

Research Article

**SYNTHESIS AND CHARACTERIZATION OF LANTHANUM-
DOPED COBALT FERRITE NANOPARTICLES PREPARED VIA
SIMPLE CO-PRECIPIATION***Le Kim Chung, Nguyen Hoang Huy,**Vo Cong Minh, Le Thi Viet Hoa, Truong Chi Hien***Ho Chi Minh City University of Education, Vietnam***Corresponding author: Truong Chi Hien – Email: hientc@hcmue.edu.vn**Received: June 12, 2024; Revised: June 27, 2024; Accepted: July 10, 2024***ABSTRACT**

In this study, spinel ferrite nanoparticles $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ ($x = 0, 0.025, \text{ and } 0.05$) were successfully synthesized via a simple co-precipitation method using a 5% NaOH solution as a precipitating agent. The physicochemical properties of the materials, annealed at 850 °C for one hour, were characterized using powder X-ray diffraction (PXRD), energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM), and vibrating-sample magnetometry (VSM) at room temperature. The average crystallite size, calculated from PXRD, and the particle size, determined by TEM, of the $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ samples ranged from 20 to 30 nm and decreased with increasing La^{3+} ion doping percentage. The La-doped CoFe_2O_4 nanomaterials exhibited high coercivity ($H_c = 902.00\text{--}1045.26 \text{ Oe}$) and high saturation magnetization ($M_s = 83.33\text{--}65.17 \text{ emu}\cdot\text{g}^{-1}$), making them ideal for use in magnetic recording materials such as hard drives, magnetic tapes, and the production of permanent magnets.

Keywords: Cobalt ferrite; co-precipitation method; La-doping; magnetic characteristics; nanoparticles

1. Introduction

One category of inorganic nanomaterials currently attracting considerable research interest consists of those with MFe_2O_4 ferrite spinel structures ($\text{M} = \text{Fe}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$) (Dang et al., 2021; Elayakumar et al., 2019; Hoang et al., 2022; Lamouri et al., 2020; Nguyen et al., 2017; Nguyen et al., 2021). In MFe_2O_4 , the M^{2+} ion is located in the tetrahedral site (A-site) while Fe^{3+} occupies the octahedral site (B-site). Due to their unique properties, MFe_2O_4 spinels are attracting attention in various fields, including adsorption, catalysis, electrode materials, electromagnetic materials, and magneto-optical materials (Chung et al.,

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2023; Dang et al., 2021; Fabricio et al., 2020; Kumar et al., 2016; Ngo et al., 2018; Wang et al., 2012). Additionally, compared to metals and alloys, spinel ferrites offer advantages such as thermal stability, long-term durability, and low cost.

Among spinel ferrites, cobalt ferrite (CoFe_2O_4) stands out as a promising hard magnetic material for permanent magnets due to its high coercivity and moderate saturation magnetization (Ngo et al., 2018; Rachidi et al., 2019). Additionally, cobalt ferrite exhibits high chemical stability and mechanical hardness. The structural characteristics and properties of spinel cobalt ferrite are influenced by its chemical composition, the distribution of cations within the crystal lattice, particle size and morphology, synthesis method, and dopant concentration (Lamouri et al., 2020; Maaz et al., 2007; Patankar et al., 2017; Zhao et al., 2014).

Doped CoFe_2O_4 and CoFe_2O_4 nanoparticles have been synthesized using various methods such as hydrothermal, sol-gel, gel combustion, and sol-gel complexing. These methods offer advantages such as uniformly distributed precursors, low sintering temperatures, reduced particle sizes, and uniform particle size distributions (Maaz et al., 2007; Ngo et al., 2018). However, the sol-gel technique presents limitations, particularly in selecting the appropriate gelling organic compound and determining the correct molar ratio between the gelling agent and the total metal ions. Additionally, it is essential to strictly control factors such as temperature, time, and pH value during the gelling process.

