memory devices [2, 3]. The incorporation of rare-earth elements into the YIG structure is a promising approach to further enhance its properties and tailor them for specific applications. Among these rare-earth elements, holmium (Ho) stands out due to its unique electronic configuration and magnetic characteristics. Holmium-doped yttrium garnet ( $Y_{3-x}Ho_xFe_5O_{12}$ ) introduces intriguing possibilities for modifying the magnetic and optical behavior of YIG [4]. Holmium ions ( $Ho^{3+}$ ) can replace yttrium ions ( $Y^{3+}$ ) in the garnet structure, potentially leading to significant changes in the material's properties due to the high magnetic moment and anisotropic nature of holmium [5]. This doping strategy aims to explore and optimize the material's performance across a range of concentrations ( $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$ ), providing a comprehensive understanding of how varying holmium content influences the garnet's characteristics.  $Y_{3-x}Ho_xFe_5O_{12}$  exhibits several notable physical properties influenced by the presence of holmium ions ( $Ho^{3+}$ ). Structurally, it retains the cubic garnet lattice of pure yttrium iron garnet ( $Y_3Fe_5O_{12}$ ), maintaining high phase stability even with varying holmium concentrations. The magnetic properties are significantly enhanced due to holmium's large magnetic moment and strong spin-orbit coupling, leading to increased magnetic moment and additional magnetic anisotropy [6]. This results in altered magnetization behavior and potentially shifts in the Curie temperature. Optically, holmium introduces distinct absorption and emission spectra due to 4f-4f electronic transitions, enhancing photoluminescence and making the material suitable for luminescent applications [7]. The dielectric properties remain favorable for microwave and high-frequency uses, although doping slightly influences electrical conductivity. Thermally, the material exhibits good stability and maintains its properties across a wide temperature range, with thermal expansion variations depending on the doping level.  $Y_{3-x}Ho_xFe_5O_{12}$  is utilized in optical isolators and circulators, which are crucial for preventing feedback in optical communication systems, and in magneto-optical sensors that detect magnetic fields with high precision [8]. Its favorable dielectric properties and thermal stability make it ideal for high-frequency microwave devices, including filters and resonators used in communication systems, radar technology, and satellite communications [9]. Additionally, holmium-doped yttrium garnet serves as an effective laser host material in solid-state lasers for medical and industrial applications [10]. Its

enhanced magnetic properties also improve the sensitivity and accuracy of magnetic field sensors, benefiting navigation systems, industrial monitoring, and scientific research [11]. In telecommunications, the material contributes to the efficiency and reliability of data transmission systems. The sol-gel method is an attractive synthesis route for producing holmium-doped yttrium garnet [12]. This technique is renowned for its simplicity, cost-effectiveness, and ability to produce homogeneous, fine powders with excellent compositional control [13-15]. By utilizing the sol-gel method, it is possible to achieve uniform doping and fine particle sizes, which are critical for enhancing the material's properties and ensuring high performance in practical applications. This research aims to investigate the sol-gel synthesis of holmium-doped yttrium garnet  $Y_{3-x}Ho_xFe_5O_{12}$  with varying holmium concentrations ( $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$ ) and to systematically study the impact of holmium doping on the structural, magnetic, and optical properties of the material. By exploring the effects of holmium doping, this research aims to provide valuable insights into the tunability of yttrium garnet's properties, paving the way for the development of advanced materials with enhanced performance for a wide range of technological applications.

