

EFFECT OF Al^{3+} SUBSTITUTION ON THE STRUCTURAL, MAGNETIC, ELECTRICAL, AND DIELECTRIC PROPERTIES OF M-TYPE BARIUM HEXAFERRITE NANOPARTICLES PREPARED BY THE SOL-GEL AUTO-COMBUSTION TECHNIQUE

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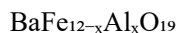
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Introduction

Ferrites are technologically important magnetic oxide materials used in electronic devices, permanent magnets, high-frequency components, magnetic recording systems, and electromagnetic interference suppression. Among the different ferrite families, hexagonal ferrites are particularly important because of their distinctive crystal structures, high magnetic anisotropy, chemical stability, and useful magnetic characteristics. M-type barium hexaferrite, with the chemical formula $\text{BaFe}_{12}\text{O}_{19}$, is one of the most widely studied hard magnetic ferrites. Its properties make it suitable for permanent magnets, magnetic recording media, microwave components, and other applications requiring stable magnetic performance. The material possesses a hexagonal crystal structure and exhibits ferrimagnetic ordering arising from the different magnetic sublattices occupied by Fe^{3+} ions.

The physical properties of barium hexaferrite depend on its chemical composition, crystal structure, particle size, morphology, preparation method, and heat-treatment conditions. One effective method of modifying these properties is the partial substitution of Fe^{3+} ions with other metal ions. Aluminium is particularly interesting because Al^{3+} has the same formal charge as Fe^{3+} but is essentially nonmagnetic under the conditions relevant to these ferrites. Therefore, substitution can alter magnetic exchange interactions without requiring charge compensation in the ideal stoichiometric composition. M-type barium hexaferrite ($\text{BaFe}_{12}\text{O}_{19}$) is an important hard magnetic material because of its high magnetocrystalline anisotropy, good chemical stability, relatively high coercivity, and potential applications in permanent magnets, magnetic recording, microwave devices, and electromagnetic shielding. Substitution of trivalent aluminium ions (Al^{3+}) for iron ions (Fe^{3+}) provides an effective approach for modifying the structural, magnetic, electrical, and dielectric properties of this material. The present paper examines the influence of Al^{3+} substitution on M-type barium hexaferrite nanoparticles prepared using the sol-gel auto-combustion technique. The general composition is represented by $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$, where x denotes the concentration of substituted aluminium ions. The theoretical principles of aluminium substitution, the synthesis mechanism, and the characterization techniques used to investigate the material are discussed. Published studies indicate that aluminium substitution can decrease saturation magnetization by replacing magnetic Fe^{3+} ions with essentially nonmagnetic Al^{3+} ions. It can also modify coercivity, crystallite size, lattice parameters, electrical resistivity, and dielectric response. In particular, the effects on coercivity are composition-dependent and cannot be assumed to increase continuously at every substitution level. The sol-gel auto-combustion method offers advantages in chemical homogeneity, compositional control, and relatively simple powder synthesis. This review establishes that aluminium-substituted barium hexaferrite is a promising material for applications requiring tailored magnetic and dielectric properties.

The general composition of aluminium-substituted barium hexaferrite is written as:



where x represents the number of Fe^{3+} ions replaced by Al^{3+} ions per formula unit. Increasing x changes the concentration of magnetic iron ions and can modify the distribution of cations among the crystallographic sites. These changes influence the net magnetic moment, magneto crystalline anisotropy, coercive field, and other physical properties. Several studies have investigated aluminium-substituted barium hexaferrite synthesized using solution combustion and sol-gel auto-combustion techniques. Dhage et al. (2011) examined the structural and magnetic behaviour of aluminium-doped barium hexaferrite, while Hojjati et al. (2015) investigated its microstructure and magnetic properties. Sarkar et al. (2021) reported the synthesis and characterization of nanosized aluminium-substituted barium hexaferrite. Dhage et al. (2021) further examined the electrical, dielectric, and alternating-current conductivity properties of the same material system.