In the studies by Nguyen et al. (2021, 2023) and Truong et al. (2022), spinel ferrites MFe_2O_4 ($\text{M} = \text{Fe}, \text{Co}$) and orthoferrite AFeO_3 ($\text{A} = \text{Ho}, \text{Eu}$) nanoparticles have been successfully synthesized with sizes ranging from 20 to 50 nm using a simple co-precipitation method. This method involves the hydrolysis of M(II) , A(III) , and Fe(III) cations in boiling water without the introduction of surfactants. The hydrolysis of M(II) , A(III) , and Fe(III) cations at high temperatures, followed by cooling, results in stable sol particles and limits the increase in particle size compared to precipitation at room temperature (Nguyen et al., 2017). To date, there have been no reports in the literature on the study of lanthanum-doped cobalt spinel ferrite using this simple co-precipitation method. The significance of rare-earth elements in various applications has motivated further investigation into the role of La^{3+} ion substitution in the structural and magnetic characteristics of CoFe_2O_4 .

2. Experiments and research methods

Chemicals used to synthesize $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ ($x = 0, 0.025, \text{ and } 0.05$) nanomaterials include lanthanum nitrate hexahydrate ($\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99.9% purity, Sigma-Aldrich), cobalt nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 99.9% purity, Sigma-Aldrich), iron (III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 99.9% purity, Sigma-Aldrich), sodium hydroxide (NaOH , 98% purity, Sigma-Aldrich), double-distilled water, filter paper, and pH indicator paper. All chemicals were of analytical grade and used directly without further purification.

La -doped CoFe_2O_4 nanomaterials were synthesized via co-precipitation following the procedure described in previous publications (Chung et al., 2023; Nguyen et al., 2023;

Truong et al., 2022;). A mixture of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ was weighed in the appropriate molar ratios, dissolved in 50 mL of distilled water, and stirred for 15 minutes to ensure complete dissolution. The resulting saline solution was then slowly added dropwise into 500 mL of boiling distilled water on a heating magnetic stirrer, maintaining the temperature at approximately 95 °C. After the complete addition of the salts, the mixture was heated and stirred for an additional five minutes to ensure complete hydrolysis and then allowed to cool to room temperature (~30 °C).

A 5% NaOH solution was added dropwise to the system until reaching a pH of 9 (Chung et al., 2023; Nguyen et al., 2023) to precipitate all Co^{2+} , Fe^{3+} , and La^{3+} cations. The resulting precipitate was stirred for about 45 minutes, allowed to settle for 15 minutes, and then filtered using a vacuum filtration setup. The precipitate was washed three times with distilled water and allowed to dry naturally at room temperature until a constant volume was reached, which took approximately five days. The dried precipitate was finely ground and then calcined at 850 °C for one hour. The selection of the calcination temperature and duration was based on the study by Chung et al. (2023) for the Y-doped CoFe_2O_4 system.

Powder X-ray diffraction (PXRD) analysis of the obtained $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ samples was conducted using an EMPYREAN X-ray diffractometer (CuK α radiation, $\lambda = 0.15406$ nm, angle range of $2\theta = 10-80^\circ$). The average crystallite size (D_{hkl} , nm) of the $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ samples was calculated using the Debye-Scherrer equation (Nguyen et al., 2017).

$$D_{hkl} = \frac{k \cdot \lambda}{\beta_{hkl} \cdot \cos\theta} \quad (1)$$

where β_{hkl} is the full-width at half maximum (FWHM, radian), and θ is the corresponding diffraction angle of the maximum reflection (in degrees); k is the shape factor (for the orthorhombic structure, $k = 0.89$).

The X-ray patterns were used to calculate the lattice parameter (a) from the d-spacing using equation (2), for a cubic structure (Chung et al., 2023; Nguyen et al., 2017):

$$a = d_{hkl} \sqrt{(h^2 + k^2 + l^2)}. \quad (2)$$

where (h , k , and l) represent the Miller's indices.

The quantitative and qualitative composition of the samples was determined by energy-dispersive X-ray spectroscopy (EDX-analysis) using an FE-SEM S-4800 scanning electron microscope.

The morphology and particle size of the obtained $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ samples were determined by transmission electron microscopy (TEM) using a Joel JEM-1400 microscope.

The magnetic properties, including the coercivity (H_c , Oe), remanent magnetization (M_r , $\text{emu} \cdot \text{g}^{-1}$), and saturation magnetization (M_s , $\text{emu} \cdot \text{g}^{-1}$) of the $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ samples ($x = 0, 0.025, \text{ and } 0.05$) were investigated using a vibrating sample magnetometer MICROSENE EV11. Their hysteretic loop was obtained by varying the magnetic field from -16.000 to $+16.000$ Oe at room temperature.