## II. EXPERIMENTAL METHOD

### Materials:

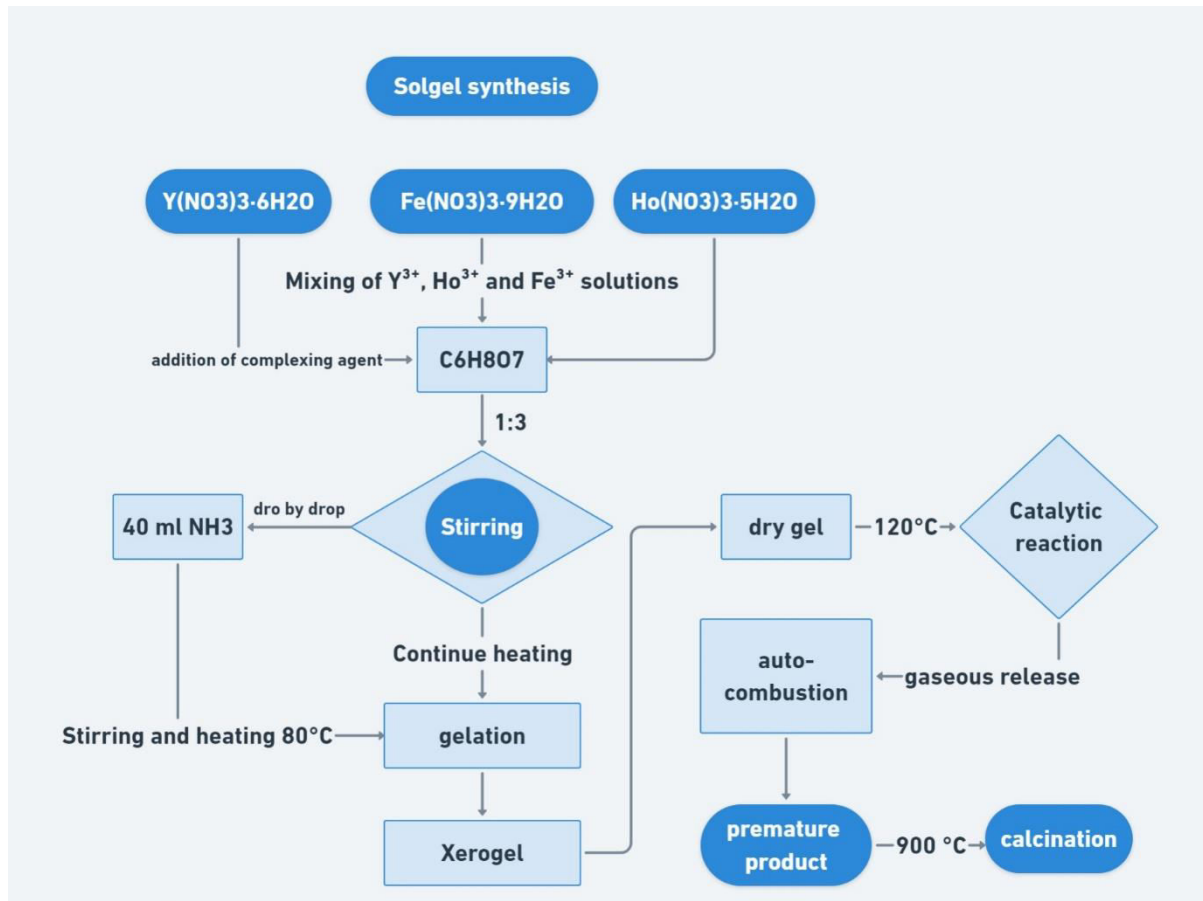
$Y_{3-x}Ho_xFe_5O_{12}$  garnet ferrite nanoparticles were synthesized using the sol-gel method. Stoichiometric amounts of Yttrium nitrate hexahydrate ( $Y(NO_3)_3 \cdot 6H_2O$ ) 383.01 g/mol, Iron (III) nitrate nonahydrate ( $Fe(NO_3)_3 \cdot 9H_2O$ ) 404.00 g/mol, and Holmium(III) nitrate pentahydrate ( $Ho(NO_3)_3 \cdot 5H_2O$ ) 441.02 g/mol were dissolved in distilled water to obtain the desired compositions with  $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$ . The citric acid ( $C_6H_8O_7$ ) 210.14 g/mol with the nitrate to acid ratio 1:3; acts as a complexing agent, forming metal-citrate complexes that help with homogeneous mixing at the molecular level.  $NH_3$  was used to maintain the Ph at 7.

