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Synthesis, characterization and magnetic properties of LaFeO₃ nanoparticles by EDTA assisted Sol-Gel method

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Abstract

In this study, phase-pure orthorhombic perovskite-type LaFeO₃ (LFO) nanoparticles were successfully synthesized at 450 °C using the Sol-Gel auto-combustion method, employing EDTA as a complexing agent. The synthesized nanoparticles were characterized using X-ray diffraction (XRD) and a superconducting quantum interference device (SQUID) magnetometer. XRD analysis, supported by Rietveld refinement, confirmed the formation of a single-phase perovskite orthorhombic structure (space group Pbnm) with a crystallite size of 25 nm. SQUID magnetometry revealed unsaturated magnetization, remanence, and coercivity in the low-field region at both 5 K and 300 K, indicating predominant antiferromagnetic ordering with a weak ferromagnetic component. Notably, the synthesized LaFeO₃ nanoparticles exhibited high coercivity values of 880 Oe at 5 K and 3450 Oe at 300 K significantly higher than previously reported highlighting their potential for use in magnetic memory applications.

Keywords: Sol-gel, Autocombustion, Nanoparticles, XRD, Magnetic nanoparticles

1. Introduction

Perovskite-type oxides with the general formula ABO₃ have garnered considerable attention due to their versatile functional properties and wide-ranging applications in catalysis, gas sensing, magnetic devices, and fuel cells [1-7]. Among them, lanthanum orthoferrite (LaFeO₃) stands out as a prominent member of the perovskite family, exhibiting excellent chemical stability, narrow bandgap, and antiferromagnetic behavior [8, 9]. Lanthanum orthoferrite (LaFeO₃) crystallizes in a distorted perovskite structure, which deviates from the ideal cubic perovskite form due to the size mismatch between the A-site (La³⁺) and B-site (Fe³⁺) cations. This structural distortion results in an orthorhombic unit cell, where the three lattice parameters are unequal ($a \neq b \neq c$) but the interaxial angles remain at 90°, a characteristic of orthorhombic symmetry [10]. LaFeO₃ specifically belongs to the Pbnm space group, which is a common subgroup of perovskite structures associated with tilting of the BO₆ octahedra and reduced symmetry [11]. Experimental studies, particularly those involving X-ray diffraction and Rietveld refinement, have determined the lattice constants of LaFeO₃ to be approximately $a = 5.557 \text{ \AA}$, $b = 5.565 \text{ \AA}$, and $c = 7.854 \text{ \AA}$, indicative of the slight distortion from cubic symmetry [12]. The perovskite framework is composed of corner-sharing FeO₆ octahedra, where each Fe³⁺ ion is surrounded by six O²⁻ ions in an octahedral geometry. These octahedra are interconnected in a three-dimensional network, with the larger La³⁺ ions occupying the interstitial A-sites [8, 13]. Magnetic characterization through neutron diffraction has revealed that LaFeO₃ exhibits a canted G-type antiferromagnetic (AFM) ordering. In this magnetic arrangement, each Fe³⁺ ion is magnetically coupled with six nearest-neighbor Fe³⁺ ions in an antiparallel (Antiferromagnetic) fashion, forming a G-type pattern [11]. However, due to a slight misalignment or "canting" of spins attributed to the antisymmetric Dzyaloshinskii–Moriya interaction a weak ferromagnetic component is also present [14, 15]. The material undergoes antiferromagnetic ordering at a relatively high