These studies demonstrate that aluminium substitution provides a useful means of controlling the properties of M-type barium hexaferrite. However, the relationship between aluminium concentration and magnetic performance is not necessarily linear. The optimum composition depends on the intended application and the interaction between crystal structure, particle morphology, magnetic anisotropy, and microstructure.

Objectives of the paper:

The main objective of this paper is to examine the structural and physical-property modifications produced by Al^{3+} substitution in M-type barium hexaferrite nanoparticles synthesized using the sol-gel auto-combustion technique.

1. To explain the structural characteristics and fundamental magnetic properties of M-type barium hexaferrite.
2. To describe the sol-gel auto-combustion method for preparing $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ nanoparticles.
3. To investigate, using published literature, how Al^{3+} substitution influences lattice parameters, crystallite size, phase formation, and particle morphology.
4. To examine the effect of aluminium substitution on saturation magnetization, remanent magnetization, and coercivity.
5. To discuss the influence of Al^{3+} concentration on electrical resistivity, dielectric properties, and alternating-current conductivity.
6. To identify the potential technological applications and limitations of aluminium-substituted barium hexaferrite.

Hypothesis of the Paper:

The research hypothesis is that partial substitution of Fe^{3+} ions by Al^{3+} ions in M-type barium hexaferrite modifies the crystal structure and magnetic exchange interactions, producing measurable changes in its structural, magnetic, electrical, and dielectric properties. The following specific hypotheses are proposed:

1. Increasing aluminium concentration will generally reduce saturation magnetization because essentially nonmagnetic Al^{3+} ions replace magnetic Fe^{3+} ions.
2. Aluminium substitution will modify lattice parameters and may reduce crystallite growth because the ionic size and bonding characteristics of Al^{3+} differ from those of Fe^{3+} .
3. Coercivity will depend on aluminium concentration, particle size, and magnetocrystalline anisotropy; therefore, an optimum substitution level may exist rather than a continuously increasing trend.
4. Increasing aluminium substitution will generally increase electrical resistivity and modify dielectric behaviour by influencing charge transport and interfacial polarization.

These hypotheses are consistent with trends reported in the published literature, but their validity for a particular synthesis procedure must be established experimentally.

Theoretical Background:

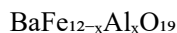
➤ Crystal Structure of M-Type Barium Hexaferrite

M-type barium hexaferrite has the nominal chemical formula $\text{BaFe}_{12}\text{O}_{19}$. Its structure is commonly described using the hexagonal space group $\text{P6}_3/\text{mmc}$. The crystal structure consists of blocks of iron-oxygen polyhedra and barium-containing structural units arranged along the crystallographic c-axis. Iron ions occupy five crystallographically distinct sites: three octahedral sites, one tetrahedral site, and one trigonal-bipyramidal site. In the conventional description, these are denoted as 12k, 2a, 2b, 4f₁, and 4f₂ sites. Their magnetic moments are coupled through super exchange interactions mediated by oxygen ions.

The magnetic moments associated with different iron sublattices point in opposite directions. Because the populations and magnetic contributions of the opposing sublattices are unequal, the material exhibits ferrimagnetism and a nonzero net magnetic moment. The structural and magnetic properties of barium hexaferrite are sensitive to cation substitution, defects, grain boundaries, and particle dimensions. Consequently, changes in chemical composition can influence both intrinsic magnetic interactions and extrinsic microstructural effects.

➤ Aluminium Substitution in Barium Hexaferrite

In aluminium-substituted barium hexaferrite, Al^{3+} ions replace a fraction of the Fe^{3+} ions according to:



The substitution is nominally charge-neutral because both Al^{3+} and Fe^{3+} have the same oxidation state. Therefore, oxygen vacancies or other compensating defects are not required by the ideal substitution formula, although real samples may contain defects resulting from synthesis and processing. Al^{3+} has a smaller ionic radius than Fe^{3+} in comparable coordination environments. Its incorporation can therefore change interatomic distances, lattice parameters, local strain, and the distribution of cations among crystallographic sites. The magnitude and direction of structural changes should be determined from diffraction measurements rather than assumed solely from ionic radii.