## III. SYNTHESIS

### Solgel synthesis of $Y_{3-x}Ho_xFe_5O_{12}$ garnet nanoparticles:

In this garnet structure, yttrium acts as a base cation and is typically partially replaced by holmium to achieve the desired properties. Holmium, belonging to the lanthanide series, substitutes for yttrium to fine-tune the magnetic and optical characteristics of the nanoparticles. Iron, from Group 8, contributes magnetic properties due to its d-orbital electrons, while oxygen forms the anionic framework of the garnet, coordinating with the metal cations to maintain the structural integrity of the nanoparticles. The flowchart outlines the sol-gel synthesis process for  $Y_{3-x}Ho_xFe_5O_{12}$  ( $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$ ) garnet nanoparticles, a well-established technique for creating these materials at the nanoscale. This process offers precise control over the composition, size, and morphology of the nanoparticles, making them valuable for various technological applications. The sol-gel synthesis of  $Y_{3-x}Ho_xFe_5O_{12}$  garnet nanoparticles begins with the careful selection and preparation of starting materials. Yttrium nitrate hexahydrate ( $Y(NO_3)_3 \cdot 6H_2O$ ) serves as the source of yttrium (Y) ions, which are integral to the garnet structure. Iron (III) nitrate nonahydrate ( $Fe(NO_3)_3 \cdot 9H_2O$ ) provides the necessary iron (Fe) ions, with the nonahydrate form indicating nine water molecules associated with each iron nitrate molecule. Holmium (III) nitrate pentahydrate ( $Ho(NO_3)_3 \cdot 5H_2O$ ) introduces holmium (Ho) ions, allowing for modification of the nanoparticles' properties by partially substituting yttrium with holmium. The pentahydrate form signifies five water molecules per holmium nitrate molecule. Citric acid ( $C_6H_8O_7$ ) acts as a complexing agent, binding to the metal ions to prevent premature aggregation and ensure a homogeneous mixture, which is crucial for controlled reactions in subsequent steps. The choice of solvent, such as ethanol or methanol, affects processing conditions and final nanoparticle properties. It needs to be miscible with the starting materials and possess suitable volatility for efficient drying. In the mixing stage, all materials are combined to form a uniform solution, ensuring the proper distribution of precursors. This is followed by aging, where the solution is maintained at around 80°C. During aging, hydrolysis reactions occur, forming metal hydroxides and releasing nitric acid as a byproduct. Simultaneously, condensation reactions initiate, linking metal hydroxides into a nascent network. As processing continues, condensation becomes more pronounced, leading to the formation of a three-dimensional network that entraps solvent molecules, signifying the gelation stage. The result is a wet gel, a jelly-like substance containing a metal oxide network with trapped solvent. The properties of this gel, such as pore size and density, are influenced by the conditions during earlier stages. To obtain a solid material known as xerogel, the wet gel is dried to remove the solvent. Drying can be achieved through slow evaporation or accelerated methods like vacuum drying. Careful control of the drying rate is essential to avoid cracking or excessive shrinkage. Finally, calcination involves heating the dried xerogel to high temperatures, typically exceeding 500°C. This step removes residual organic residues, such as citric acid, and promotes the crystallization of the amorphous xerogel into the crystalline  $Y_3$ .

$Y_{3-x}Ho_xFe_5O_{12}$  garnet phase. The calcination temperature and duration significantly impact the final product's crystallinity, phase purity, and magnetic properties. By optimizing these parameters, researchers can tailor the size, morphology, and properties of the  $Y_{3-x}Ho_xFe_5O_{12}$  nanoparticles for various applications, including magnetic recording media. The flowchart in **Figure 1** depicts the subsequent stages involved in the sol-gel synthesis.



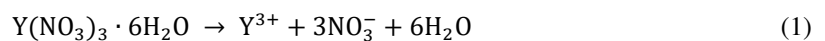
(a)

Fig. 1. Synthesis (a) The subsequent stages involved in the sol-gel synthesis of  $Y_{3-x}Ho_xFe_5O_{12}$  garnet nanoparticles

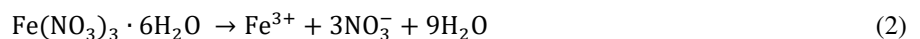
### 3.1 Chemical reactions during $Y_{3-x}Ho_xFe_5O_{12}$ formation