3. Results and discussion

Figure 1 depicts the powder X-ray diffraction (PXRD) pattern of the dried precipitate samples following calcination at 850 °C for one hour. The PXRD spectra in Figure 1 reveal peaks corresponding to the positions of the CoFe_2O_4 standard peaks in the reference database, indicating a cubic structure belonging to the $Fd3m$ space group (JCPDS 22-1086) (Chung et al., 2023). The Miller indices (hkl), including (111), (220), (311), (400), (422), (511), (440), and (533), are recorded (Figure 1). All three samples exhibited flat baselines, indicating high crystallinity with no observable peaks corresponding to impurity phases. Additionally, a shift in the 2θ angular position was observed with changes in the x value (Table 1), indicating partial replacement of Fe^{3+} ions by La^{3+} ions. This observation is consistent with findings in other spinel ferrite systems synthesized using various methods, such as $\text{CoFe}_{2-x}\text{Y}_x\text{O}_4$ (Chung et al., 2023), $\text{CuFe}_{2-x}\text{Ho}_x\text{O}_4$ (Hoang et al., 2022), $\text{CuFe}_{2-x}\text{Ce}_x\text{O}_4$ (Elaykumar et al., 2019), $\text{CoFe}_{2-x}\text{Y}_x\text{O}_4$ (Patankar et al., 2017), and $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ (Fabricio et al., 2020).

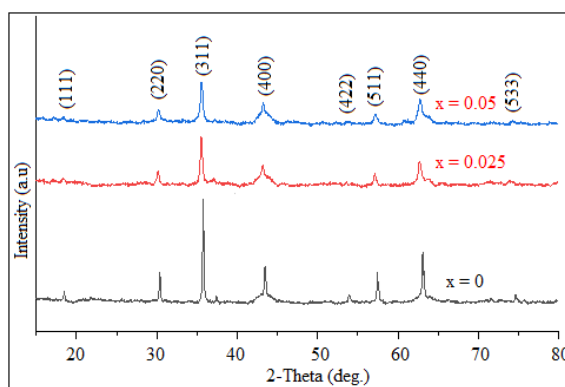


Figure 1. PXRD patterns of La-doped CoFe_2O_4 nanoparticles annealed at 850 °C

Table 1. PXRD characteristics of $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ nanocrystals annealed at 850 °C for 1h

$\text{CoFe}_{2-x}\text{La}_x\text{O}_4$	$2\theta_{(311)}, ^\circ$	$FWHM, ^\circ$	D_{hkl}, nm	$a, \text{Å}$
$x = 0$	35.757	0.154	54.2	8.32
$x = 0.025$	35.524	0.312	26.7	8.36
$x = 0.05$	35.558	0.343	24.3	8.39

A noticeable reduction in the 2θ angle corresponding to the (311) peak occurs as x increases from 0 to 0.025 ($\Delta 2\theta = 0.233^\circ$), followed by a slight increase as x further increases to 0.05 ($\Delta 2\theta = 0.034^\circ$) (Table 1). This observation confirms the substitution of Fe^{3+} ions for La^{3+} ions (Chung et al., 2023; Fabricio et al., 2020). Moreover, an expansion of the peak width is observed as x increases from 0 to 0.05 (Figure 1 and Table 1), indicating a gradual decrease in the average crystal size (D_{hkl} , nm) calculated using equation (1) while the cubic lattice parameter (a , Å) determined by equation (2) slightly increases (Table 1). Similar trends of decreasing crystal size and increasing cubic lattice parameters with increasing

dopant content have been reported in doped spinel ferrite systems such as $\text{CuFe}_{2-x}\text{Ce}_x\text{O}_4$ and $\text{CuFe}_{2-x}\text{Ho}_x\text{O}_4$ (Elaykumar et al., 2019; Hoang et al., 2022).

To determine the qualitative and quantitative composition of the elements present in the sample, EDX analysis was performed on the $\text{CoFe}_{1.95}\text{La}_{0.05}\text{O}_4$ sample. The results are presented in Figure 2 and Table 2.