Because Al^{3+} is essentially nonmagnetic, replacing Fe^{3+} changes the magnetic contributions of the sublattices. The effect depends partly on which iron sites are preferentially occupied by aluminium. If substitution occurs at sites contributing strongly to the net magnetic moment, saturation magnetization may decrease substantially.

➤ Magnetic Properties

The principal magnetic parameters used to characterize aluminium-substituted barium hexaferrite are saturation magnetization, remanent magnetization, and coercivity. Saturation magnetization (M_s) is the magnetization approached when the magnetic moments are aligned under a sufficiently strong applied magnetic field. Replacing Fe^{3+} with nonmagnetic Al^{3+} generally decreases M_s because the number and distribution of magnetic iron ions change. Remanent magnetization (M_r) is the magnetization remaining after the external magnetic field is removed from a magnetized sample. The ratio M_r/M_s provides information about magnetic reversal, particle orientation, and domain structure.

Coercivity (H_c) is the reverse magnetic field required to reduce the magnetization to zero after prior magnetization. It depends on magnetocrystalline anisotropy, particle size, domain configuration, defects, strain, and microstructural interactions. Aluminium substitution may increase coercivity over a particular composition range, but excessive substitution can produce a different trend. Consequently, a decrease in saturation magnetization does not necessarily mean that all other magnetic properties deteriorate. The best composition depends on the required balance between magnetization, coercivity, and magnetic energy product.

➤ Electrical and Dielectric Properties

The electrical properties of ferrites are often interpreted in terms of charge transport, grain boundaries, and localized hopping mechanisms. In iron-containing ferrites, electronic conduction may involve $\text{Fe}^{2+}/\text{Fe}^{3+}$ hopping when such mixed-valence states are present. Aluminium substitution can influence this process by changing the iron concentration, defect chemistry, grain structure, and availability of charge-transport pathways. Published work on $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ reports increasing direct-current electrical resistivity with increasing aluminium content over the investigated composition range (Dhage et al., 2021).

The dielectric constant and dielectric loss are also composition- and frequency-dependent. The Maxwell-Wagner interfacial polarization model and Koop's phenomenological approach are commonly used to interpret the contributions of relatively conductive grains and more resistive grain boundaries. At low frequencies, charge carriers may accumulate at interfaces, resulting in relatively high dielectric polarization. At higher frequencies, charge carriers may be unable to follow the alternating electric field, causing a decrease in the dielectric response. The exact behaviour depends on composition, microstructure, temperature, and measurement frequency.

Materials and Methodology:

➤ Raw Materials

A typical preparation of aluminium-substituted barium hexaferrite requires high-purity precursor chemicals, such as: Barium nitrate, $\text{Ba}(\text{NO}_3)_2$, as the barium source.

Iron nitrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, as the iron source.

Aluminium nitrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, as the aluminium source.

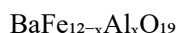
Citric acid or another suitable organic fuel and complexing agent.

Distilled or deionized water as the solvent.

The precise precursor quantities depend on the selected value of x and the hydration state and purity of the chemicals. The oxidizer-to-fuel ratio must be controlled to support reproducible combustion and minimize residual carbon or unwanted phases.

➤ **Stoichiometric Composition**

The general chemical composition is:



For example, a composition series may be selected as $x = 0.0, 0.4$, and 0.8 , allowing the undoped material to be compared with two aluminium-substituted samples. These values are an illustrative experimental design, not newly measured results. For each composition, the required molar amounts of barium, iron, and aluminium precursors are calculated from the nominal formula. Maintaining the intended Ba:Fe:Al ratio is essential for comparing the effects of aluminium substitution.

➤ **Sol-Gel Auto-Combustion Process**

The sol-gel auto-combustion technique combines solution-based chemical mixing with an exothermic combustion reaction. It can produce fine, chemically homogeneous oxide powders without relying exclusively on prolonged solid-state diffusion.