Néel temperature (T_N) of around 740 K, highlighting its thermal stability and potential for high-temperature magnetic applications [13, 16]. The structural stability of the ABO_3 perovskite lattice is predominantly governed by the Goldschmidt tolerance factor (t), which provides insight into the geometric compatibility of the A and B-site cations within the oxygen framework. In the case of LaFeO_3 , the tolerance factor is found to be less than unity ($t < 1$), indicating a mismatch in ionic radii that causes a deviation from the ideal cubic symmetry. This deviation favors the formation of a lower-symmetry orthorhombic structure, characterized by tilted and distorted FeO_6 octahedra. As a consequence of this structural distortion, the Fe–O–Fe bond angle is reduced from the ideal 180° , resulting in bent connections between adjacent FeO_6 octahedra. This tilting significantly affects the electronic bandwidth and magnetic exchange interactions, thereby influencing the overall physical properties of the material [17]. The properties of LaFeO_3 can be strategically tailored through several modification techniques. One common approach involves reducing the particle size, which increases the surface area and can enhance properties such as catalytic activity and magnetic behavior [18]. Another effective strategy is cationic substitution at the A-site (La^{3+}) or B-site (Fe^{3+}), which can alter the structural, electronic, and magnetic characteristics of the material [19–22]. At the nanoscale, LaFeO_3 exhibits behaviors that are markedly different from its bulk counterpart due to a higher surface-to-volume ratio and quantum confinement effects. These nanoscale features enable fine-tuning of its functional properties, making nanostructured LaFeO_3 a promising candidate for advanced applications in catalysis, sensing, and spintronic devices [23–25]. In response to the increasing demand for nanomaterials with controlled size, morphology, and enhanced functionality, attention has shifted toward more refined wet chemical synthesis techniques. These methods offer significant advantages, including lower processing temperatures, improved phase purity, and greater control over particle uniformity and distribution [26]. Recent advancements in solution-based synthesis routes have further enabled the fabrication of ultrafine, stoichiometrically precise, and highly homogeneous mixed-metal oxide nanoparticles, paving the way for scalable and reproducible production tailored for advanced technological applications [27]. A wide range of wet chemical synthesis techniques have been explored for the fabrication of nanosized LaFeO_3 , offering improved control over morphology and composition. These methods include hydrothermal synthesis [3, 28], co-precipitation [29, 30], auto-combustion [31, 32], sol–gel processing [2, 33, 34], microwave-assisted synthesis [1, 35, 36], sonochemical methods [37, 38], polymerizable complex (Pechini) routes [39, 40], microemulsion techniques [41, 42] and the sucrose-driven auto-combustion [43]. Although many of these strategies have successfully produced nearly phase-pure LaFeO_3 nanoparticles, certain methods still face challenges such as the presence of trace secondary phases or the need for elevated calcination temperatures to attain complete crystallization and structural integrity.

In the present work, we examine the use of ethylenediaminetetraacetic acid (EDTA) as a complexing agent in the sol-gel auto-combustion synthesis of LaFeO_3 nanoparticles. EDTA is a well-known multidentate ligand, possessing four carboxylic acid groups and two amine

groups, which enables it to form stable chelate complexes with metal ions. This strong chelating ability allows EDTA to effectively bind with La^{3+} and Fe^{3+} ions, leading to the formation of a homogeneous and stable metal-ligand network in the sol-gel matrix. The presence of EDTA plays a crucial role in enhancing the gelation process, improving the distribution of metal ions, and ensuring better molecular-level mixing of the precursors. As a result, this promotes enhanced phase purity and improved compositional uniformity of the final product. Moreover, the combustion behavior of the EDTA-assisted gel facilitates the removal of organic residues during calcination, enabling the formation of phase-pure LaFeO_3 nanoparticles at relatively lower temperatures compared to conventional methods. The synthesized materials characterized by X-Ray diffraction (XRD) and Rietveld refinement has been done using Fullprof software.

2. Experimental

2.1 Synthesis Process

To prepare LaFeO_3 nanoparticles, lanthanum nitrate hexahydrate [$\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$] and ferric nitrate nonahydrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] were first dissolved separately in distilled water in their respective stoichiometric ratios. The two solutions were then combined to form a homogeneous precursor solution. Following this, ethylenediaminetetraacetic acid (EDTA) was introduced into the mixed precursor solution in a 1:1 molar ratio with respect to the total metal ions ($\text{La}^{3+} + \text{Fe}^{3+}$). The mixture was continuously stirred and heated at approximately 100°C to promote