Dissolution and Complexation

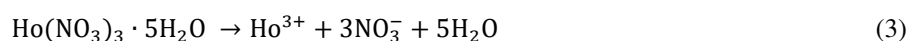
Yttrium Nitrate Hexahydrate

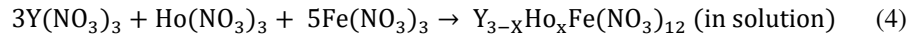


Iron (III) Nitrate Nonahydrate

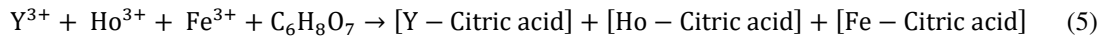


Holmium (III) Nitrate Pentahydrate

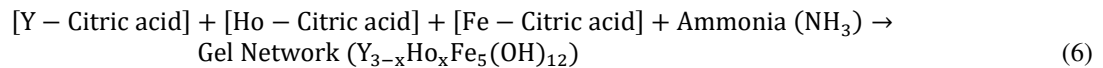




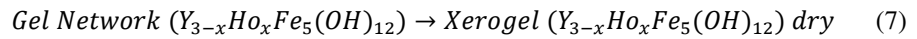
Citric acid ( $C_6H_8O_7$ ) chelates the metal ions, forming Metal-Citric Acid Complexes



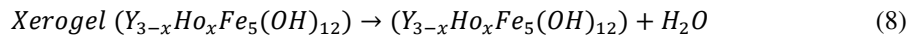
Gel formation involves the formation of a metal-organic network, which includes



Obtain Xerogel



Calcination to Form Garnet



### 3.2 Role of $NH_3$

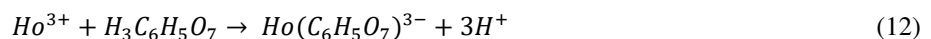
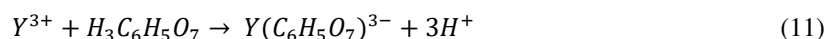
In the sol-gel synthesis of  $Y_{3-x}Ho_xFe_5O_{12}$  garnet nanoparticles, ammonia ( $NH_3$ ) plays a critical role in controlling the solution's pH, which significantly influences the complexation of metal ions, gel formation, and the final properties of the nanoparticles. By increasing the pH,  $NH_3$  promotes the deprotonation of citric acid, facilitating the formation of metal-citrate complexes that ensure uniform distribution of  $Y^{3+}$ ,  $Ho^{3+}$ , and  $Fe^{3+}$  ions, thereby preventing premature precipitation. The adjusted pH is crucial for effective polymerization and gelation processes, leading to a stable and homogeneous gel network. Additionally,  $NH_3$  helps to minimize the formation of insoluble hydroxides or oxides by maintaining an optimal pH range, typically between 6 and 8 [16]. Ammonia is added to the solution to increase the pH and facilitate the complexation process.

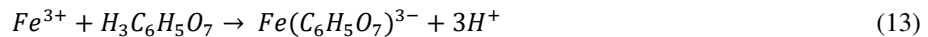


This controlled pH environment supports uniform hydrolysis and condensation reactions, essential for forming a consistent sol that transforms into a stable gel. Throughout the synthesis,  $NH_3$  ensures the pH remains stable, enabling uniform gelation and preventing undesirable precipitation. This careful pH management by  $NH_3$  is essential for producing high-quality  $Y_{3-x}Ho_xFe_5O_{12}$  garnet nanoparticles with consistent and reproducible properties.

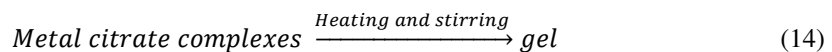
### 3.3 Role of citric acid

In the sol-gel synthesis of  $Y_{3-x}Ho_xFe_5O_{12}$  garnet nanoparticles, the metal nitrate to citric acid ratio of 1:3 is crucial for the successful formation of the gel and the ultimate synthesis of the nanoparticles. This ratio refers to the molar ratio of total metal nitrates (Yttrium, Holmium, and Iron) to citric acid. Citric acid serves multiple roles in the synthesis process: it acts as a complexing agent, binding with the metal ions ( $Y^{3+}$ ,  $Ho^{3+}$ , and  $Fe^{3+}$ ) to prevent premature precipitation and ensure uniform distribution; it facilitates the polymerization process, aiding in the formation of a stable gel that traps the metal ions uniformly; and it helps reduce nitrate ions to gases like  $NO_x$  during heating, preventing the formation of unwanted side products. Citric acid ( $H_3C_6H_5O_7$ ) complexes with the metal ions to form metal-citrate complexes