The typical procedure is as follows:

Dissolve the calculated amounts of barium nitrate, iron nitrate, and aluminium nitrate separately in distilled water.

Mix the precursor solutions thoroughly to obtain a uniform metal-ion solution.

Add citric acid in a controlled proportion to complex the metal ions and promote homogeneous distribution.

Adjust the solution pH if required by the chosen synthesis protocol.

Heat the solution under continuous stirring until a viscous gel forms.

Continue heating under controlled conditions until the gel undergoes self-sustained combustion, producing a porous precursor powder.

Collect the combustion product and calcine it under a selected temperature and duration to promote formation of the M-type hexaferrite phase.

Grind the calcined powder gently to improve uniformity and prepare it for characterization.

The calcination temperature, heating rate, dwell time, fuel ratio, and pH should be optimized experimentally. These parameters strongly influence phase purity, crystallite size, agglomeration, and magnetic properties. Because metal nitrates and organic fuels can react vigorously, the combustion process must be performed using appropriate laboratory safety procedures, suitable ventilation, and controlled quantities of precursor materials.

➤ **Characterization Techniques**

The following characterization methods are appropriate for investigating the synthesized nanoparticles.

X-ray diffraction (XRD): XRD is used to identify crystalline phases, examine phase purity, and estimate lattice parameters. The average crystallite size may be estimated from diffraction peak broadening using the Scherrer equation:

$$D = K\lambda/(\beta \cos \theta)$$

where D is the average crystallite size, K is the shape factor, λ is the X-ray wavelength, β is the corrected full width at half maximum of a selected diffraction peak in radians, and θ is the Bragg angle. This estimate represents coherent diffraction-domain size and should not be treated as identical to the particle size measured by electron microscopy.

Scanning electron microscopy (SEM): SEM is used to examine surface morphology, particle agglomeration, and grain dimensions.

Transmission electron microscopy (TEM): TEM can provide direct information about nanoscale particle dimensions, morphology, and, where suitable imaging is available, lattice fringes.

Energy-dispersive X-ray spectroscopy (EDX/EDS): This technique can verify the presence of Ba, Fe, Al, and O and provide an approximate elemental composition. It does not, by itself, establish the precise crystallographic site occupied by aluminium.

Fourier-transform infrared spectroscopy (FTIR): FTIR can identify characteristic metal-oxygen vibrational bands associated with the ferrite structure.

Vibrating sample magnetometry (VSM): VSM is used to measure magnetic hysteresis loops and determine saturation magnetization, remanent magnetization, and coercivity.

Electrical and dielectric measurements: Two-probe or four-probe resistivity measurements and impedance analysis can be used to investigate electrical transport, dielectric constant, dielectric loss, and frequency-dependent conductivity.

For a reliable comparison, all samples should be prepared and measured under consistent conditions, and uncertainties should be reported.

Discussion – is based on published Literature

➤ Phase Formation and Structural Properties

Published investigations show that aluminium-substituted barium hexaferrite can be synthesized using solution combustion and sol-gel auto-combustion methods. Dhage et al. (2011) studied $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ compositions over $x = 0.00$ – 1.00 using a solution-combustion route. Hojjati et al. (2015) examined a broader substitution range, from $x = 0.0$ to $x = 3.0$, using sol-gel auto-combustion processing. These studies establish the relevance of combustion-based synthesis for preparing aluminium-substituted barium hexaferrite. Nevertheless, the presence of an M-type phase and the absence of secondary phases must be verified separately for every composition and heat-treatment condition. Aluminium incorporation can modify the lattice dimensions and introduce local structural strain. Because the observed lattice response depends on site occupancy, phase purity, and processing conditions, lattice parameters should be calculated from measured diffraction patterns. Rietveld refinement, when appropriate, can provide a more reliable assessment than relying on a small number of diffraction peaks.

➤ Crystallite Size and Morphology

Particle size and morphology are important because they affect magnetic-domain behaviour, surface effects, agglomeration, and coercivity. The sol-gel auto-combustion process can produce porous precursor powders with relatively fine particles, but the subsequent calcination treatment can promote grain growth and agglomeration.