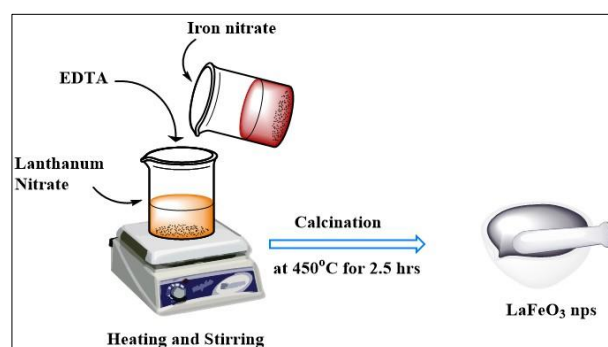


Fig 1: Synthesis of LaFeO_3 nanoparticles via EDTA assisted Sol Gel method

complexation between the metal ions and EDTA. This process was maintained until the majority of the solvent had evaporated and the solution gradually transformed into a viscous, gel-like mass. Upon further heating, this gel underwent self-sustained auto-combustion, resulting in the formation of a puffy, porous precursor powder due to the rapid release of gases during combustion. The obtained powder was then calcined in a muffle furnace at 450°C for 2.5 hours to remove residual organics and to facilitate the formation of well-crystallized LaFeO_3 nanoparticles. The overall synthesis route for the EDTA-assisted sol-gel auto-combustion process is illustrated schematically in Figure 1.

2.2 Characterization technique

The phase purity and crystalline structure of the calcined LaFeO_3 powders were analyzed using X-ray diffraction (XRD), performed on a PANalytical X'Pert Pro diffractometer operating at 40 kV and 40 mA, utilizing Ni-

filtered Cu K α radiation ($\lambda = 0.15406$ nm). XRD patterns were recorded over a 2θ range of 20° to 70° , with a step size of 0.02° and a scanning rate of 2° min^{-1} to ensure high-resolution data acquisition. To obtain detailed structural information, the diffraction data were subjected to Rietveld refinement using the FullProf Suite software, employing the orthorhombic Pbnm (space group no. 62) as the structural model. Furthermore, the magnetic behavior of the synthesized LaFeO₃ nanoparticles was examined by recording the magnetization (M) response to varying magnetic fields (H) at both room temperature (300 K) and at 5 K. These measurements were performed using a superconducting quantum interference device (SQUID) magnetometer (Quantum Design, San Diego, CA), known for its exceptional sensitivity to weak magnetic signals.

3. Results and Discussion

3.1 X-ray diffraction

X-ray diffraction (XRD) serves as a powerful tool for evaluating the crystallinity, crystal structure, and phase purity of synthesized materials. The XRD pattern of LaFeO₃ powders calcined at 450°C for 2.5 hours is presented in Figure 2. The observed diffraction peaks at 2θ values of 22.70° , 25.32° , 32.25° , 34.09° , 38.03° , 39.76° , 41.55° , 46.20° , 47.81° , 52.11° , 53.54° , 57.47° , 63.80° and 67.43° correspond to the (101), (111), (121), (210), (112), (220), (131), (202), (212), (103), (311), (123), (331) and (242)

(131), (202), (212), (103), (311), (240), (331) and (242) crystal planes, respectively. These peaks are in good agreement with the standard reference data from JCPDS Card No. 75-0541, confirming the formation of single-phase orthorhombic LaFeO₃ with Perovskite-type structure. The absence of any secondary peaks in the pattern further supports the phase purity of the synthesized material. Moreover, the notable broadening of the diffraction peaks indicates the nanocrystalline nature of the LaFeO₃ powders, consistent with the low-temperature synthesis approach employed in this study. These results confirm that phase-pure LaFeO₃ with well-defined crystallinity was successfully achieved at a relatively low calcination temperature of 450°C . The average crystallite size (D) of LaFeO₃ was calculated using Scherrer's which is given follow

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where D is crystallite size, K is equal to $0.89 \text{ \AA}$, β is FWHM in radians, θ is the Bragg's angle, λ is the X-ray wavelength (0.15406 nm). The average crystallite size (D) is found to be 25 nm which confirms the nanocrystalline nature of synthesized LaFeO₃.