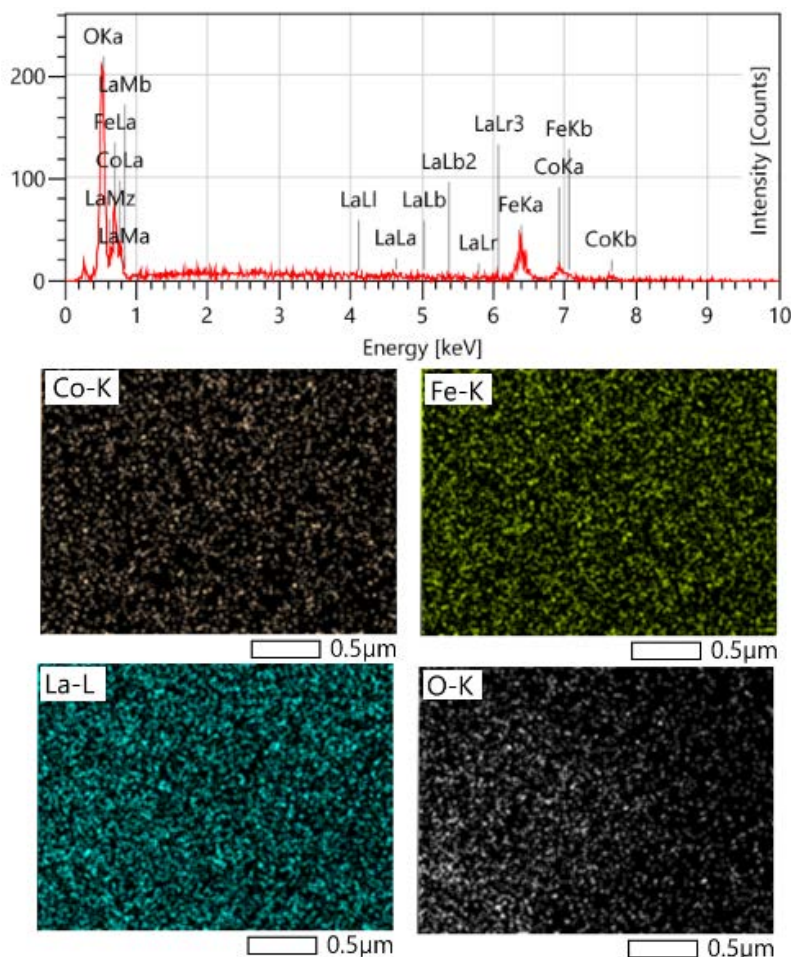


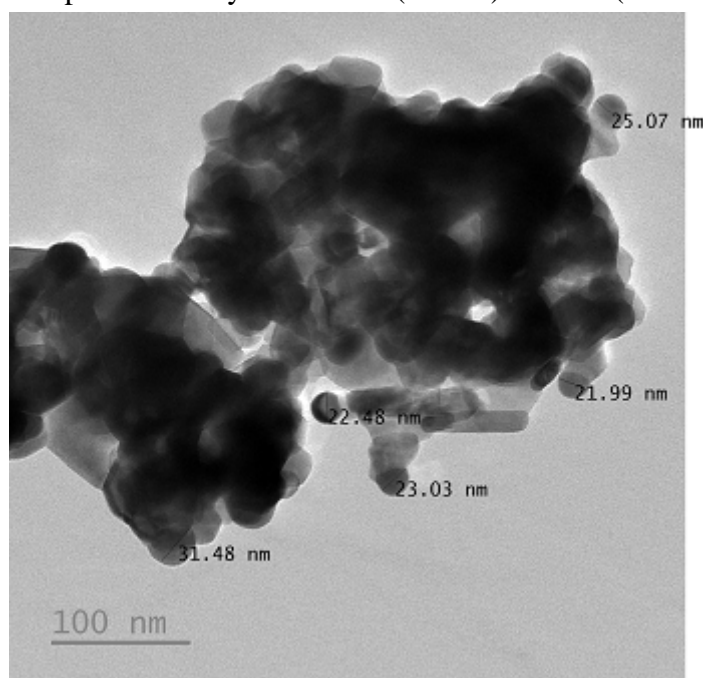
Figure 2. EDX and EDX-mapping images of $\text{CoFe}_{1.95}\text{La}_{0.05}\text{O}_4$ nanoparticles annealed at $850\text{ }^\circ\text{C}$

The EDX diagram reveals peaks corresponding only to the elements present in the sample, namely La, Fe, Co, and O, with no peaks observed for the impurity element Na. The mass percentage and atomic percentage compositions of Co, Fe, La, and O closely match their expected proportions in the $\text{CoFe}_{1.95}\text{La}_{0.05}\text{O}_4$ formula, with deviations of less than 5% (Table 2). This indicates a uniform distribution of Co, Fe, La, and O atoms within the $\text{CoFe}_{1.95}\text{La}_{0.05}\text{O}_4$ crystal (Figure 2).