Sarkar et al. (2021) reported nanosized aluminium-substituted barium hexaferrite synthesized by sol-gel auto-combustion using urea as fuel. Their investigation used XRD, SEM, EDX, TEM, and VSM to evaluate the structural, morphological, compositional, and magnetic characteristics.

Their work supports the use of complementary characterization methods because XRD estimates crystallite dimensions, while SEM and TEM reveal particle morphology and dimensions more directly. The distinction is important when interpreting the effects of aluminium concentration on magnetic behaviour.

➤ Effect of Aluminium Substitution on Saturation Magnetization

The saturation magnetization of M-type barium hexaferrite is determined by the net contribution of the magnetic iron sublattices. Since Al^{3+} is essentially nonmagnetic, replacing Fe^{3+} with Al^{3+} generally reduces the net magnetic moment.

Sarkar et al. (2021) reported that saturation magnetization decreased from 57.1 emu g^{-1} for their undoped sample to 33.6 emu g^{-1} for the composition containing $x = 0.8$. This provides a quantitative example of the reduction in saturation magnetization associated with aluminium substitution under their synthesis and measurement conditions.

The decrease is consistent with magnetic dilution and changes in exchange interactions. However, the magnitude of the decrease can vary with aluminium concentration, cation distribution, crystallinity, particle size, and synthesis conditions. Therefore, the reported values should not be assumed to apply to every preparation route.

➤ Effect on Coercivity

Coercivity is controlled by the energy required to reverse the magnetization. In barium hexaferrite nanoparticles, this energy is influenced by magnetocrystalline anisotropy, particle dimensions, defects, and magnetic-domain configuration.

Sarkar et al. (2021) reported an increase in coercivity from 1737 G for the undoped sample to 2071 G for the sample containing $x = 0.8$. Hojjati et al. (2015) also reported a composition-dependent coercivity trend, with the coercive field increasing from approximately 2.7 kOe for the undoped composition to approximately 6.1 kOe at $x = 1.5$, followed by a decrease at higher substitution levels.

These results show that aluminium substitution can improve coercivity over selected composition ranges. However, coercivity does not necessarily increase monotonically with aluminium concentration. The optimum concentration depends on the interplay between anisotropy, particle size, microstructure, and magnetic interactions.

The distinction between saturation magnetization and coercivity is especially important in material design. A composition that exhibits high coercivity may have lower saturation magnetization, so both parameters must be considered when evaluating permanent-magnet performance.

➤ Electrical Resistivity and Dielectric Properties

Dhage et al. (2021) investigated $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ nanoparticles with $x = 0.00, 0.25, 0.50, 0.75$, and 1.00 prepared using the sol-gel auto-combustion technique. Their study reported that direct-current electrical resistivity and activation energy increased with aluminium concentration over the investigated range. The authors also examined dielectric constant, dielectric loss, loss tangent, alternating-current conductivity, and impedance behaviour. The increase in resistivity can be associated with changes in the charge-transport pathways and the reduced availability of iron-related hopping sites. However, resistivity is also influenced by grain boundaries, porosity, defect concentrations, electrode contacts, and thermal history.

The dielectric response depends on both frequency and composition. At lower frequencies, interfacial polarization may contribute strongly to the measured dielectric constant. As frequency increases, the polarization mechanisms become less able to follow the applied field, and the dielectric constant and loss may decrease. Dhage et al. (2021) reported decreasing dielectric parameters with increasing frequency and aluminium content under their investigated conditions.

The same study reported increasing AC conductivity with increasing frequency, consistent with frequency-dependent charge transport in the samples. These findings indicate that aluminium substitution may be useful for tailoring electrical and dielectric characteristics in addition to magnetic properties.

➤ Summary of Reported Findings

The following table summarizes selected results from the cited literature. These values are published observations, not measurements obtained in the present paper.