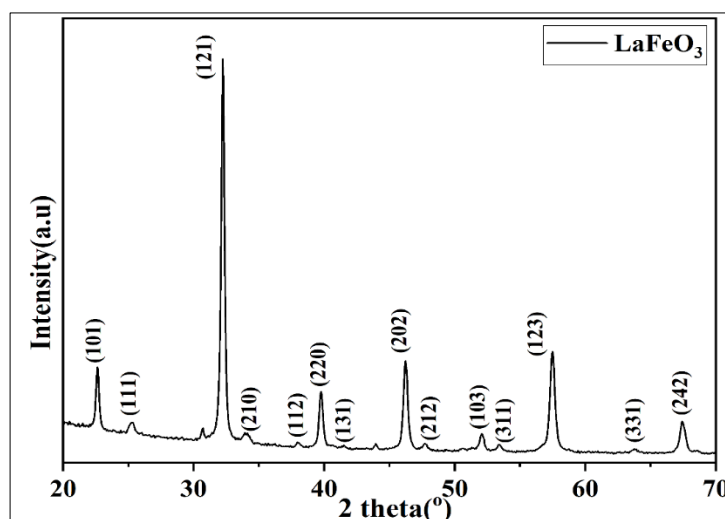


Fig 2: XRD result of LaFeO₃ nanoparticles synthesized via EDTA assisted sol gel method calcined at 450°C

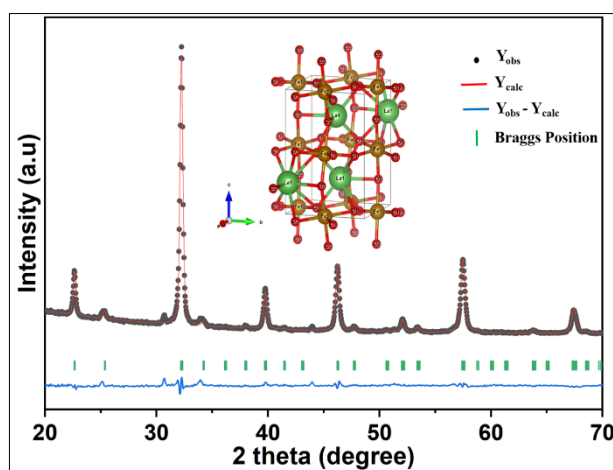


Fig 3: Rietveld refinement of LaFeO₃ synthesized via EDTA assisted Sol Gel method.

The lattice parameters of the synthesized LaFeO₃ nanoparticles were determined through Rietveld refinement of the XRD data. Figure 3 presents the results of the Rietveld analysis for LaFeO₃ nanoparticles prepared using citric acid at 450°C . The XRD data were refined assuming an orthorhombic crystal structure with the Pbnm space group, which accurately accounts for all diffraction peaks associated with the LaFeO₃ perovskite phase. The structural parameters obtained from this refinement are summarized in Table 1 and show good consistency with previously reported values for LaFeO₃ nanoparticles [14]. A strong agreement between the observed and calculated patterns is indicated by $\chi^2 = 2.02$, and R-factors of $R_{wp} = 13.6$, $R_p = 12.1$, $R_{Bragg} = 5.38$, and RF-factor = 5.38. The refined lattice parameters were found to be $a = 5.546 \text{ \AA}$, $b = 5.561 \text{ \AA}$, $c = 7.852 \text{ \AA}$, with $\alpha = \beta = \gamma = 90^\circ$, and a unit cell volume of $242.197 \text{ \AA}^3$, which aligns well with reported literature values for LaFeO₃ nanoparticles [14]. The crystal structure of LaFeO₃, based on

the refined crystallographic data, was visualized using VESTA software and is shown in Figure 3.

Table 1: Crystallographic data of LaFeO₃ obtained from Rietveld refinement of XRD data using Fullprof and VESTA software.

Cell parameter	Atoms	Wyckoff Position	x	y	z	R factor (%)
a=5.546 Å	La	4c	1.008	0.026	0.250	$\chi^2 = 2.02$
b=5.561 Å	Fe	4b	0.000	0.500	0.000	$R_p = 12.1$
c=7.852 Å	O1	8d	1.198	0.234	0.049	$R_{wp} = 13.6$
$\alpha=\beta=\gamma=90^\circ$	O2	4c	0.028	0.492	0.250	$R_{Bragg} = 5.38$
						RF factor = 5.78

Space group - Pbnm (62) Crystal Structure - Orthorhombic
 Cell Volume (Å³) – 242.197 Crystallite size (nm) – 25 nm
 Fe-O1 (Å) – 2.08(0) Fe-O1-Fe (°) – 157.08(0)
 Fe-O2 (Å) – 2.01(0) Fe-O2-Fe (°) – 154.04 (0)