Table 2. EDX analysis results for $\text{CoFe}_{1.95}\text{La}_{0.05}\text{O}_4$ annealed at $850\text{ }^\circ\text{C}$ for 1h

Element	O	Fe	La	Co
Mass, %	22.78 ± 0.64	46.13 ± 4.45	4.75 ± 1.21	26.34 ± 4.35
Atom, %	52.14 ± 1.46	30.26 ± 2.92	1.26 ± 0.32	16.38 ± 2.70

Transmission electron microscopy (TEM) images of a La-doped CoFe_2O_4 nanomaterial sample ($x = 0.05$) reveal significant agglomeration within the sample (Figure 3). This phenomenon may be attributed to the strong magnetism of the nanoparticles, leading to their mutual attraction and resulting in difficult dispersion during TEM analysis. Nonetheless, some particles located at the boundary of the agglomerated cluster are visible, with sizes ranging from 20 to 30 nm. Interestingly, this size range slightly exceeds the crystal size obtained from the powder X-ray diffraction (PXRD) method (Table 1)

**Figure 3.** TEM image of La-doped CoFe_2O_4 nanoparticles annealed at $850\text{ }^\circ\text{C}$

Analysis of $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ nanomaterial samples ($x = 0, 0.025, \text{ and } 0.05$) using a vibrating sample magnetometer (VSM) at room temperature revealed that the concentration of doped lanthanum ions influenced the magnetic properties of the synthesized CoFe_2O_4 spinel nanoparticles (Figure 4 and Table 3).

The data presented in Table 3 and the VSM plot in Figure 4 demonstrate that the introduction of lanthanum ions into the spinel ferrite CoFe_2O_4 crystal lattice enhances the coercivity (H_c) while reducing the remanent magnetization (M_r) and particularly the saturation magnetization (M_s). This decrease in M_r and M_s values may be attributed to the exchange interaction between the non-magnetic La^{3+} ions ($[36\text{Xe}]5d^1$) and the magnetic Fe^{3+}

([18Ar]3d⁵)/Co²⁺ ([18Ar]3d⁷) ions. Conversely, the increase in H_c is attributed to the enhancement of crystal anisotropy upon incorporation of La³⁺ ions into the spinel cobalt crystal structure (Cullity et al., 2009; Demirci et al., 2018).

It is worth noting that the M_r and M_s values of the La-doped CoFe₂O₄ nanomaterial samples synthesized in this study were higher than those of CoFe_{2-x}Y_xO₄ synthesized via solution combustion (Patankar et al., 2017). Furthermore, the saturation magnetization values of the doped samples, and particularly the CoFe₂O₄ samples (M_s = 65.17–83.33 emu·g⁻¹), were significantly greater than those of CoFe₂O₄ synthesized via sol-gel or gel combustion methods (Ngo et al., 2018; Patankar et al., 2017).

Table 3. Magnetic parameters of La-doped CoFe₂O₄ nanoparticles at 300 K

CoFe _{2-x} La _x O ₄	H _c , Oe	M _r , emu g ⁻¹	M _s , emu g ⁻¹
x = 0	902.00	34.58	83.33
x = 0.025	1067.08	33.55	68.80
x = 0.05	1045.26	25.87	65.17