Reference	Composition or range	Main reported findings
Dhage et al. (2011)	$\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$, $x = 0.00-1.00$	Investigated structural and magnetic behaviour using solution combustion.
Hojjati et al. (2015)	$\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$, $x = 0.0-3.0$	Reported decreasing saturation magnetization and a non-monotonic coercivity trend.
Sarkar et al. (2021)	$x = 0.0, 0.4, 0.8$	Reported decreasing saturation magnetization and increasing coercivity over the studied range.
Dhage et al. (2021)	$x = 0.00-1.00$	Reported increasing DC resistivity and activation energy, with composition- and frequency-dependent dielectric properties.

Taken together, these findings support the conclusion that Al^{3+} substitution is an effective means of modifying the physical properties of M-type barium hexaferrite. The structural, magnetic, and electrical responses should nevertheless be evaluated together because optimization of one property may involve a trade-off with another.

Potential Applications

Aluminium-substituted barium hexaferrite nanoparticles may be useful in several technological fields.

Permanent magnets: Selected substitution levels may provide useful coercivity and magnetic stability, although the reduction in saturation magnetization must be considered.

Magnetic recording: Changes in coercivity and particle dimensions may be useful for tailoring magnetic reversal behaviour and recording performance.

Microwave devices: The magnetic and dielectric responses of hexaferrites make them candidates for high-frequency components, subject to suitable measurements of permeability, permittivity, and microwave losses.

Electromagnetic interference suppression: Ferrite-based materials may contribute to electromagnetic absorption or shielding, but application-specific testing is required to establish their effectiveness.

Electronic components: The composition-dependent resistivity and dielectric properties may be relevant to selected electronic and high-frequency applications.

These applications represent potential uses based on the general properties of hexaferrites. Actual performance depends on the final composition, microstructure, operating frequency, temperature, and device design.

➤ Limitations and Future Scope

Although published studies establish important trends, several questions remain relevant for future investigations. First, aluminium substitution must be optimized for the intended application because the magnetic response is not necessarily monotonic. Second, the relationship between cation distribution and magnetic properties requires careful analysis, potentially using advanced structural or spectroscopic methods. Third, synthesis variables such as fuel ratio, pH, calcination temperature, and dwell time should be systematically controlled. Further studies should include quantitative phase analysis, crystallite-size and particle-size distributions, reproducibility tests, and uncertainty estimates. Magnetic measurements should use clearly specified field ranges, sample masses, and correction procedures. Electrical and dielectric measurements should report temperature, frequency range, electrode configuration, and pellet preparation conditions. Finally, comparisons between different publications should be made cautiously because synthesis methods, heat treatments, particle morphology, and measurement protocols can differ substantially.

Conclusion:

Al^{3+} -substituted M-type barium hexaferrite, represented by $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$, is a promising magnetic oxide material whose properties can be modified through controlled aluminium substitution and suitable synthesis conditions. The

sol-gel auto-combustion technique offers an effective route for preparing compositionally controlled ferrite powders and investigating the relationship between chemical composition, microstructure, and physical properties. The literature indicates that replacing magnetic Fe^{3+} ions with essentially nonmagnetic Al^{3+} ions generally decrease saturation magnetization. Published studies also show that coercivity can increase over selected substitution ranges, although the trend may reverse at higher aluminium concentrations. Structural changes, particle dimensions, and microstructural effects contribute to these magnetic responses.

Electrical investigations further demonstrate that aluminium substitution can increase direct-current resistivity and alter dielectric and alternating-current conductivity behaviour. These properties make the material relevant to research on magnetic components, electronic devices, and high-frequency applications.

The research hypothesis is therefore supported by the trends reported in the literature: aluminium substitution modifies the structural, magnetic, and electrical characteristics of M-type barium hexaferrite. However, the optimum aluminium concentration cannot be specified universally. It must be established experimentally by balancing phase purity, saturation magnetization, coercivity, particle size, resistivity, and dielectric performance for the intended application.

Systematic composition-dependent investigation using XRD, SEM/TEM, elemental analysis, VSM, and electrical characterization is necessary to establish reliable structure-property relationships and optimize aluminium-substituted barium hexaferrite nanoparticles.

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