3.2 Magnetic Properties

The magnetic characteristics of the prepared LaFeO₃ nanoparticles were evaluated at 5 K and 300 K under an applied field ranging from -70 kOe to +70 kOe. Fig. 4 explains the behavior of magnetization (M) versus applied magnetic field (H). The M-H curves do not get saturated till maximum applied field 70 k Oe which is the characteristic of antiferromagnetic behavior of nanocrystalline LaFeO₃ nanoparticles. However, in the low field region presence of coercivity and remanence suggests the existence of ferromagnetic behavior at both temperatures. The coercivity (H_c) and remanence magnetization (M_r) values were calculated from hysteresis loops using formula $H_c = |H_L - H_R|/2$ where H_R and H_L are right and left coercivity respectively and $M_r = |M_+ - M_-|/2$, where M₊ and M₋ are upper and lower remanence, respectively [44]. The coercivity

values of pure LaFeO₃ nanoparticles at 5K and 300K were found to be 880 and 3450 Oe, respectively. From the hysteresis loop, M_r Values were found to be 0.03 and 0.07 emu/g at 5 K and 300 K, respectively. The magnetization at maximum applied field (M_s) were 0.95 and 0.81 emu/g at 5 K and 300 K, respectively. This M-H behavior in LaFeO₃ nanoparticles are different from the linear behavior of bulk antiferromagnetic LaFeO₃ [16, 45, 46]. The M-H hysteresis loop suggests the presence of two magnetic phases in the synthesized LaFeO₃ nanoparticles. Furthermore, the room-temperature M-H curve exhibits a broader loop compared to the low-temperature curve, which has a higher coercive magnetization. The large values of coercivity indicate the presence of high anisotropy, and surface defects in prepared LaFeO₃ nanoparticles.