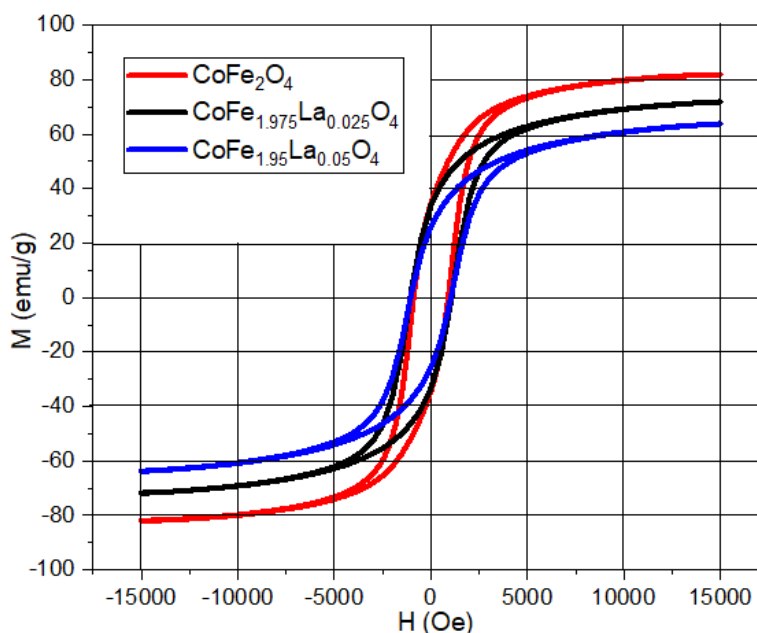


Figure 4. Room-temperature VSM curves of CoFe_{2-x}La_xO₄ nanoparticles annealed at 850 °C

The CoFe_{2-x}La_xO₄ doped nanomaterial samples (x = 0, 0.025, and 0.05) synthesized in this study exhibit remarkably high coercivity values (H_c = 902.00–1045.26 Oe), indicating their characteristics as hard magnetic materials. These materials possess strong magnetic attraction properties, akin to rare earth magnets, suggesting their potential utility in the fabrication of permanent magnets or as magnetic recording materials in hard disk drives and magnetic tapes (Cullity et al., 2009; Fabricio et al., 2020; Nguyen et al., 2017).

4. Conclusions

La-doped CoFe_2O_4 nanoparticles were successfully synthesized via a simple co-precipitation method involving the hydrolysis of Co^{2+} , Fe^{3+} , and La^{3+} cations in hot water (temperature ≥ 95 °C), with 5% NaOH serving as a precipitant. Upon dry precipitation calcination at 850 °C for one hour, single-phase nanocrystals of $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ with a cubic structure belonging to the Fd3m space group were obtained.

The increase in the concentration of La^{3+} dopant ions within the spinel ferrite CoFe_2O_4 crystal lattice led to a reduction in average crystal size, residual magnetism, and saturation magnetization, accompanied by an increase in coercivity. The synthesized La-doped CoFe_2O_4 nanoparticles exhibit excellent coercivity and saturation magnetization, making them suitable for applications such as permanent magnets and magnetic recording materials in hard disk drives and magnetic tapes.

❖ **Conflict of Interest:** Authors have no conflict of interest to declare.

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**TỔNG HỢP VÀ ĐẶC TÍNH CỦA VẬT LIỆU NANO COBALT FERRITE
PHA TẠP LANTHANUM TỔNG HỢP BẰNG PHƯƠNG PHÁP ĐỒNG KẾT TỬA**

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TÓM TẮT

Trong nghiên cứu này, các hạt nano spinel ferrite $CoFe_{2-x}La_xO_4$ ($x = 0, 0.025$ và 0.05) đã được tổng hợp thành công bằng phương pháp đồng kết tủa đơn giản sử dụng tác nhân kết tủa là dung dịch $NaOH$ 5%. Các đặc tính hoá lý của vật liệu sau khi nung ở $850^\circ C$ trong 1 giờ được đánh giá bằng các phương pháp nhiễu xạ tia X bột (PXRD), phổ tán sắc năng lượng tia X (EDX), kính hiển vi điện tử truyền qua (TEM), và hệ đo từ kế mẫu rung ở nhiệt độ phòng (VSM). Kích thước tinh thể trung bình tính theo PXRD và kích thước hạt xác định theo TEM của các mẫu $CoFe_{2-x}La_xO_4$ dao động trong khoảng 20-30 nm và giảm theo chiều tăng nồng độ ion La^{3+} pha tạp. Các mẫu vật liệu nano La-doped $CoFe_2O_4$ đều có lực kháng từ ($H_c = 902,00-1045,26$ Oe) và độ từ hoá bão hoà ($M_s = 83,33-65,17$ emu·g⁻¹) rất lớn, thích hợp làm vật liệu ghi từ trong các ổ đĩa cứng, các băng từ hoặc chế tạo nam châm vĩnh cửu.

Keywords: Cobalt ferrite; phương pháp đồng kết tủa; pha tạp La; từ tính; hạt nano