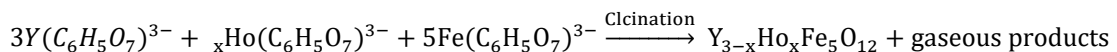




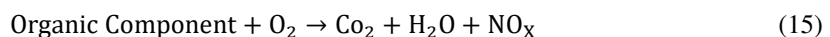
Upon heating and stirring, the metal-citrate complexes polymerize to form a gel



During drying, water is removed, and during calcination, the organic components are decomposed, and the desired garnet phase is formed



The gaseous byproducts typically include CO<sub>2</sub>, NO<sub>2</sub>, and H<sub>2</sub>O



This set of reactions simplifies the complex chemical processes involved in the sol-gel synthesis of garnet nanoparticles. The actual reactions may involve intermediate steps and various side reactions depending on specific synthesis conditions. This ensures a homogeneous gel formation, preventing premature metal hydroxide or oxide precipitation and promoting uniform nanoparticle synthesis. Thus, maintaining the 1:3 ratio optimizes the synthesis process, ensuring consistent and reproducible production of Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> garnet nanoparticles.

#### IV. STRUCTURE

##### Structural parameters of Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> garnet nanoparticles

Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> garnet nanoparticles, with the chemical symbol Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> and CAS number 12063-56-8, comprise elements from specific groups in the periodic table: yttrium from Group 3, iron from Group 8, and oxygen from Group 16. The electronic configurations of these elements are as follows: yttrium (Y) is [Kr] 4d<sup>1</sup> 5s<sup>2</sup>, holmium (Ho) is [Xe] 4f<sup>11</sup> 6s<sup>2</sup>, iron (Fe) is [Ar] 3d<sup>6</sup> 4s<sup>2</sup>, and oxygen (O) is 1s<sup>2</sup> 2s<sup>2</sup> 2p<sup>4</sup> [17-19]. The garnet structure is a type of cubic crystal system known for its general formula AB<sub>3</sub>C<sub>2</sub>O<sub>12</sub>, where the cations occupy specific sites within the crystal lattice. Holmium-doped yttrium garnet (Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub>) features a cubic crystal structure typical of Space Group: I4-3d for the garnet structure, characterized by its specific arrangement of metal and oxygen ions. In this structure, yttrium (Y<sup>3+</sup>) and holmium (Ho<sup>3+</sup>) ions occupy the 8-coordinate dodecahedral (A) sites, while iron (Fe<sup>3+</sup>) ions are located in the 6-coordinate octahedral (B) sites. The oxygen ions form a three-dimensional framework around these metal ions, contributing to the stability and functionality of the material. The overall crystal lattice of Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> is highly ordered, with the oxygen ions creating a tetrahedral network that interlinks the metal ions. This arrangement results in a stable and robust structure, which is crucial for the material's enhanced magnetic and optical properties. The presence of holmium ions introduces additional magnetic anisotropy and shifts in the Curie temperature, while also contributing to distinct optical absorption and emission characteristics due to their 4f-4f electronic transitions. This well-defined structure underpins the material's effectiveness in advanced applications such as optical isolators, microwave devices, and magnetic sensors [20-23].

#### V. CONCLUSION

The sol-gel method is a powerful and versatile technique for synthesizing holmium-doped yttrium iron garnet (Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub>) with excellent control over composition and microstructure. By carefully tailoring the starting material ratios, processing conditions, and calcination parameters, this research demonstrates the ability to produce high-quality Y<sub>3-x</sub>Ho<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> garnet nanoparticles with tunable holmium concentrations (x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9). The systematic investigation of the effects of holmium doping on the structural, magnetic, and optical properties provides valuable insights into the versatility of this material system. The sol-gel approach enables the synthesis of homogeneous, fine powders with uniform holmium distribution, which is critical for enhancing the material's performance in various applications. The findings from this research pave the way for the development of advanced functional materials with optimized properties, opening up new possibilities in areas such as magnetic recording media, microwave devices, and optical communications. The ability to precisely control the composition and tailor the

characteristics of  $Y_{3-x}Ho_xFe_5O_{12}$  garnets through the sol-gel method highlights its potential as a powerful tool for materials engineering and innovation.

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