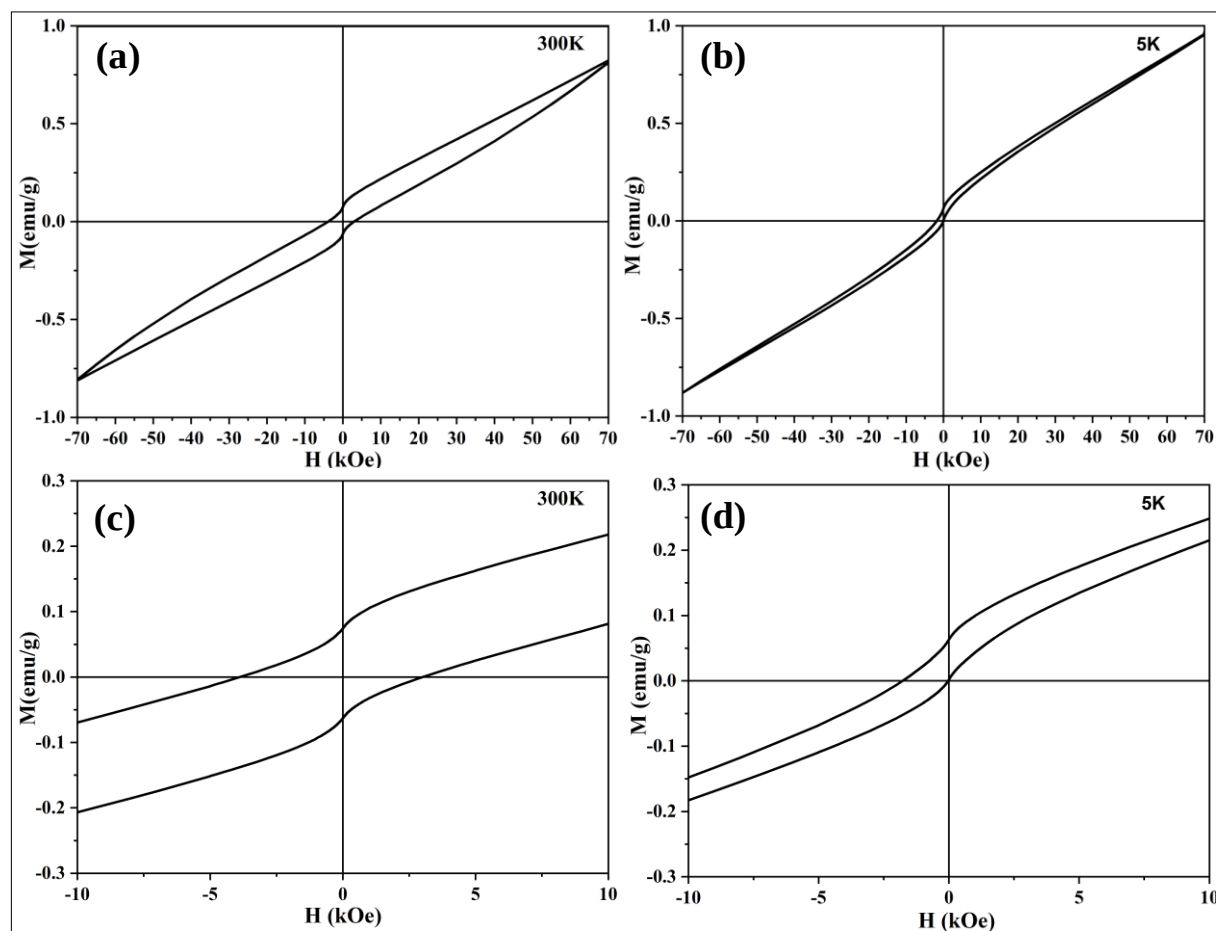


Fig 4: M-H curve of LaFeO₃ nanoparticles (a) at 300 K, (b) at 5 K, (c) magnified view at 300K, and (d) magnified view at 5K

It has been reported that at nanoscale, antiferromagnetic systems such as NiO^[47, 48], CuO^[49], Co₃O₄^[50] and BiFeO₃^[51] shows the weak ferromagnetic behavior along with dominant antiferromagnetic behavior. According to Néel's model for small antiferromagnets, the spins of the two ferromagnetic sublattices in the antiferromagnetic lattice are not completely balanced due to their small size and surface defects^[52]. This imbalance in ferromagnetic spin sublattices at the surface of LaFeO₃ nanoparticles leads to uncompensated spins and results in net magnetic moment. The presence of "uncompensated" surface spin surrounding the LaFeO₃ antiferromagnetic nanoparticles is further confirmed by shifted M-H hysteresis loop at low temperature. It should be noted in the magnified view of Fig.7b that (at 5K) the M-H hysteresis loop shifts toward the positive magnetization axis and negative magnetic field axis, a behavior not observed in the room-temperature M-H loop. This result suggests that magnetic unidirectional anisotropy can be isothermally induced at room temperature during the initial magnetization, likely arising from interfacial coupling between the ferrimagnetic and antiferromagnetic phases present in the LaFeO₃ nanoparticles. This intriguing feature of shifted hysteresis loop is known as exchange bias effect which is a characteristic of hybrid antiferromagnetic-ferromagnetic (AFM/FM) systems. Therefore, the M-H hysteresis loop of LaFeO₃ nanoparticles unveil a superposition of contribution attributed to AFM core and FM component arises from uncompensated surface spins as a result LaFeO₃ nanoparticles consist of AFM cores and FM-like shells^[43, 45, 53]. At room temperature, the magnetic properties of LaFeO₃ nanoparticles are primarily influenced by the antiferromagnetic core, while at low temperatures, surface spins become dominant, leading to the formation of a spin glass state.

4. Conclusions

In summary, we have presented a simple methodology for the synthesis of single phase LaFeO₃ nanoparticle using the Sol-Gel auto-combustion technique with EDTA as the complexing agent. XRD analysis along with Rietveld refinement confirms the formation of phase-pure orthorhombic perovskite-type LaFeO₃ with Pbnm (62) space group and having average crystallite size of is ~ 25 nm. The magnetic measurements suggested the presence of antiferromagnetic phase along with weak ferromagnetic phase and M-H loop exhibited high coercivity values of 880 Oe and 3450 Oe at 5K and 300K, respectively. The LaFeO₃ nanoparticle are composed of FM shells and AFM cores because of uncompensated surface spins. The shifted hysteresis at low temperature suggested the presence of exchange bias in LaFeO₃ nanoparticle which has great applications in spintronics.

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